Supporting Information

Cu(I)- and Pd(II)-Catalyzed Decarboxylative Cross-Couplings of Alkynyl Carboxylic Acids with *N*-Tosylhydrazones: Access to Trisubstituted Allenes and Conjugated Enynes

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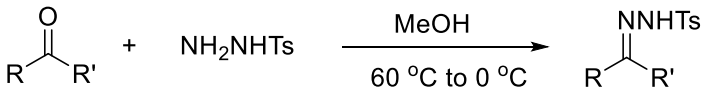
1. General remarks

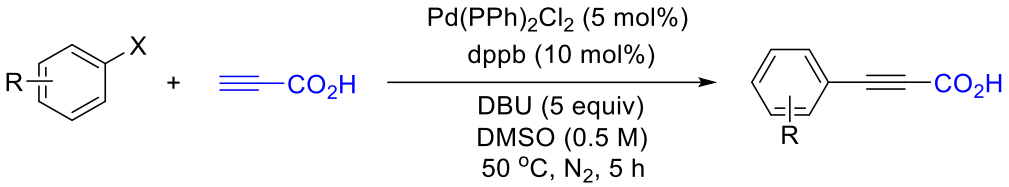
^{1}H, ^{13}C NMR data were obtained on AVANCE III Bruker 400 M Hz nuclear resonance spectrometers unless otherwise noted. $CDCl_3$ was used as solvent and tetramethylsilane (TMS) was used as the internal standard. Chemical shifts were reported in units (ppm) by assigning TMS resonance in the ^{1}H NMR spectrum as 0.00 ppm. The data of ^{1}H NMR was reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet and br = broad), coupling constant (*J* values) in Hz and integration. Chemical shifts for ^{13}C NMR spectra were recorded in ppm from TMS using the central peak of $CDCl_3$ (77.0 ppm) as the internal standard. Flash chromatography was performed using 200-300 mesh silica gel with the indicated eluent according to standard techniques. Analytical thin-layer chromatography (TLC) was performed on pre-coated, glass-backed silica gel plates. High-resolution mass (HRMS) data were recorded on Bruker APEX IV Fourier transform ion cyclotron resonance mass spectrometer using electrospray ionization (ESI and EI) by the Analytical Center of Peking University. PE: petroleum ether; EA: ethyl acetate; DCM: dichloromethane; BQ: benzoquinone.

2. General procedure for the preparation of *N*-tosylhydrazones[1]



A solution of $TsNHNH_2$ (5 mmol) in methanol (5 mL) was stirred and heated to 60 ºC until the $TsNHNH_2$ was completely dissolved. Then carbonyl compounds were dropped to the mixture slowly. After approximately 5-30 min the crude products was obtained as precipitates. The precipitates were washed by petroleum ether then were dried in vacuo to afford the pure products. The reaction provides the *N*-tosylhydrazones **1** in about 85-99% yields.

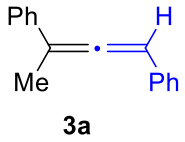
3. General procedure for the synthesis of alkynyl carboxylic acids[2]



Alkynyl carboxylic acids were prepared according to the literature. The indicated aryl halides (2 mmol, 1 equiv), $Pd(PPh_3)_2Cl_2$ (0.1 mmol, 0.05 equiv), 1,4-bis(diphenyl-phosphino)butane (0.2 mmol, 0.1 equiv), DBU (1.0 mmol, 5 equiv) and DMSO (4.0 mL) were combined in an oven-dried round-bottom flask equipped with a stir bar. Propiolic acid (2 mmol, 1 equiv) was added and the resulting mixture was placed in an oil bath at 50 °C for 5 h. The reaction was poured into ethyl acetate and extracted with water saturated by $NaHCO_3$. The aqueous layer was acidified to pH 2.0 by cold 1.0 N HCl (aq) and extracted with CH_2Cl_2. The organic layer dried over $MgSO_4$, and filtered. The solvent was removed under vacuum, and the resulting crude product was purified by flash chromatography on silica gel to give the alkynyl carboxylic acid products.

4. Characterization data of trisubstituted allenes and conjugated enynes

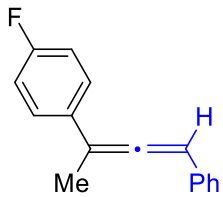
Buta-1,2-diene-1,3-diyldibenzene (3a) [1]



3a

30 mg, 72% yield; colorless oil; R_f = 0.7 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.42 (m, 2H), 7.36-7.26 (m, 6H), 7.25-7.17 (m, 2H), 6.47 (q, *J* = 2.9 Hz, 1H), 2.22 (d, *J* = 2.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.9, 136.4, 134.6, 128.7, 128.5, 127.1, 127.1, 126.9, 125.9, 104.6, 96.6, 16.8. Analytical data are in accordance with the literature values.

1-Fluoro-4-(4-phenylbuta-2,3-dien-2-yl)benzene (3b)

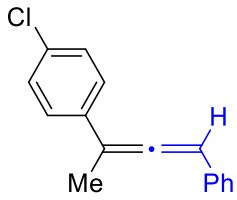


3b

29 mg, 65% yield; yellow oil; R_f = 0.7 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.37 (m, 2H), 7.35-7.25 (m, 4H), 7.25-7.17 (m, 1H), 7.04-6.96 (m, 2H), 6.51-6.40 (m, 1H), 2.20 (d, *J* = 2.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.6 (d, *J* = 2.1 Hz), 162.1 (d, *J* = 246.3 Hz), 134.4 , 132.3 (d, *J* = 3.3 Hz), 128.8 , 127.4 (d, *J* = 8.0 Hz),

127.1, 126.9 , 115.3 (d, J = 21.6 Hz), 103.8 , 96.8 , 17.0; ^{19}F NMR (471 MHz, $CDCl_3$): δ -115.64 (m); HRMS (EI) *m/z* calcd for $[C_{16}H_{13}F]^+$: 224.1001, found 224.0096.

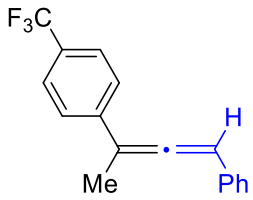
1-Chloro-4-(4-phenylbuta-2,3-dien-2-yl)benzene (3c) [1]



3c

30 mg, 63% yield; yellow oil; R_f = 0.7 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.34 (m, 2H), 7.34-7.25 (m, 6H), 7.25-7.18 (m, 1H), 6.48 (q, J = 2.9 Hz, 1H), 2.20 (d, J = 2.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.8, 134.9, 134.2, 132.8, 128.8, 128.6, 127.2, 127.1, 126.9, 103.8, 96.9, 16.8. Analytical data are in accordance with the literature values.

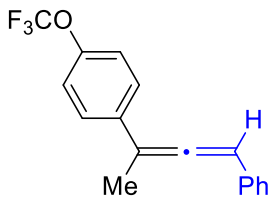
1-(4-Phenylbuta-2,3-dien-2-yl)-4-(trifluoromethyl)benzene (3d) [1]



3d

34 mg, 62% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, J = 1.9 Hz, 4H), 7.32 (d, J = 4.2 Hz, 4H), 7.26-7.19 (m, 1H), 6.53 (q, J = 3.0 Hz, 1H), 2.24 (d, J = 3.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.5, 140.3, 133.8, 128.9 (q, J = 32.4 Hz), 128.8, 127.4, 127.0, 126.0, 125.4 (q, J = 3.9 Hz), 124.3 (q, J = 271.8 Hz), 103.8, 97.2, 16.7; ^{19}F NMR (471 MHz, $CDCl_3$): δ -62.47 (s); Analytical data are in accordance with the literature values.

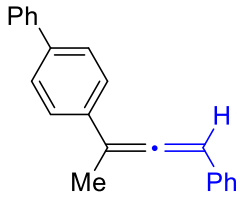
1-(4-Phenylbuta-2,3-dien-2-yl)-4-(trifluoromethoxy)benzene (3e)



3e

36 mg, 62% yield; viscous oil; R_f = 0.2 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.42 (m, 2H), 7.36-7.27 (m, 4H), 7.26-7.13 (m, 3H), 6.49 (q, J = 2.9 Hz, 1H), 2.21 (d, J = 2.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.9, 148.19, 148.17, 135.2, 134.1, 128.8, 127.3, 127.1, 127.0, 121.0, 120.5 (q, J = 256.9 Hz), 103.6, 97.0, 16.8; ^{19}F NMR (471 MHz, $CDCl_3$): δ -57.9 (s); HRMS (EI) *m/z* calcd for $[C_{17}H_{13}F_3O]^+$: 290.0918, found 290.0913.

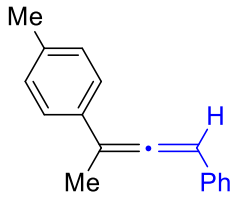
4-(4-Phenylbuta-2,3-dien-2-yl)-1,1'-biphenyl (3f) [3]



3f

31 mg, 55% yield; viscous oil; R_f = 0.2 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.50 (m, 6H), 7.42 (dd, *J* = 8.4, 6.9 Hz, 2H), 7.37-7.28 (m, 5H), 7.21 (tt, *J* = 6.3, 1.6 Hz, 1H), 6.51 (d, *J* = 2.9 Hz, 1H), 2.26 (d, *J* = 2.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.1, 140.8, 139.9, 135.4, 134.5, 128.8, 128.8, 127.3, 127.2, 127.1, 127.00, 126.97, 126.3, 104.3, 96.7, 16.8. Analytical data are in accordance with the literature values.

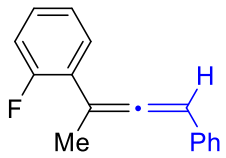
1-Methyl-4-(4-phenylbuta-2,3-dien-2-yl)benzene (3g) [1]



3g

23 mg, 52% yield; viscous oil; R_f = 0.7 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.43 (m, 2H), 7.35-7.28 (m, 2H), 7.25-7.18 (m, 3H), 7.11 (d, *J* = 7.8 Hz, 2H), 6.45 (d, *J* = 2.9 Hz, 1H), 2.33 (s, 3H), 2.21 (d, *J* = 2.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.6, 136.9, 136.6, 131.5, 129.4, 128.5, 127.0, 126.8, 125.8, 104.4, 96.4, 21.2, 16.8. Analytical data are in accordance with the literature values.

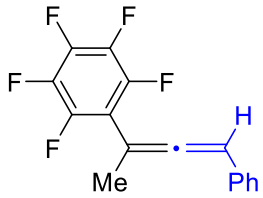
1-Fluoro-2-(4-phenylbuta-2,3-dien-2-yl)benzene (3h)[3]



3h

32 mg, 71% yield; viscous oil; R_f = 0.7 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.27 (m, 5H), 7.25-7.15 (m, 2H), 7.13-6.99 (m, 2H), 6.33 (q, *J* = 2.9 Hz, 1H), 2.23 (dd, *J* = 3.0, 1.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.5, 160.3 (d, *J* = 250.0 Hz), 134.6, 129.0 (d, *J* = 3.5 Hz), 128.69, 128.66, 128.6, 127.04, 126.99, 125.2, 124.0 (d, *J* = 3.7 Hz), 116.1 (d, *J* = 22.5 Hz), 100.2, 94.7 (d, *J* = 1.7 Hz), 18.9 (d, *J* = 2.7 Hz); ^{19}F NMR (471 MHz, $CDCl_3$): δ -112.64 (d, *J* = 5.7 Hz). Analytical data are in accordance with the literature values.

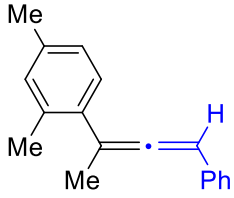
1,2,3,4,5-Pentafluoro-6-(4-phenylbuta-2,3-dien-2-yl)benzene (3i)



3i

40 mg, 68% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.30 (m, 4H), 7.28-7.18 (m, 1H), 6.33 (q, *J* = 3.0 Hz, 1H), 2.17 (dt, *J* = 3.0, 1.0 Hz, 3H; ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.8, 133.0, 128.8, 127.6, 127.4, 95.6, 92.2, 19.4; ^{19}F NMR (400 MHz, $CDCl_3$): δ -140.69 (dd, *J* = 23.1, 8.7 Hz), -156.30 (t, *J* = 21.4 Hz), -162.32 (dd, *J* = 21.6, 14.5 Hz); HRMS (EI) *m/z* calcd for $[C_{16}H_9F_5]^+$: 296.0624, found 296.0618.

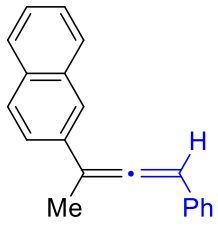
2,4-Dimethyl-1-(4-phenylbuta-2,3-dien-2-yl)benzene (3j) [3]



3j

25 mg, 53% yield; colorless oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 7.3 Hz, 4H), 7.20 (dd, *J* = 12.8, 7.5 Hz, 2H), 7.01 (d, *J* = 8.7 Hz, 2H), 6.19 (d, *J* = 3.1 Hz, 1H), 2.34 (s, 3H), 2.30 (s, 3H), 2.16 (d, *J* = 3.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.2, 136.7, 135.86, 135.2, 134.5, 131.5, 128.6, 127.4, 127.0, 126.7, 126.6, 103.6, 93.9, 21.0, 21.0, 20.6. Analytical data are in accordance with the literature values.

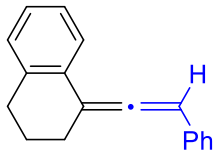
2-(4-Phenylbuta-2,3-dien-2-yl)naphthalene (3k) [1]



3k

25 mg, 49% yield; colorless oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.76 (m, 3H), 7.72 (d, *J* = 8.7 Hz, 1H), 7.63 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.49-7.40 (m, 2H), 7.39-7.27 (m, 4H), 7.26-7.18 (m, 1H), 6.55 (q, *J* = 2.9 Hz, 1H), 2.34 (d, *J* = 2.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.6, 134.5, 133.8, 133.7, 132.6, 128.8, 128.1, 127.9, 127.6, 127.1, 127.0, 126.2, 125.8, 125.0, 123.7, 104.9, 96.9, 16.9. Analytical data are in accordance with the literature values.

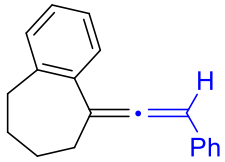
1-(2-Phenylvinylidene)-1,2,3,4-tetrahydronaphthalene **(3l)** [3]



3l

23 mg, 50% yield; light yellow oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (dd, *J* = 6.4, 2.1 Hz, 1H), 7.35-7.26 (m, 4H), 7.23-7.17 (m, 1H), 7.14-7.06 (m, 3H), 6.51 (t, *J* = 3.0 Hz, 1H), 2.86 (t, *J* = 6.2 Hz, 2H), 2.67 (dddd, *J* = 14.9, 9.4, 4.4, 2.2 Hz, 2H), 2.02 – 1.93 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.4, 136.8, 134.7, 130.8, 129.4, 128.7, 127.1, 127.01, 126.96, 126.9, 126.2, 105.8, 97.5, 30.1, 28.8, 23.1. Analytical data are in accordance with the literature values.

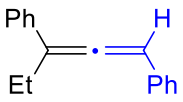
5-(2-Phenylvinylidene)-6,7,8,9-tetrahydro-5H-benzo[7]annulene (3m)



3m

21 mg, 42% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.34 (m, 2H), 7.34-7.26 (m, 3H), 7.23-7.16 (m, 1H), 7.15-7.06 (m, 3H), 6.29 (t, *J* = 1.9 Hz, 1H), 2.91 (dt, *J* = 7.5, 3.8 Hz, 2H), 2.61-2.40 (m, 2H), 1.99-1.74 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.0, 140.8, 138.7, 135.1, 129.7, 129.0, 128.7, 127.3, 126.8, 126.7, 126.2, 111.6, 93.9, 35.9, 32.8, 30.5, 27.3; HRMS (EI) *m/z* calcd for $[C_{19}H_{18}]^+$: 246.1409, found 246.1403.

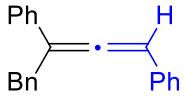
Penta-1,2-diene-1,3-diyldibenzene (3n) [4]



3n

28 mg, 64% yield; viscous liquid; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.42 (m, 2H), 7.37-7.27 (m, 6H), 7.25-7.16 (m, 2H), 6.56 (t, *J* = 3.4 Hz, 1H), 2.68-2.47 (m, 2H), 1.20 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.3, 136.3, 134.8, 128.7, 128.5, 127.05, 127.03, 126.7, 126.1, 111.7, 98.6, 23.2, 12.6. Analytical data are in accordance with the literature values.

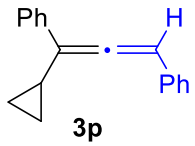
Buta-2,3-diene-1,2,4-triyltribenzene (3o)



3o

24 mg, 43% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.42 (m, 2H), 7.36 – 7.11 (m, 13H), 6.48 (t, *J* = 2.5 Hz, 1H), 4.00 – 3.83 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.0, 139.1, 135.6, 134.2, 128.8, 128.7, 128.5, 128.3, 127.2, 126.9, 126.4, 126.3, 109.0, 97.8, 37.2; HRMS (EI) *m/z* calcd for $[C_{22}H_{18}]^+$: 282.1409, found 282.1402.

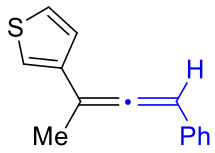
(1-Pyclopropylpropa-1,2-diene-1,3-diyl)dibenzene (3p) [5]



3p

24 mg, 52% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66-7.57 (m, 2H), 7.38-7.27 (m, 6H), 7.26-7.19 (m, 2H), 6.55 (d, *J* = 2.5 Hz, 1H), 1.70 (ttd, *J* = 7.8, 5.0, 2.6 Hz, 1H), 0.97-0.83 (m, 2H), 0.68 – 0.55 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.7, 136.5, 134.3, 128.8, 128.5, 127.20, 127.15, 126.7, 126.4, 113.3, 99.0, 11.1, 7.4, 6.9. Analytical data are in accordance with the literature values.

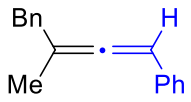
3-(4-Phenylbuta-2,3-dien-2-yl)thiophene (3q)



3q

27 mg, 64% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 5.8 Hz, 4H), 7.26-7.10 (m, 4H), 6.42 (d, *J* = 3.2 Hz, 1H), 2.20 (d, *J* = 2.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.8, 138.5, 134.6, 128.7, 127.03, 126.99, 126.7, 125.6, 119.6, 100.9, 96.1, 17.4; HRMS (EI) *m/z* calcd for $[C_{14}H_{12}S]^+$: 212.0660, found 212.0654.

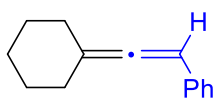
(3-Methylbuta-1,2-diene-1,4-diyl)dibenzene (3r)



3r

27 mg, 57% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.41 (m, 2H), 7.39-7.26 (m, 6H), 7.25-7.16 (m, 2H), 6.57 (t, *J* = 3.4 Hz, 1H), 2.59 (td, *J* = 7.2, 3.4 Hz, 2H), 1.20 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.3, 136.2, 134.8, 128.7, 128.5, 127.05, 127.02, 126.7, 126.1, 111.7, 98.6, 23.2, 12.6; HRMS (EI) *m/z* calcd for $[C_{17}H_{16}]^+$: 220.1252, found 220.1248.

(2-Cyclohexylidenevinyl)benzene (3s) [6]

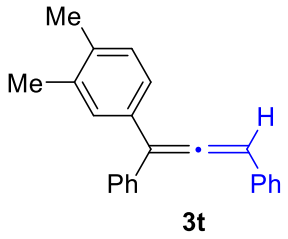


3s

12 mg, 32% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 4.4 Hz, 4H), 7.16 (q, *J* = 4.4 Hz, 1H), 6.09-5.91 (m, 1H), 2.23 (dtd, *J* = 21.9, 12.1, 11.5, 4.5 Hz, 4H), 1.77-1.53

(m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.7, 136.1, 128.5, 126.5, 126.3, 106.5, 92.3, 31.3, 27.7, 26.1; Analytical data are in accordance with the literature values.

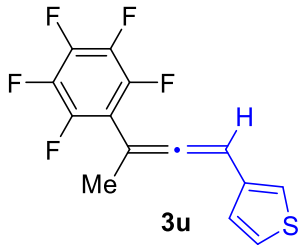
(1-(3,4-Dimethylphenyl)propa-1,2-diene-1,3-diyl)dibenzene (3t)



3t

21 mg, 36% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (td, *J* = 8.1, 7.7, 1.5 Hz, 4H), 7.36-7.25 (m, 5H), 7.25-7.18 (m, 2H), 7.17-7.08 (m, 2H), 6.68 (s, 1H), 2.26 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.1, 136.7, 136.5, 136.1, 134.2, 133.5, 129.8, 129.6, 128.8, 128.48, 128.45, 127.5, 127.3, 127.0, 126.0, 113.6, 97.5, 19.9, 19.6; HRMS (EI) *m/z* calcd for $[C_{23}H_{20}]^+$: 296.1565, found 296.1558.

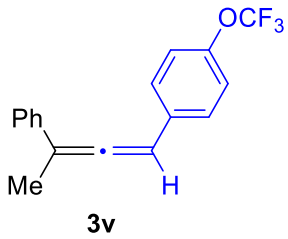
3-(3-(Perfluorophenyl)buta-1,2-dien-1-yl)thiophene (3u)



3u

45 mg, 74% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (t, *J* = 4.0 Hz, 1H), 7.22-7.05 (m, 2H), 6.41 (q, *J* = 3.2 Hz, 1H), 2.16 (d, *J* = 3.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.1, 145.6, 143.1, 139.00, 138.9, 136.45, 134.2, 126.5, 126.2, 122.3, 113.3, 91.3, 90.1, 19.5; ^{19}F NMR (400 MHz, $CDCl_3$): δ -140.94 (dd, *J* = 23.8, 9.5 Hz), -156.32 (t, *J* = 21.4 Hz), -161.98 – -162.79 (m); HRMS (EI) *m/z* calcd for $[C_{14}H_7F_5S]^+$: 302.0189, found 302.0184.

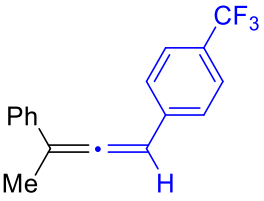
1-(3-Phenylbuta-1,2-dien-1-yl)-4-(trifluoromethoxy)benzene (3v)



3v

36 mg, 62% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.42 (m, 2H), 7.37-7.30 (m, 4H), 7.27-7.21 (m, 1H), 7.18-7.10 (m, 2H), 6.46 (d, *J* = 2.9 Hz, 1H), 2.23 (d, *J* = 3.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.0, 148.1, 148.1, 135.9, 133.4, 128.5, 128.0, 127.3, 125.9, 121.4, 105.1, 95.5, 16.7; ^{19}F NMR (471 MHz, $CDCl_3$): δ -57.92 (s); HRMS (EI) *m/z* calcd for $[C_{17}H_{13}F_3O]^+$: 290.0918, found 290.0913.

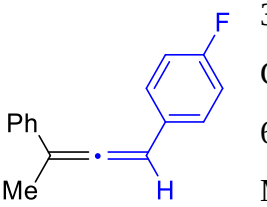
1-(3-Phenylbuta-1,2-dien-1-yl)-4-(trifluoromethyl)benzene (3w) [1]



3w

35 mg, 64% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.7 Hz, 2H), 7.46-7.39 (m, 4H), 7.34 (ddd, *J* = 7.8, 6.8, 1.2 Hz, 2H), 7.28-7.21 (m, 1H), 6.49 (q, *J* = 2.9 Hz, 1H), 2.24 (d, *J* = 2.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.8, 138.5 (d, *J* = 1.7 Hz), 135.7, 128.9 (q, *J* = 32.3 Hz), 128.6, 127.4, 127.0, 125.9, 125.6 (q, *J* = 3.8 Hz), 122.9, 105.3, 95.8, 16.6; ^{19}F NMR (471 MHz, $CDCl_3$): δ -62.43 (s). Analytical data are in accordance with the literature values.

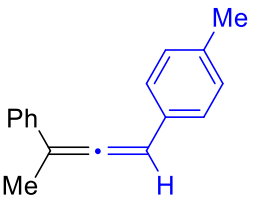
1-Fluoro-4-(3-phenylbuta-1,2-dien-1-yl)benzene (3x)



3x

32 mg, 71% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.41 (m, 2H), 7.36-7.20 (m, 5H), 7.03-6.96 (m, 2H), 6.44 (q, *J* = 2.9 Hz, 1H), 2.22 (d, *J* = 2.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.5 (d, *J* = 2.4 Hz), 162.0 (d, *J* = 246.1 Hz), 136.2, 130.5 (d, *J* = 3.3 Hz), 128.5, 128.3 (d, *J* = 8.0 Hz), 127.1, 125.8, 115.6 (d, *J* = 21.7 Hz), 104.8, 95.6, 16.8; ^{19}F NMR (471 MHz, $CDCl_3$): δ -108.52, -115.35 (t, *J* = 5.7 Hz); HRMS (EI) m/z calcd for [$C_{16}H_{13}F$]: 224.1001, found 224.0996.

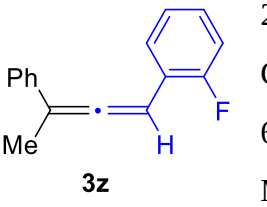
1-Methyl-4-(3-phenylbuta-1,2-dien-1-yl)benzene (3y) [1]



3y

25 mg, 57% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.41 (m, 2H), 7.34-7.28 (m, 2H), 7.25-7.18 (m, 3H), 7.11 (d, *J* = 7.8 Hz, 2H), 6.45 (q, *J* = 2.9 Hz, 1H), 2.33 (s, 3H), 2.21 (d, *J* = 2.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.6, 136.9, 136.6, 131.5, 129.4, 128.5, 127.0, 126.8, 125.8, 104.4, 96.4, 21.2, 16.8. Analytical data are in accordance with the literature values.

1-Fluoro-2-(3-phenylbuta-1,2-dien-1-yl)benzene (3z)

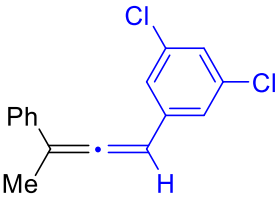


3z

22 mg, 49% yield; colorless oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50-7.29 (m, 5H), 7.26-7.12 (m, 2H), 7.08-7.00 (m, 2H), 6.70 (q, *J* = 3.0 Hz, 1H), 2.23 (d, *J* = 3.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.5 (d, *J* = 2.1 Hz), 159.8 (d, *J* = 249.4 Hz),

136.1, 128.5, 128.3 (dd, *J* = 5.9, 2.2 Hz), 127.1, 125.9, 124.2 (d, *J* = 3.5 Hz), 122.1 (d, *J* = 12.0 Hz), 115.7 (d, *J* = 21.5 Hz), 104.6, 89.0 (d, *J* = 6.5 Hz), 16.7; ^{19}F NMR (471 MHz, $CDCl_3$): δ -114.95 – -122.09 (m); HRMS (EI) *m/z* calcd for $[C_{16}H_{13}F]^+$: 290.0918, found 290.0913.

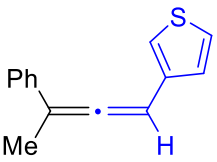
1,3-Dichloro-5-(3-phenylbuta-1,2-dien-1-yl)benzene (3aa)



3aa

46 mg, 84% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.40 (m, 2H), 7.39-7.31 (m, 2H), 7.29-7.23 (m, 1H), 7.21-7.13 (m, 3H), 6.36 (q, *J* = 2.9 Hz, 1H), 2.24 (d, *J* = 2.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 207.5, 138.0, 135.4, 135.2, 128.6, 127.5, 126.9, 126.0, 125.1, 105.8, 95.0, 16.7; HRMS (EI) *m/z* calcd for $[C_{16}H_{12}Cl_2]^+$: 274.0316, found 274.0311.

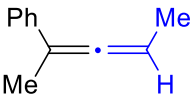
3-(3-Phenylbuta-1,2-dien-1-yl)thiophene (3ab)



3ab

25 mg, 59% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.41 (m, 2H), 7.35-7.29 (m, 2H), 7.26-7.19 (m, 2H), 7.12 (dd, *J* = 3.0, 1.2 Hz, 1H), 7.07 (dd, *J* = 5.0, 1.3 Hz, 1H), 6.54 (q, *J* = 2.9 Hz, 1H), 2.20 (d, *J* = 3.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.8, 136.5, 135.9, 128.5, 127.0, 126.5, 126.0, 125.9, 120.9, 103.7, 91.1, 17.0; HRMS (EI) *m/z* calcd for $[C_{14}H_{12}S]^+$: 212.0660, found 212.0654.

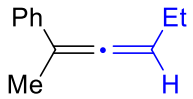
Penta-2,3-dien-2-ylbenzene (3ac) [6]



3ac

10 mg, 36% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.38 (m, 2H), 7.32 (dd, *J* = 8.5, 6.9 Hz, 2H), 7.23-7.16 (m, 2H), 5.49-5.35 (m, 1H), 2.09 (d, *J* = 2.9 Hz, 3H), 1.77 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.9, 137.8, 128.2, 126.3, 125.7, 99.7, 87.6, 17.1, 14.3. Analytical data are in accordance with the literature values.

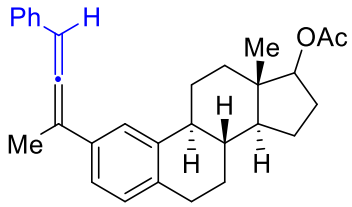
Hexa-2,3-dien-2-ylbenzene (3ad) [7]



3ad

15 mg, 47% yield; viscous oil; R_f = 0.5 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.38 (m, 2H), 7.33 (dd, *J* = 8.5, 7.0 Hz, 2H), 7.23-7.16 (m, 1H), 5.52 (dq, *J* = 6.1, 3.0 Hz, 1H), 2.20-2.07 (m, 5H), 1.08 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.7, 137.8, 128.2, 126.3, 125.6, 100.9, 94.8, 22.3, 17.3, 13.6. Analytical data are in accordance with the literature values.

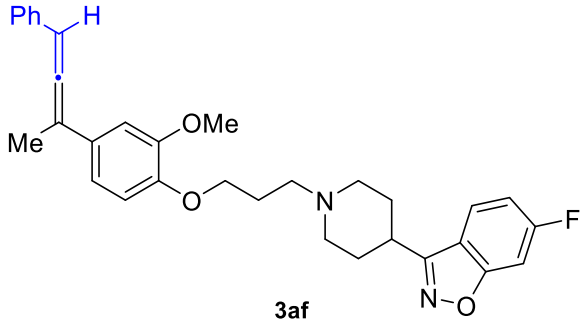
(8R,9S,13S,14S)-13-Methyl-2-(4-phenylbuta-2,3-dien-2-yl)-7,8,9,11,12,13,14,-15,16,17-decahydro-6H-cyclopenta[a]phenanthren-17-yl acetate (3ae)



3ae

55 mg, 66% yield; viscous oil; R_f = 0.2 (PE/EA = 3/1, v/v); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.17 (m, 7H), 7.04 (d, *J* = 8.0 Hz, 1H), 6.45 (t, *J* = 3.3 Hz, 1H), 4.67 (t, *J* = 8.4 Hz, 1H), 2.85 (dd, *J* = 9.1, 4.2 Hz, 2H), 2.36-2.15 (m, 5H), 2.12-2.01 (m, 3H), 1.95-1.82 (m, 2H), 1.79-1.69 (m, 1H), 1.60-1.23 (m, 7H), 0.92-0.74 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.6, 171.3, 140.2, 135.7, 134.8, 133.6, 133.5, 129.2, 128.7, 126.89, 126.86, 123.4, 122.7, 104.7, 96.4, 82.8, 49.9, 44.4, 42.9, 38.4, 36.9, 31.6, 29.3, 27.6, 27.2, 26.0, 23.3, 21.3, 17.0, 12.1; HRMS (EI) *m/z* calcd for $[C_{30}H_{34}O_2]^+$: 426.2559, found 426.2553.

6-Fluoro-3-(1-(3-(2-methoxy-4-(4-phenylbuta-2,3-dien-2-yl)phenoxy)propyl) piperidin-4-yl)benzo[d]isoxazole (3af)

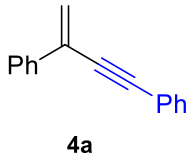


3af

56 mg, 55% yield; viscous oil; R_f = 0.5 (DCM/MeOH = 10/1, v/v); ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.11 (s, 1H), 7.69 (d, *J* = 8.9 Hz, 1H), 7.45-7.18 (m, 6H), 7.06-6.88 (m, 3H), 6.62 (s, 1H), 4.03 (t, *J* = 6.1 Hz, 2H), 3.72 (s, 3H), 3.53-2.62 (m, 6H), 2.51 (s, 2H), 2.28-1.56 (m, 10H), 1.32-1.05 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 206.2, 164.1 (d, *J* = 248.1 Hz), 163.5 (d, *J* = 14.8 Hz), 149.5, 148.0, 134.8, 129.3, 129.0, 127.5, 127.1, 124.4 (d, *J* = 11.4 Hz), 118.6, 117.5, 113.9, 113.2, 112.9, 110.1, 104.5, 98.0, 97.7, 96.6, 67.0, 56.0, 54.6, 52.8, 33.2, 29.5, 29.0, 26.0, 17.3;

^{19}F NMR (471 MHz, DMSO-d_6): δ -109.66; HRMS (ESI) *m/z* calcd for $[C_{32}H_{34}FN_2O_3]^+$ $[M+H]^+$: 513.2553, found 513.2548.

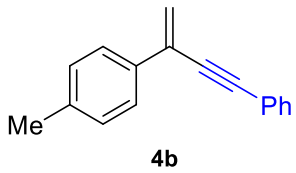
But-3-en-1-yne-1,3-diyldibenzene (4a)[8]



4a

11 mg, 55% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.80-7.71 (m, 2H), 7.60-7.51 (m, 2H), 7.46-7.32 (m, 6H), 6.01 (s, 1H), 5.77 (s, 1H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 137.1, 131.6, 130.5, 128.5, 128.4, 126.0, 123.0, 120.6, 90.7, 88.4. Analytical data are in accordance with the literature values.

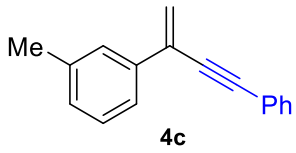
1-Methyl-4-(4-phenylbut-1-en-3-yn-2-yl)benzene (4b) [9]



4b

18 mg, 41% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.69-7.58 (m, 2H), 7.53 (dd, *J* = 6.6, 3.0 Hz, 2H), 7.36 (dd, *J* = 4.8, 2.0 Hz, 3H), 7.20 (d, *J* = 7.9 Hz, 2H), 5.97 (s, 1H), 5.70 (s, 1H), 2.36 (s, 3H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 138.5, 134.2, 131.6, 130.3, 129.1, 128.44, 128.40, 125.9, 123.1, 119.6, 90.5, 88.6, 20.9. Analytical data are in accordance with the literature values.

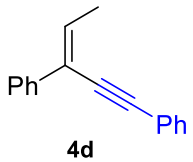
1-Methyl-3-(4-phenylbut-1-en-3-yn-2-yl)benzene (4c)[8]



4c

19 mg, 44% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.58-7.47 (m, 4H), 7.41-7.32 (m, 3H), 7.27 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.5 Hz, 1H), 5.99 (d, *J* = 1.0 Hz, 1H), 5.74 (d, *J* = 1.0 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 138.2, 137.0, 131.6, 130.6, 129.2, 128.5, 128.4, 128.3, 126.7, 123.2, 123.0, 120.5, 90.5, 88.5, 21.2. Analytical data are in accordance with the literature values.

(Z)-Pent-3-en-1-yne-1,3-diyldibenzene (4d)

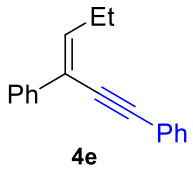


4d

12 mg, 53% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.61 (m, 2H), 7.57-7.49 (m, 2H), 7.40-7.31 (m, 5H), 7.30-7.24 (m, 1H), 6.53 (q, *J* = 7.0 Hz, 1H), 2.15 (d, *J* = 7.0 Hz, 3H; ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.3, 133.3, 131.5, 128.4, 128.35, 128.2, 127.4, 125.9,

124.5, 123.6, 95.6, 86.7, 17.1; HRMS (EI) *m/z* calcd for $[C_{17}H_{14}]^+$: 218.1096, found 218.1087.

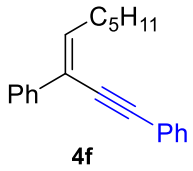
(Z)-Hex-3-en-1-yne-1,3-diyldibenzene (4e)



4e

22 mg, 47% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.69-7.62 (m, 2H), 7.56-7.49 (m, 2H), 7.40-7.33 (m, 5H), 7.30-7.24 (m, 1H), 6.49 (t, *J* = 7.4 Hz, 1H), 2.59 (p, *J* = 7.5 Hz, 2H), 1.15 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 140.5, 138.1, 131.4, 128.39, 128.36, 128.3, 127.5, 125.9, 123.5, 122.6, 95.1, 86.6, 24.8, 13.3; HRMS (EI) *m/z* calcd for $[C_{18}H_{16}]^+$: 232.1252, found 232.1245.

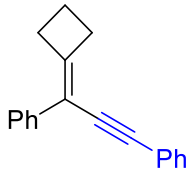
(Z)-Non-3-en-1-yne-1,3-diyldibenzene (4f)



4f

23 mg, 42% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.66 (dd, *J* = 7.7, 1.7 Hz, 2H), 7.53 (ddd, *J* = 7.1, 3.5, 1.7 Hz, 2H), 7.41-7.21 (m, 6H), 6.51 (t, *J* = 7.5 Hz, 1H), 2.58 (q, *J* = 7.5 Hz, 2H), 1.56 (dd, *J* = 9.8, 4.8 Hz, 2H), 1.39 (td, *J* = 6.2, 2.7 Hz, 4H), 0.97-0.84 (m, 3H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 139.3, 138.2, 132.5, 131.4, 128.5, 128.4, 128.3, 128.2, 127.4, 125.9, 123.5, 123.2, 95.0, 86.8, 31.6, 31.3, 28.8, 22.6, 13.8; HRMS (EI) m/z calcd for $[C_{21}H_{22}]$: 274.1722, found 274.1716.

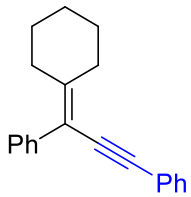
(3-Cyclobutylideneprop-1-yne-1,3-diyl)dibenzene (4g) [10]



4g

8 mg, 33% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.52 (m, 2H), 7.52-7.44 (m, 2H), 7.41-7.27 (m, 5H), 7.23 (s, 1H), 3.11 (qd, *J* = 6.6, 3.4 Hz, 4H), 2.14 (p, *J* = 7.8 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.6, 136.9, 131.4, 128.3, 128.2, 127.8, 126.9, 126.7, 123.9, 115.6, 93.1, 87.0, 33.5, 33.2, 17.5. Analytical data are in accordance with the literature values.

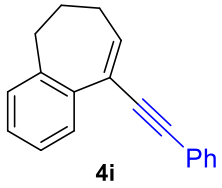
(3-Cyclohexylideneprop-1-yne-1,3-diyl)dibenzene (4h)



4h

10 mg, 37% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ (d, *J* = 4.4 Hz, 4H), 7.27 (dq, *J* = 5.1, 2.2 Hz, 4H), 2.79-2.66 (m, 2H), 2.35-2.21 (m, 2H), 1.74 (td, *J* = 8.1, 7.2, 4.4 Hz, 2H), 1.62 (td, *J* = 7.5, 6.9, 4.0 Hz, 2H), 1.59-1.54 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.9, 139.2, 131.3, 129.3, 128.2, 128.1, 127.6, 126.8, 124.1, 116.1, 92.3, 90.1, 34.2, 31.4, 28.2, 28.1, 26.5; HRMS (EI) *m/z* calcd for $[C_{21}H_{20}]^+$: 272.1565, found 272.1559.

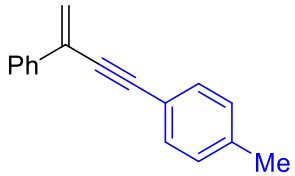
9-(Phenylethynyl)-6,7-dihydro-5H-benzo[7]annulene (4i)



4i

11 mg, 45% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54-7.42 (m, 2H), 7.38-7.27 (m, 4H), 7.24-7.16 (m, 2H), 6.72 (t, *J* = 6.8 Hz, 1H), 2.68 (t, *J* = 6.6 Hz, 2H), 2.23 – 2.04 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.0, 138.9, 137.9, 131.55, 128.9, 128.4, 128.3, 128.0, 127.5, 126.2, 124.9, 123.6, 90.5, 87.5, 33.9, 32.8, 26.7; HRMS (EI) *m/z* calcd for $[C_{19}H_{16}]^+$: 244.1252, found 244.1244.

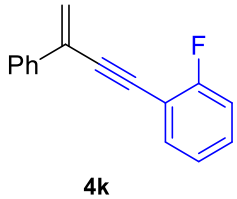
1-Methyl-4-(3-phenylbut-3-en-1-yn-1-yl)benzene (4j) [11]



4j

11 mg, 50% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, CD_2Cl_2) δ 7.82-7.62 (m, 2H), 7.45-7.31 (m, 5H), 7.15 (d, *J* = 7.8 Hz, 2H), 5.96 (d, *J* = 1.0 Hz, 1H), 5.74 (d, *J* = 1.1 Hz, 1H), 2.36 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.6, 137.4, 132.4, 131.6, 130.7, 129.2, 129.1, 128.4, 128.3, 126.1, 120.3, 91.0, 87.9, 21.5. Analytical data are in accordance with the literature values.

1-Fluoro-2-(3-phenylbut-3-en-1-yn-1-yl)benzene (4k) [8]

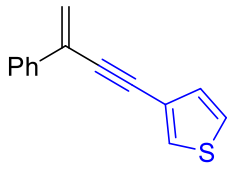


4k

9 mg, 41% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.70 (m, 2H), 7.52 (td, *J* = 7.6, 1.9 Hz, 1H), 7.42-7.30 (m, 4H), 7.16-7.09 (m, 2H), 6.03 (d, *J* = 0.9 Hz, 1H), 5.80 (d, *J* = 0.9 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.8 (d, *J* = 251.8 Hz), 136.9, 133.43, 133.42, 130.4, 130.1 (d, *J* = 8.0 Hz), 128.5, 128.4, 126.1, 124.0 (d,

J = 3.9 Hz), 121.1, 115.5 (d, J = 20.9 Hz), 93.6 (d, J = 3.3 Hz), 84.1. Analytical data are in accordance with the literature values.

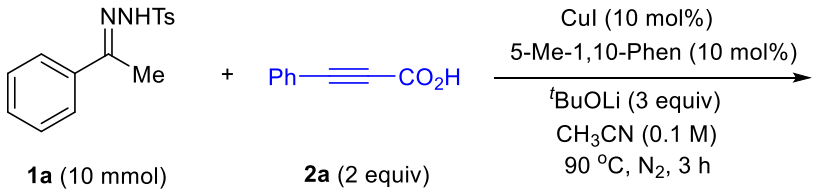
3-(3-Phenylbut-3-en-1-yn-1-yl)thiophene (4l)



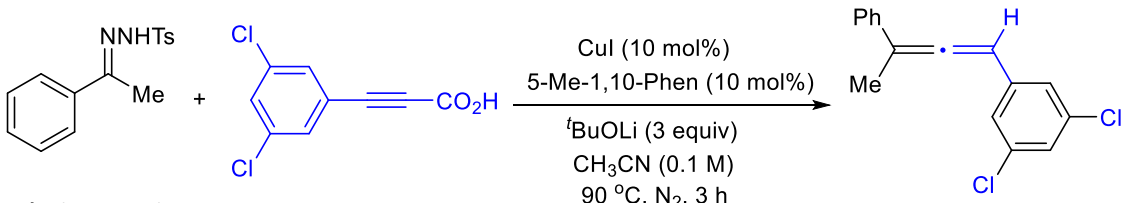
4l

8 mg, 38% yield; viscous oil; R_f = 0.4 (PE); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.66 (m, 2H), 7.56 (dd, J = 3.0, 1.2 Hz, 1H), 7.42-7.31 (m, 4H), 7.21 (dd, J = 5.0, 1.2 Hz, 1H), 5.99 (d, J = 0.9 Hz, 1H), 5.74 (d, J = 0.9 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.1, 131.5, 130.5, 130.1, 129.8, 129.0, 128.4, 126.0, 125.7, 122.0, 120.5, 87.9, 85.8; HRMS (EI) *m/z* calcd for $[C_{14}H_{10}S]^+$: 210.0503, found 210.0496.

5. Experimental procedure of large scale reactions of trisubstituted allenes



1a (10 mmol) **2a** (2 equiv) **3a** (1.31 g, 63% yield)



1a (10 mmol) **2g** (2 equiv) **3aa** (1.79 g, 65% yield)

A solution of CuI (1 mmol, 0.1 equiv), 5-Me-1,10-Phen (1 mmol, 0.1 equiv), ^{t}BuOLi (30 mmol, 3 equiv), **1a** (10 mmol, 1.0 equiv) and **2a** or **2g** (20 mmol, 2 equiv) in MeCN (100 mL) under N_2 atmosphere was stirred at indicated temperature until the complete consumption of **1a** detected by TLC analysis. The reaction mixture was filtered and evaporated under reduced pressure, and purified by column chromatography to give the desired product **3a** or **3aa**.

6. References

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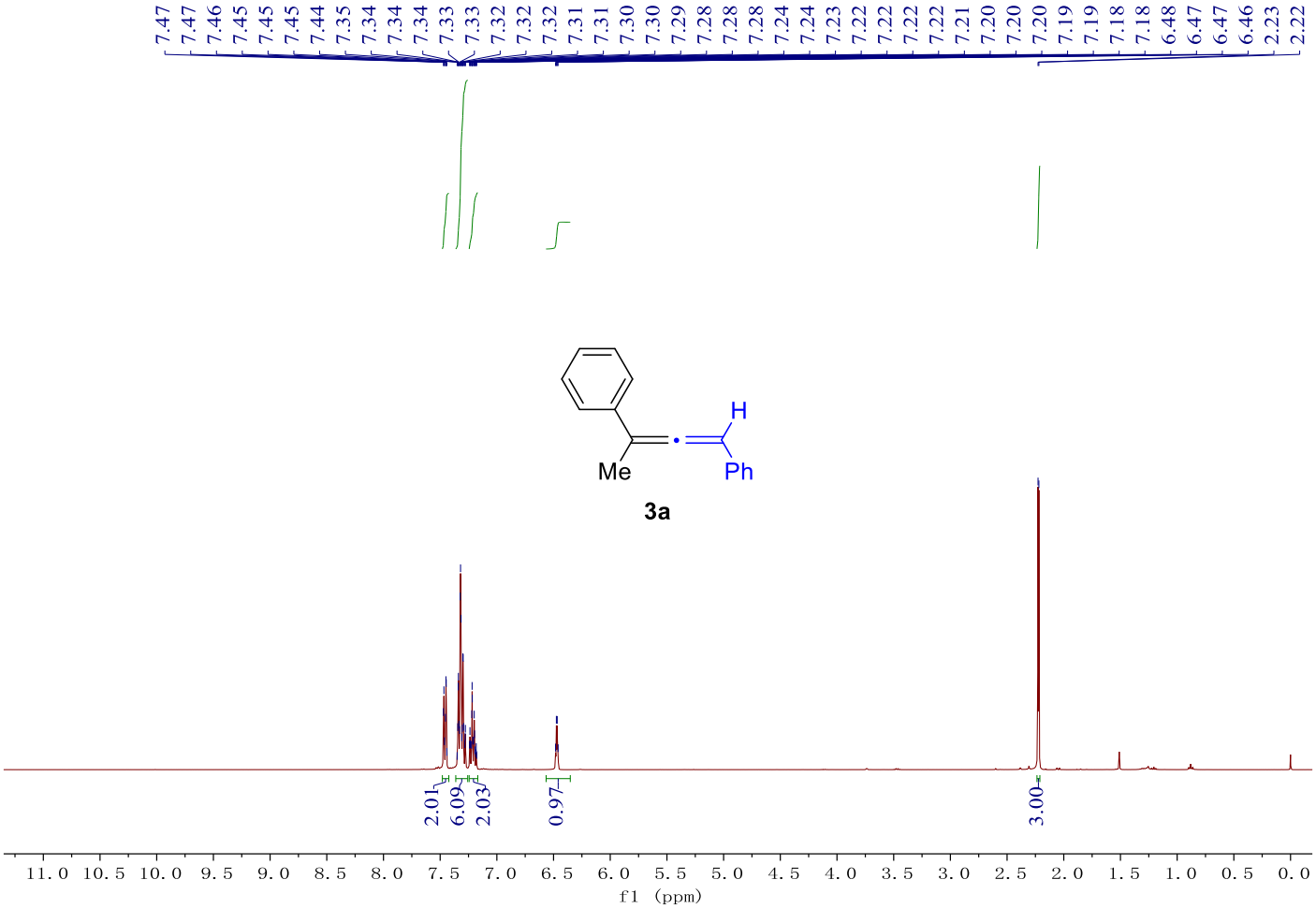
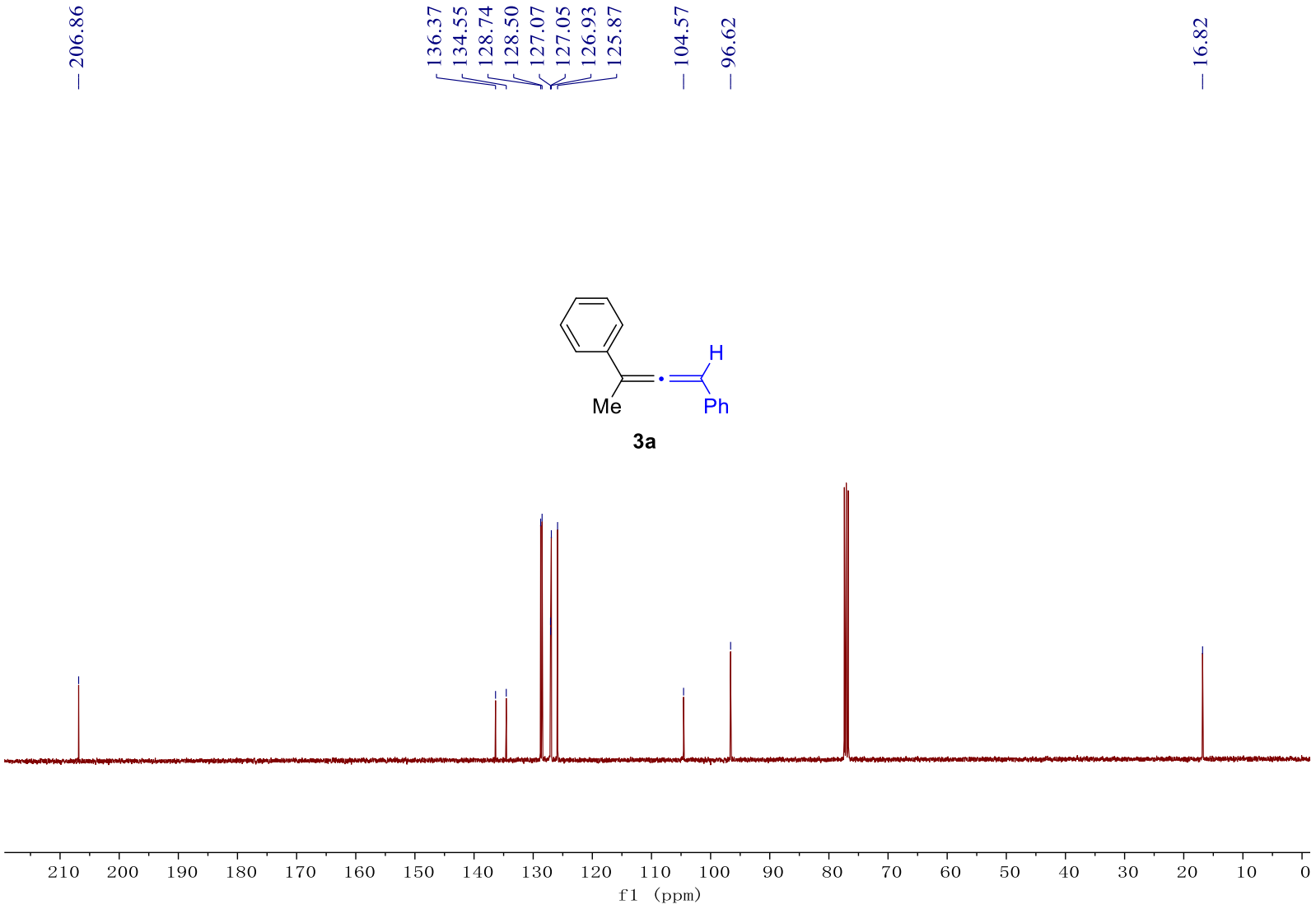
(11) M. Taj Muhammad, Y. Jiao, C. Ye, M. F. Chiou, M. Israr, X. Zhu, Y. Li, Z. Wen, A. Studer, H. Bao, Synthesis of Difluoromethylated Allenes through Trifunctionalization of 1,3-Enynes. *Nat. Commun.* 2020, **11**, 416.

7. ^{1}H and ^{13}C NMR spectra

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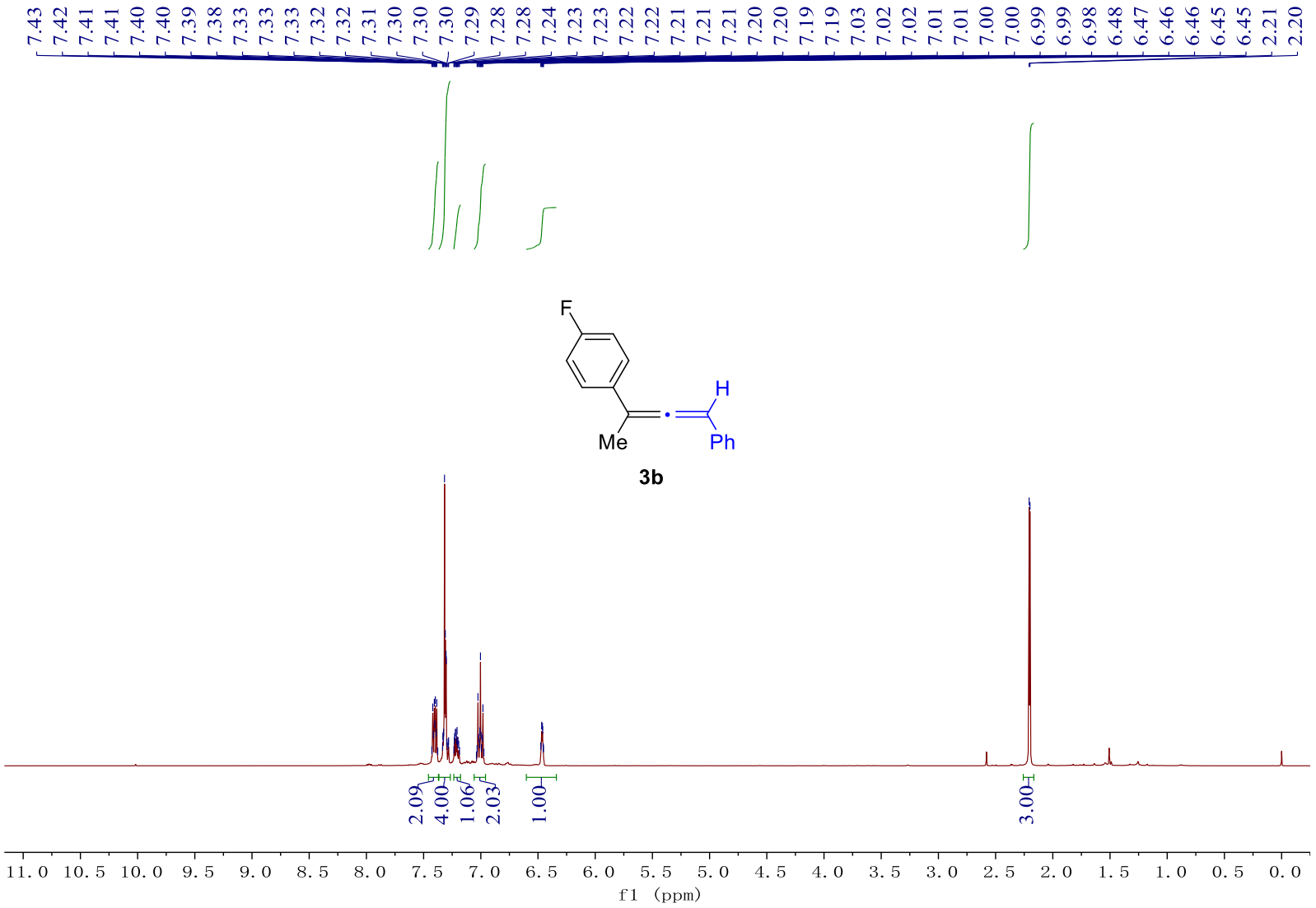
134.55
128.74
128.50
127.07
127.05
126.93
125.87
104.57
96.62
16.82
H
Me
Ph
3a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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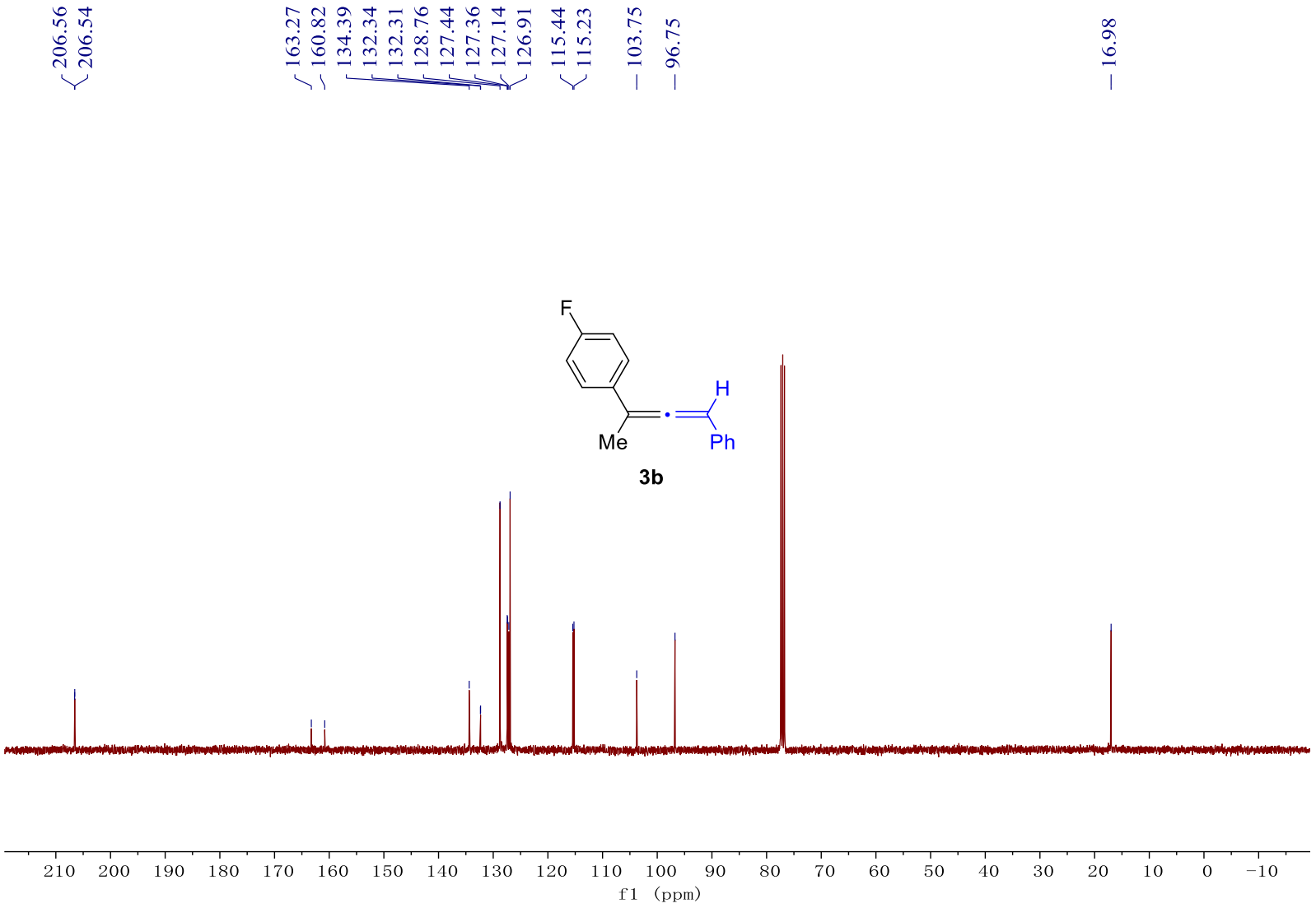
F
H
Me
Ph
2.09 4.00 1.06 2.03 1.00 3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

3b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160.82
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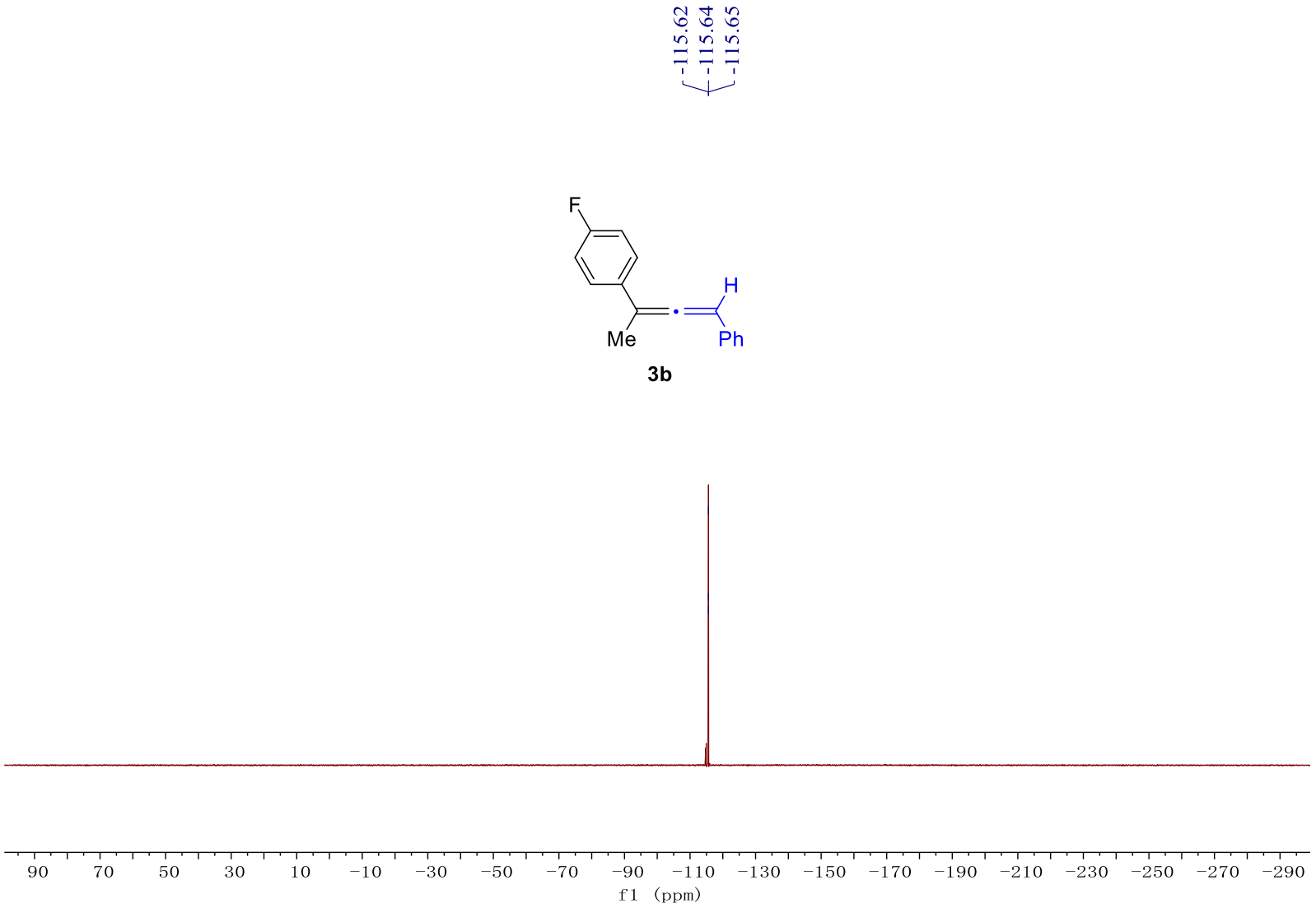
132.34
132.31
128.76
127.44
127.36
127.14
126.91
115.44
115.23
103.75
96.75
16.98
F
Me
H
Ph
3b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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-115.64
-115.65
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H
Me
Ph
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50
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−10
−30
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−210
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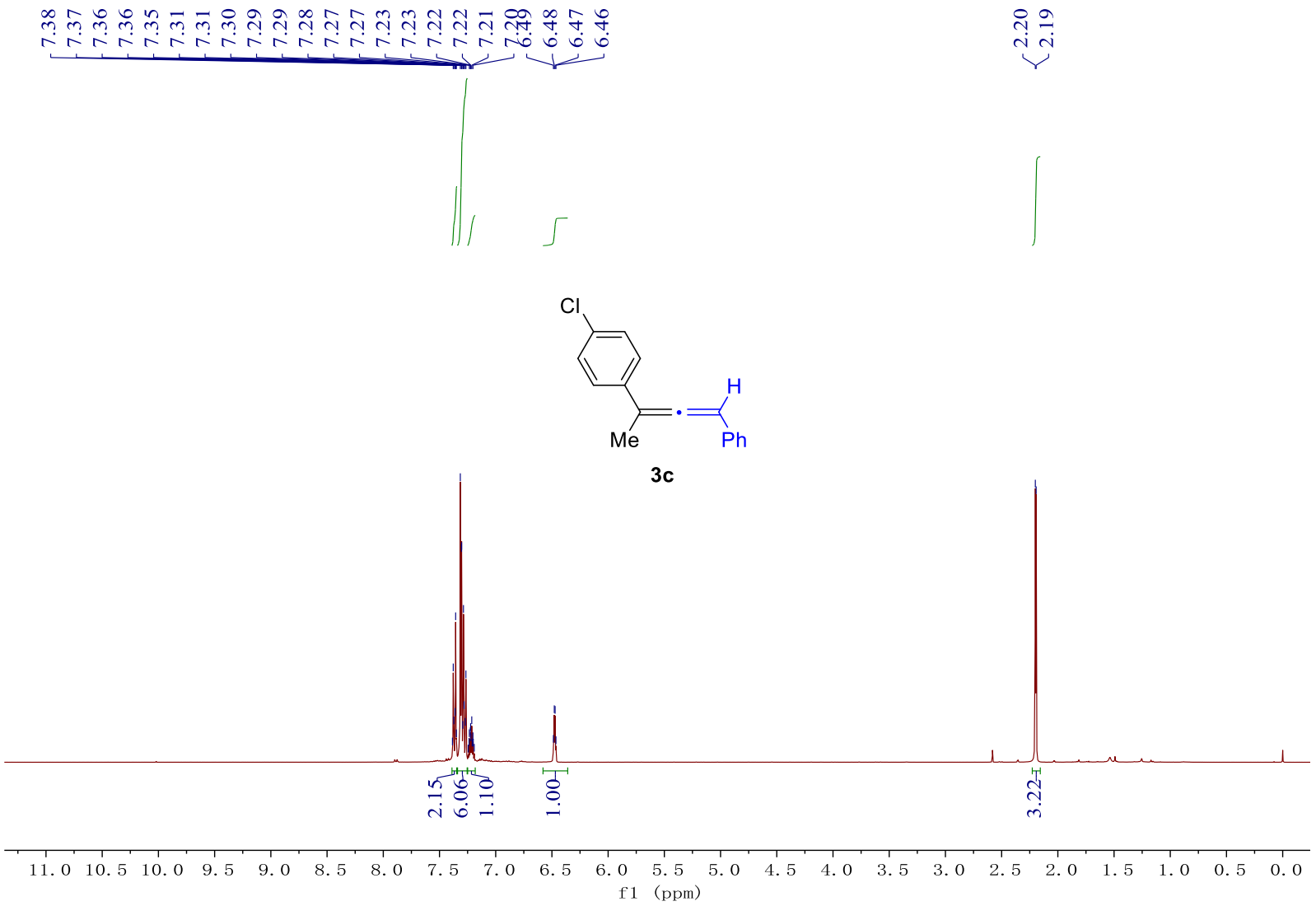
f1 (ppm)

3b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7.23
7.22
7.22
7.21
7.20
6.49
6.48
6.47
6.46
2.20
2.19
Cl
H
Me
Ph
3c
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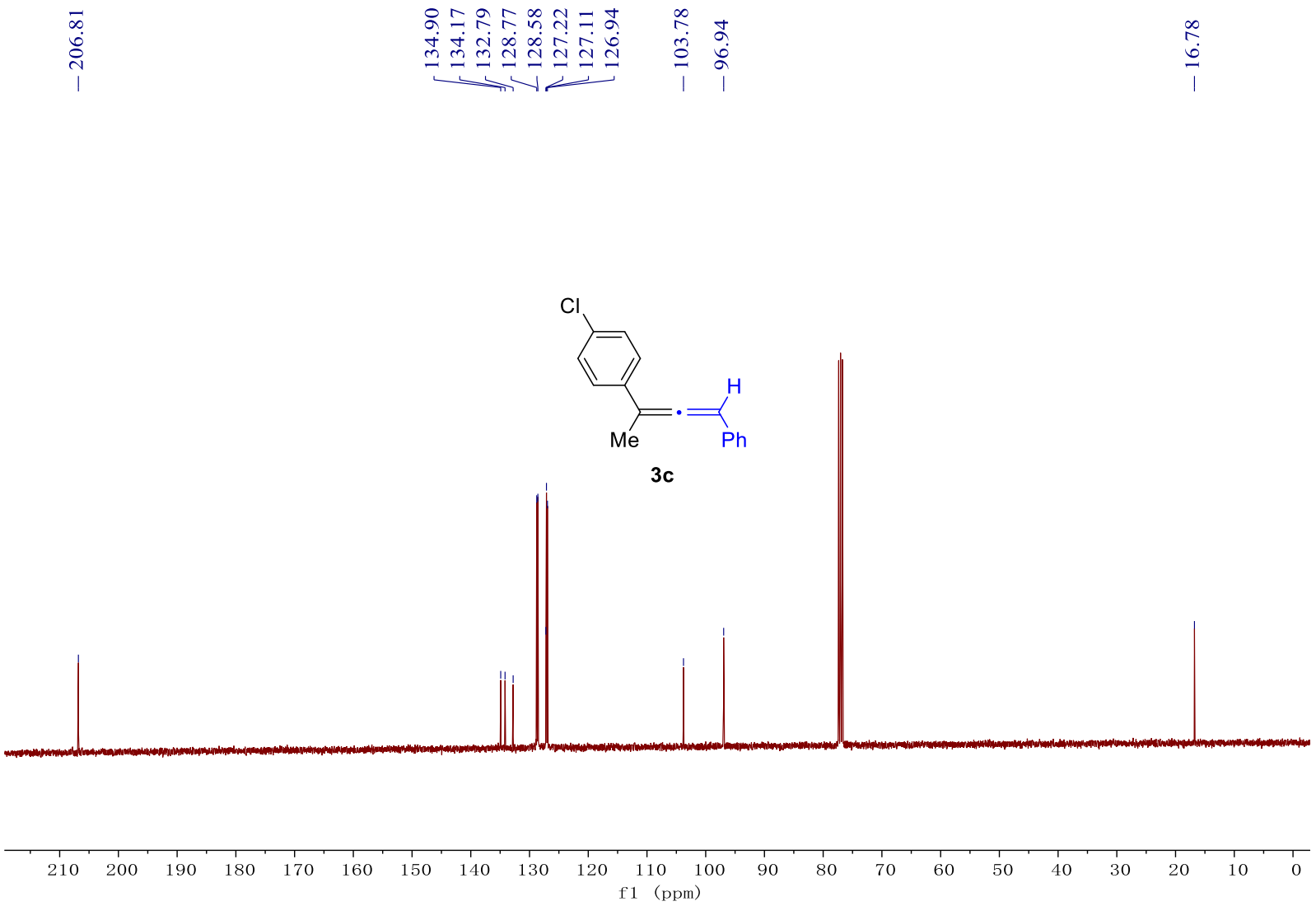
6.06
1.10
1.00
3.22
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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H
Me
Ph
3c
210
200
190
180
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130
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100
90
80
70
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50
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30
20
10
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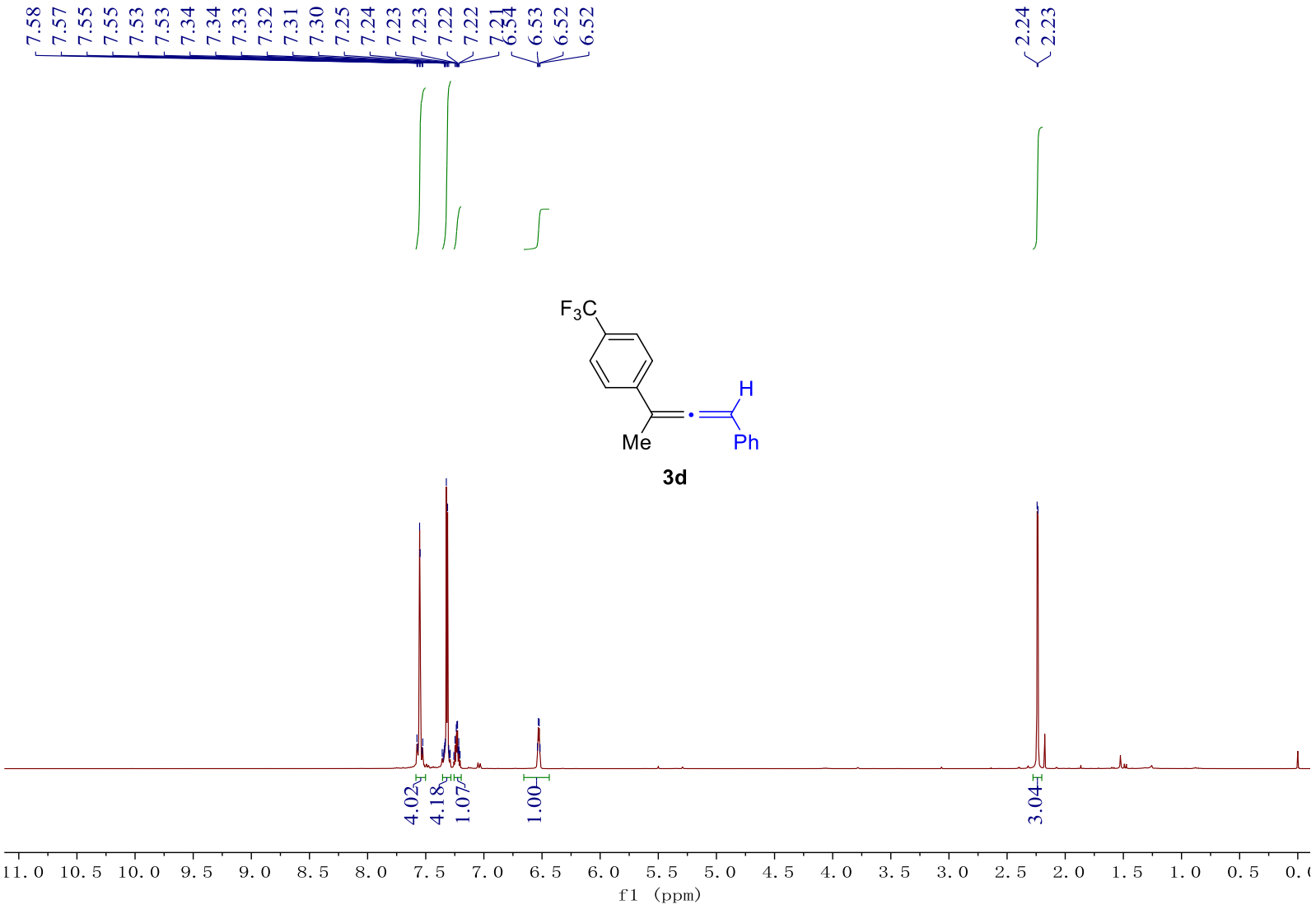
f1 (ppm)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7.22
7.21
6.54
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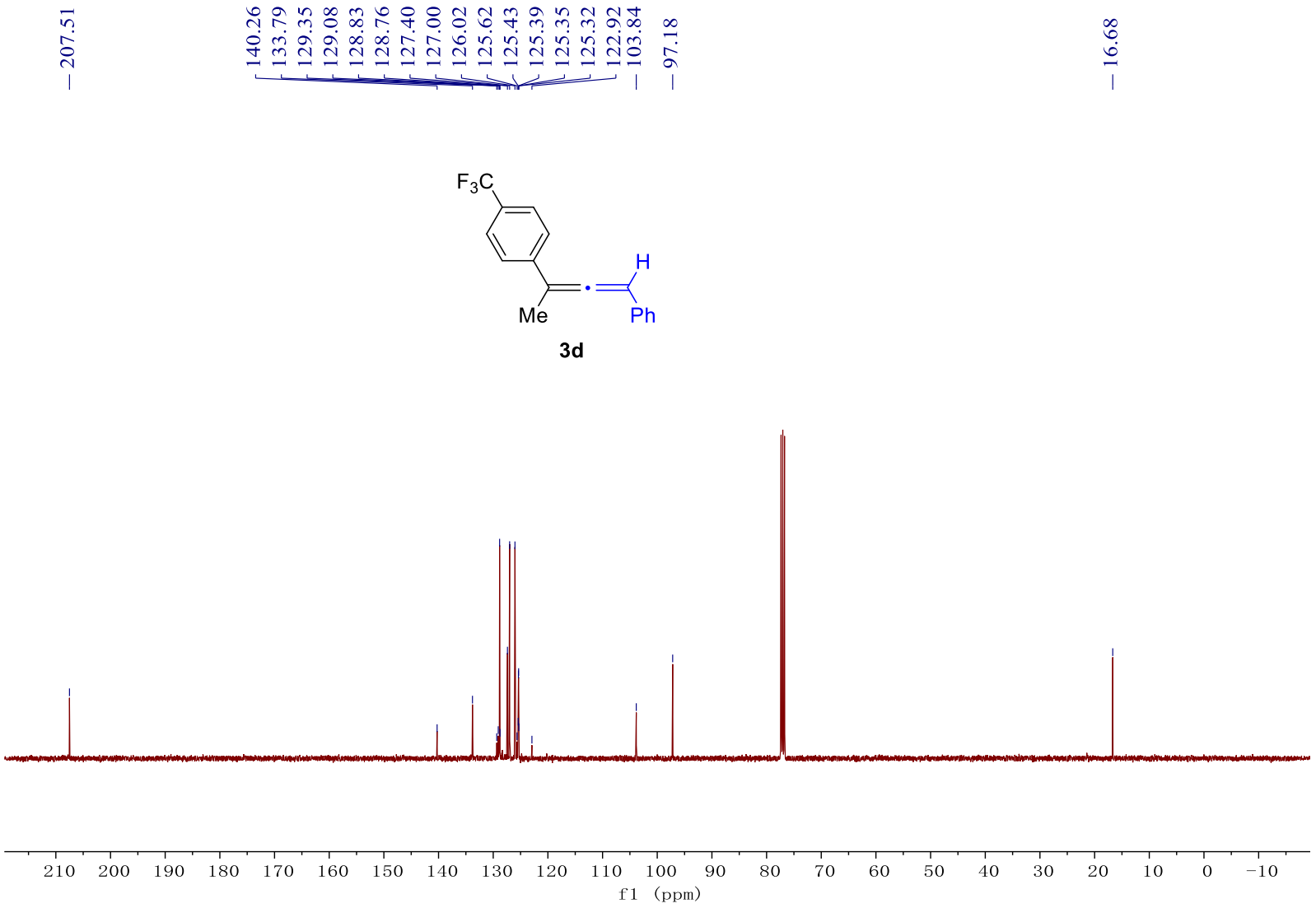
6.52
6.52
2.24
2.23
F3C
H
Me
Ph
3d
4.02
4.18
1.07
1.00
3.04
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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128.83
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126.02
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125.43
125.39
125.35
125.32
122.92
103.84
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H
Me
Ph
3d
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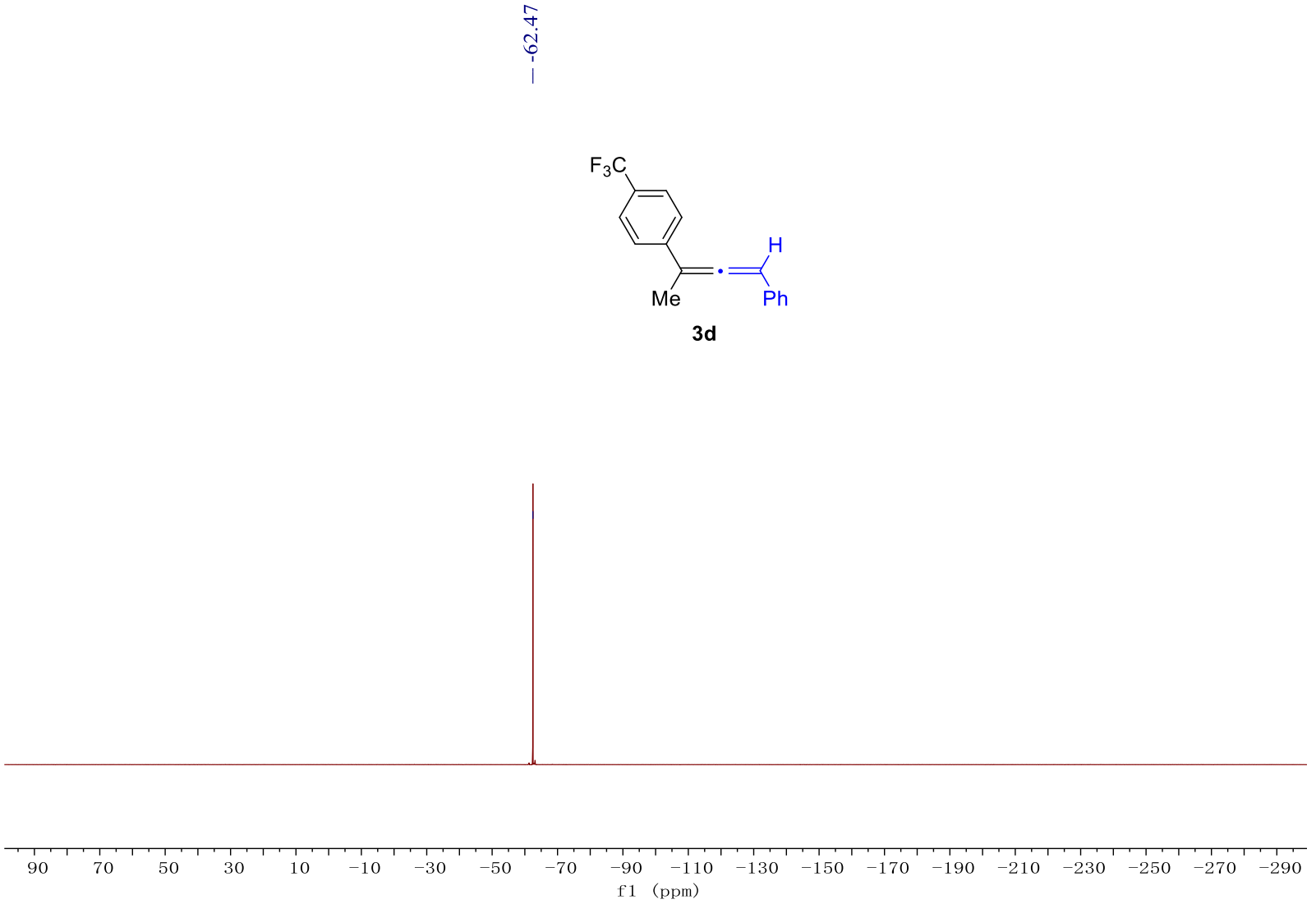
f1 (ppm)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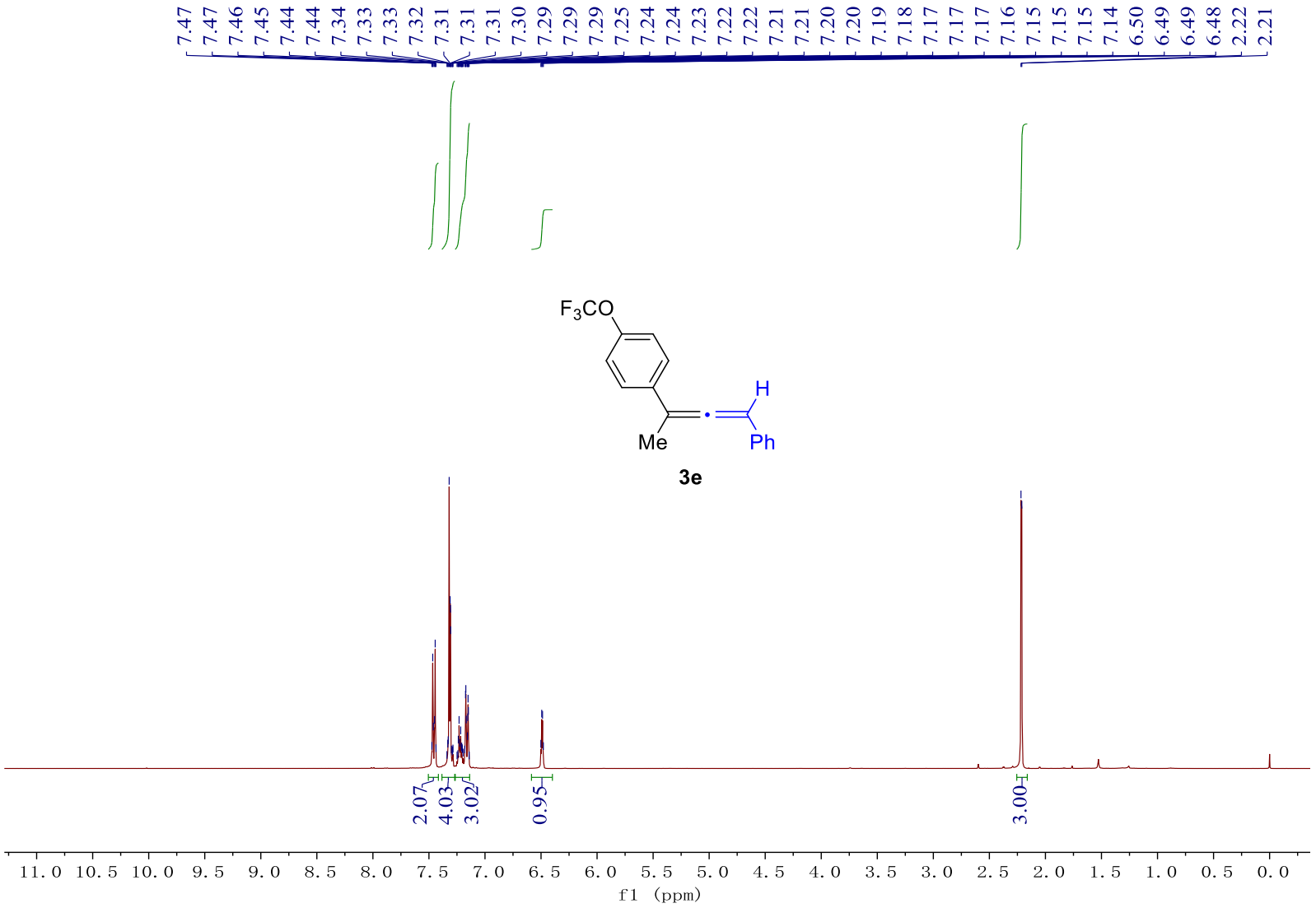
f1 (ppm)


7.47
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7.31
7.31
7.31
7.30
7.29
7.29
7.29
7.25
7.24
7.24
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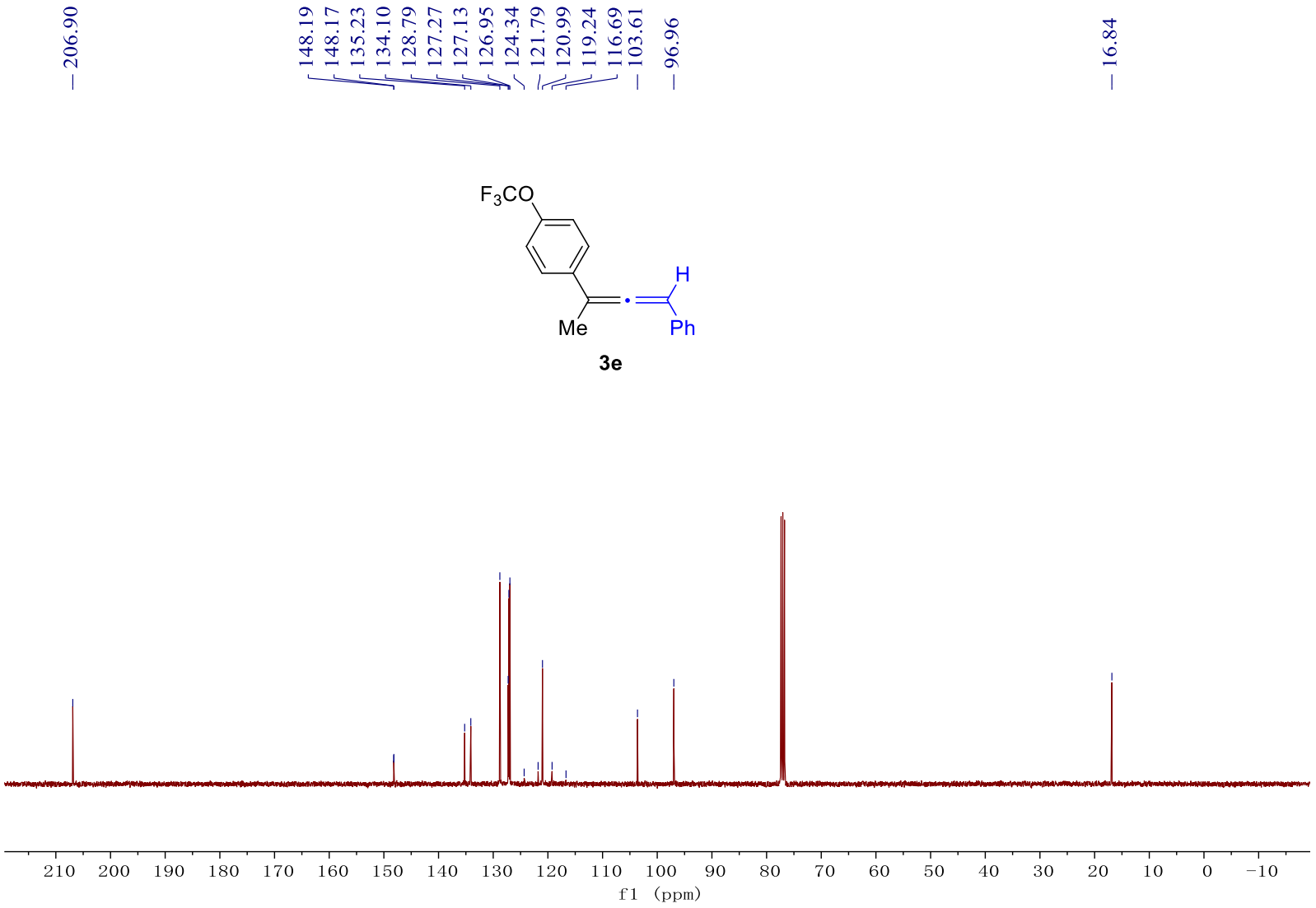
7.22
7.22
7.21
7.21
7.20
7.20
7.19
7.18
7.17
7.17
7.17
7.16
7.15
7.15
7.15
7.14
6.50
6.49
6.49
6.48
2.22
2.21
F3CO
H
Me
Ph
3e
2.07
4.03
3.02
0.95
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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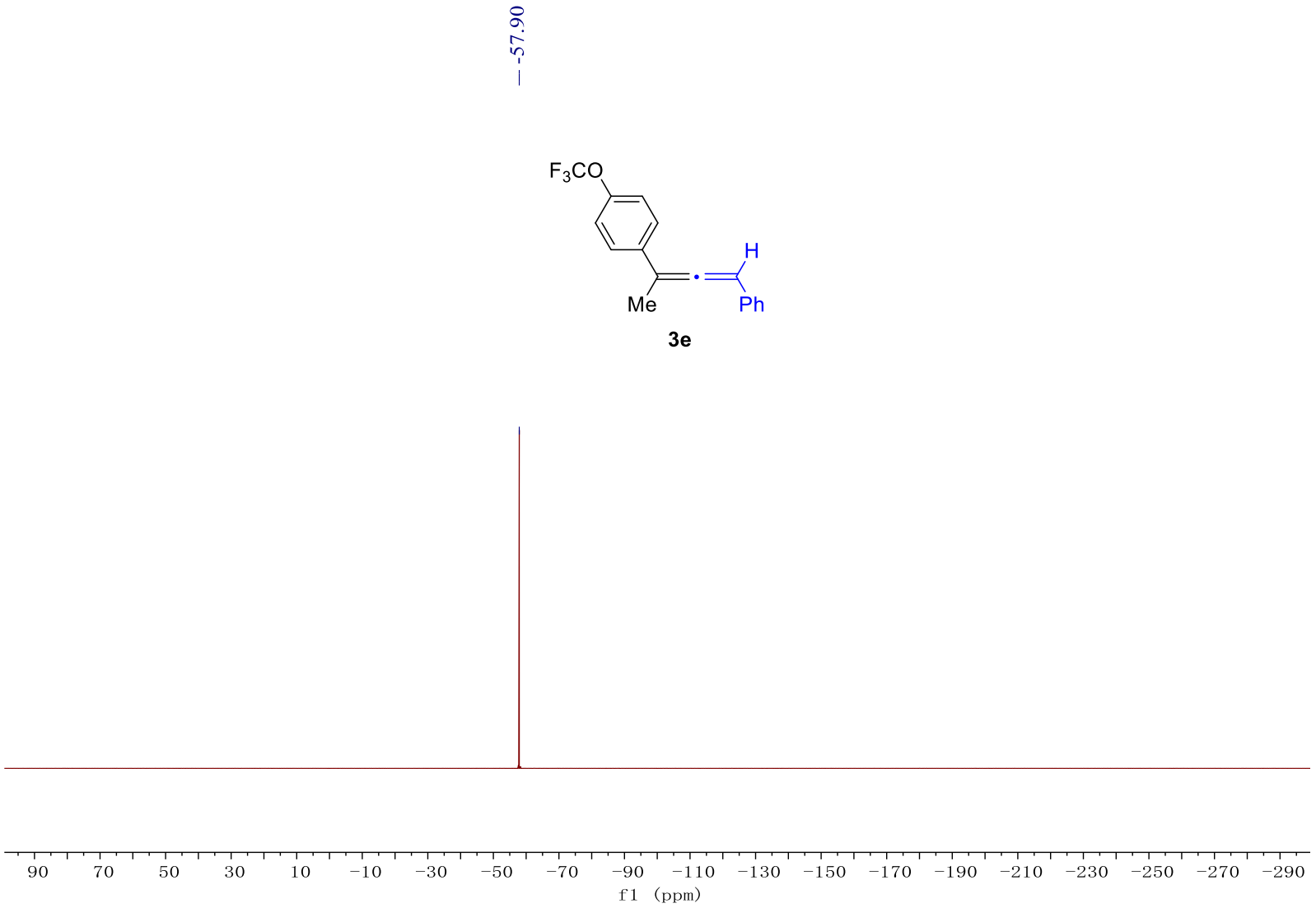
120.99
119.24
116.69
103.61
96.96
16.84
F3CO
H
Me
Ph
3e
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)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Ph
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10
−10
−30
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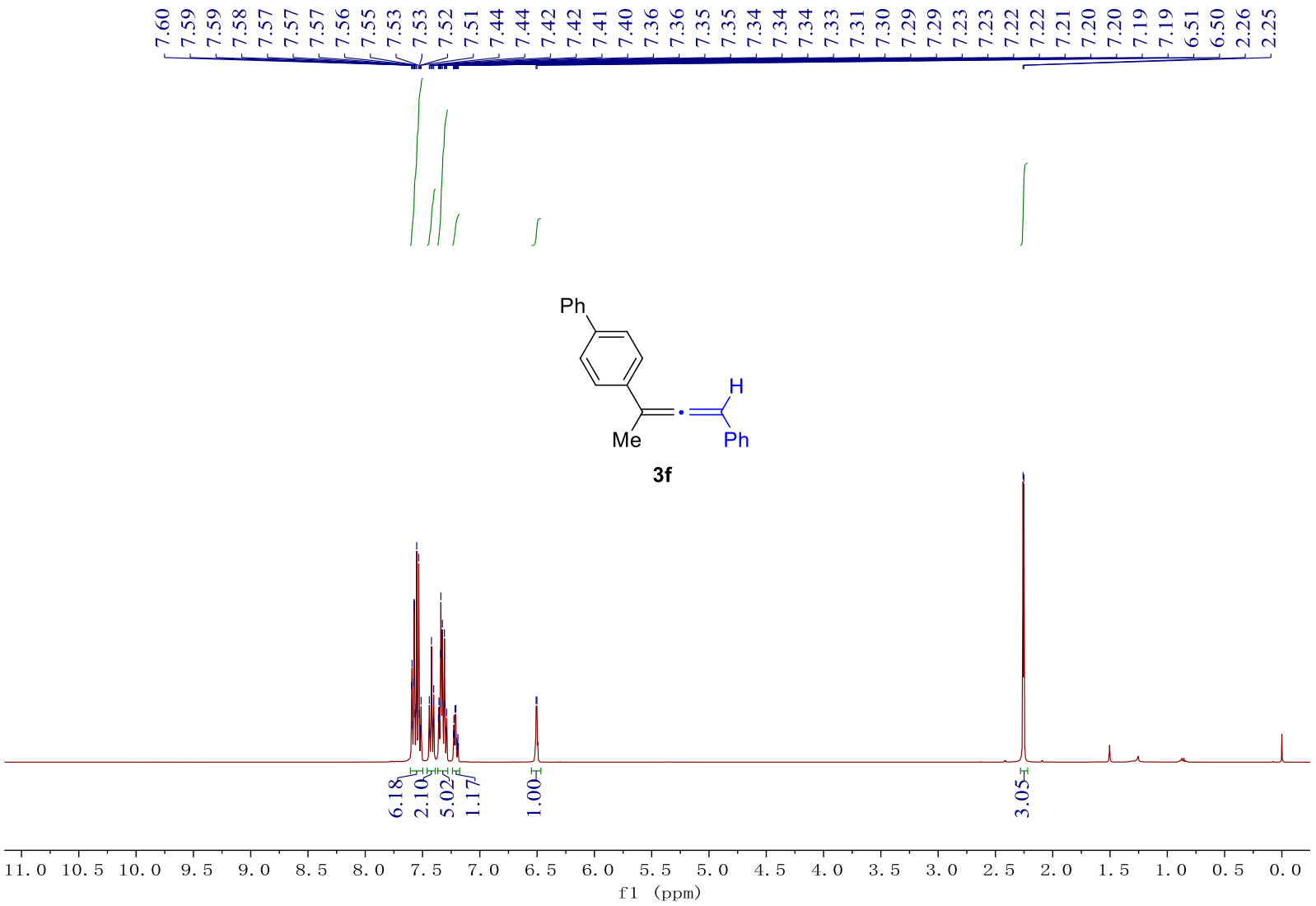
−130
−150
−170
−190
−210
−230
−250
−270
−290
f1 (ppm)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7.44
7.42
7.42
7.41
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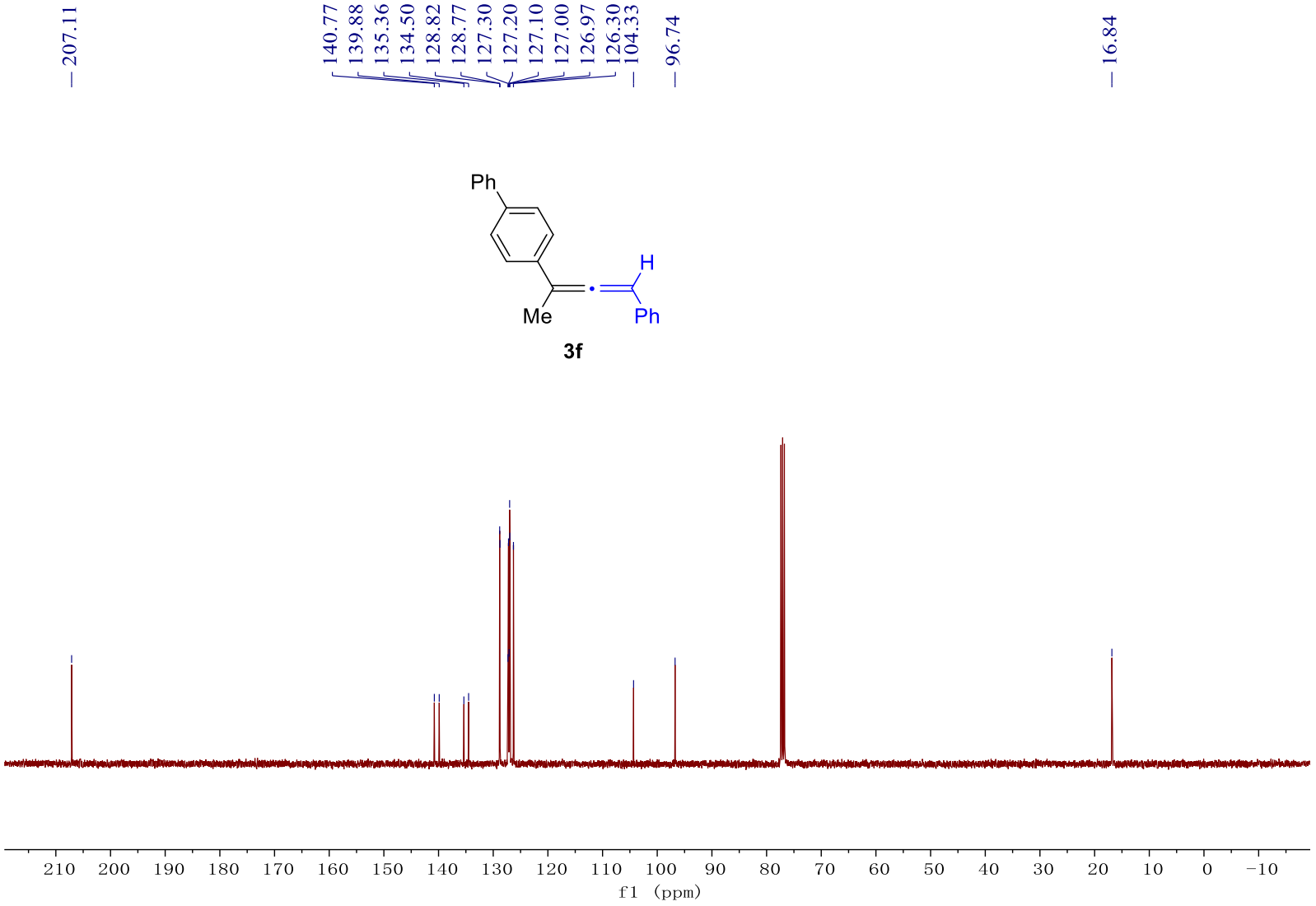
7.34
7.34
7.33
7.31
7.30
7.29
7.29
7.23
7.23
7.22
7.22
7.21
7.20
7.20
7.19
7.19
6.51
6.50
2.26
2.25
Ph
H
Me
Ph
3f
6.18
2.10
5.02
1.17
1.00
3.05
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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127.00
126.97
126.30
104.33
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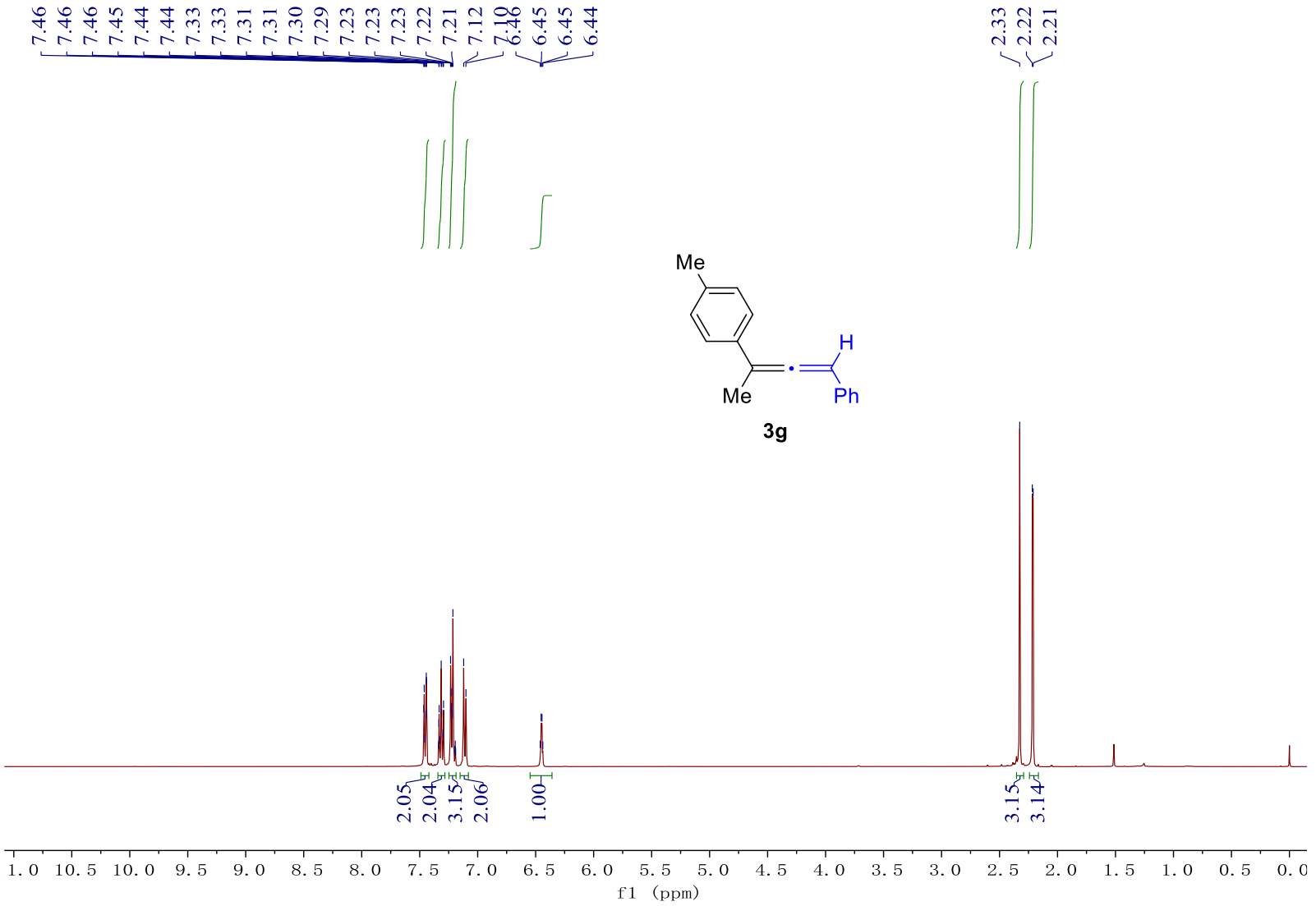
Ph
H
Me
Ph
3f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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7.33
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7.29
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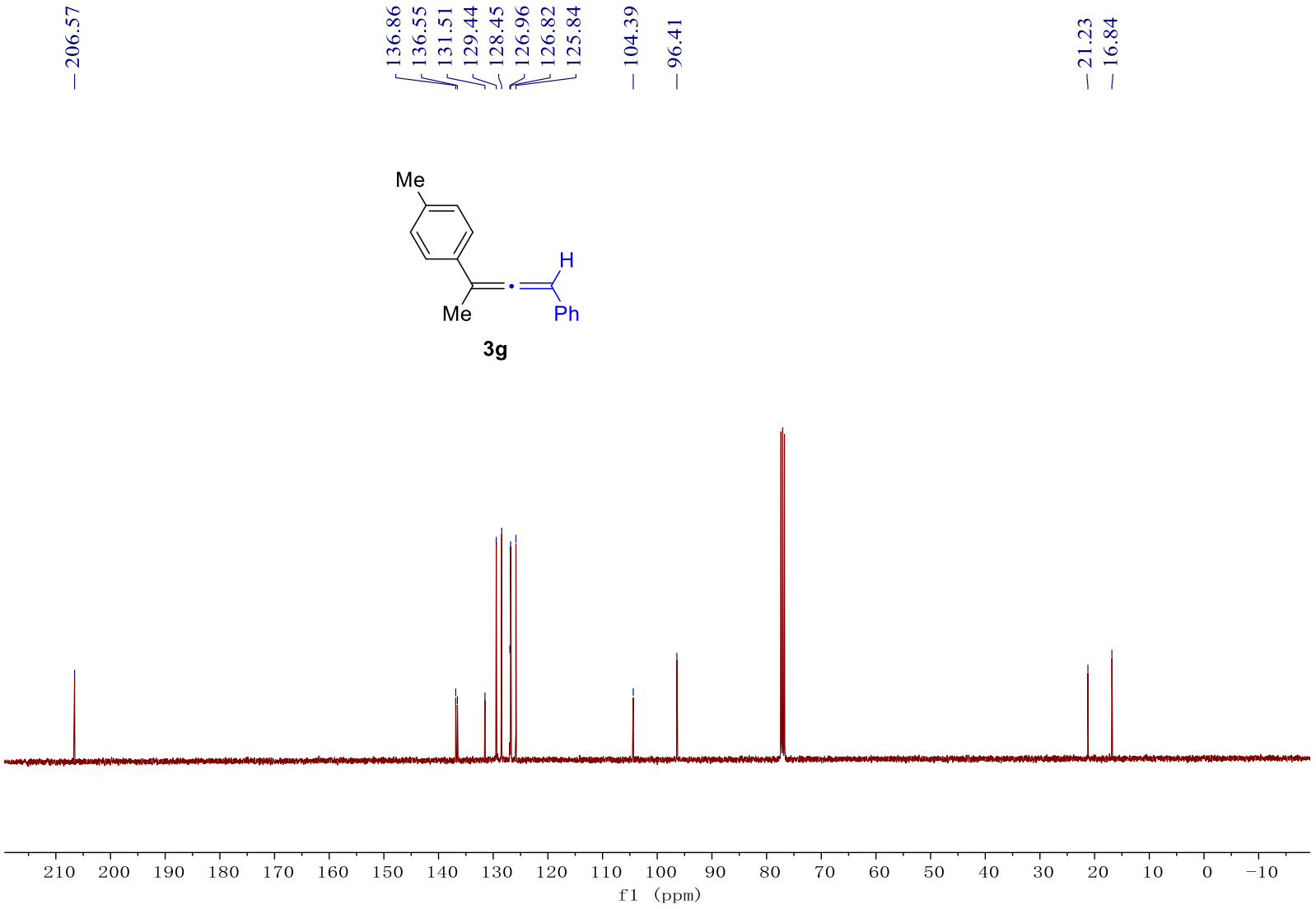
7.23
7.23
7.22
7.21
7.12
7.10
6.46
6.45
6.45
6.44
2.33
2.22
2.21
Me
H
Me
Ph
3g
2.05
2.04
3.15
2.06
1.00
3.15
3.14
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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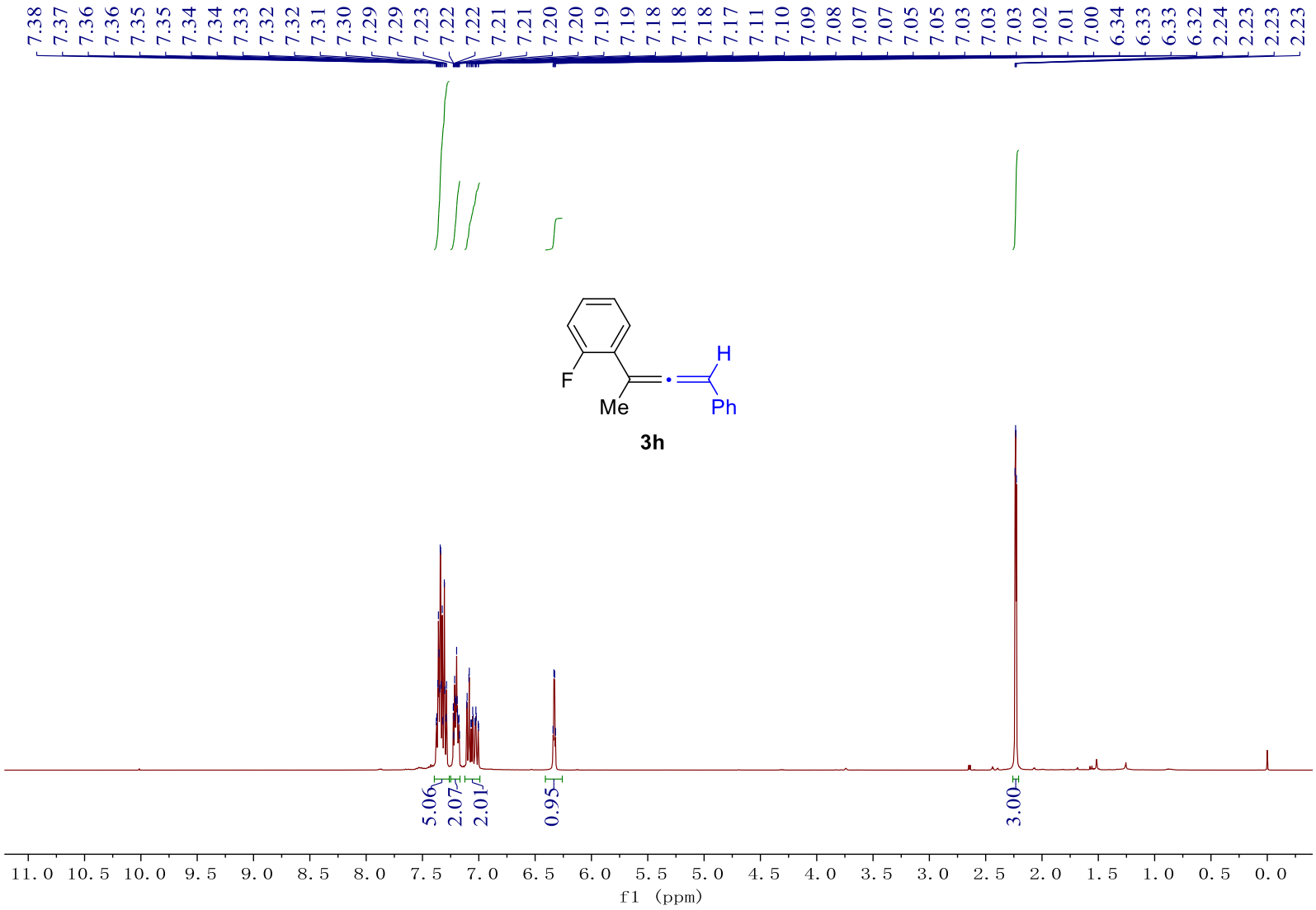
126.96
126.82
125.84
104.39
96.41
21.23
16.84
Me
Me
H
Ph
3g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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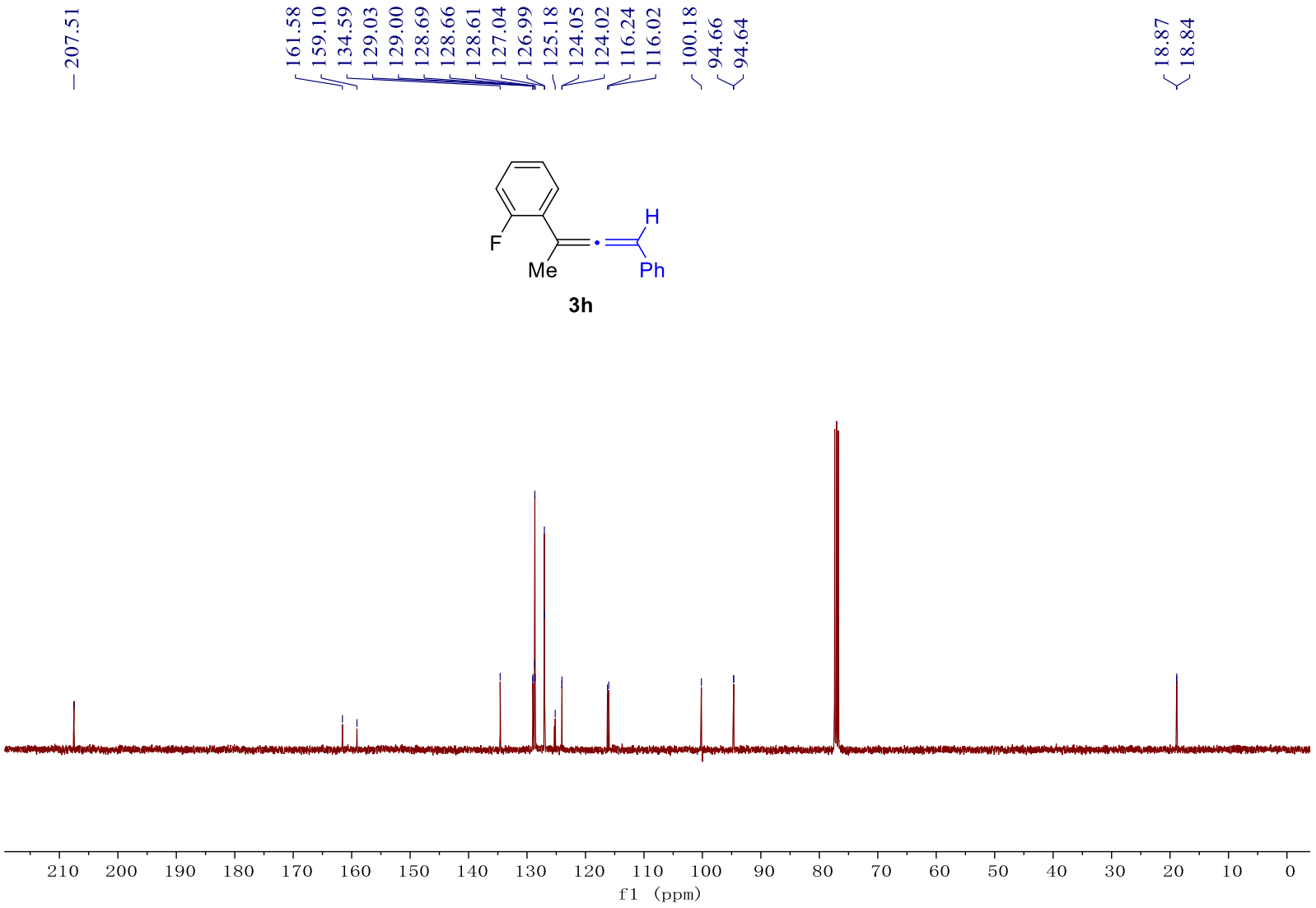
F
Me
H
Ph
3h
5.06
2.07
2.01
0.95
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.66
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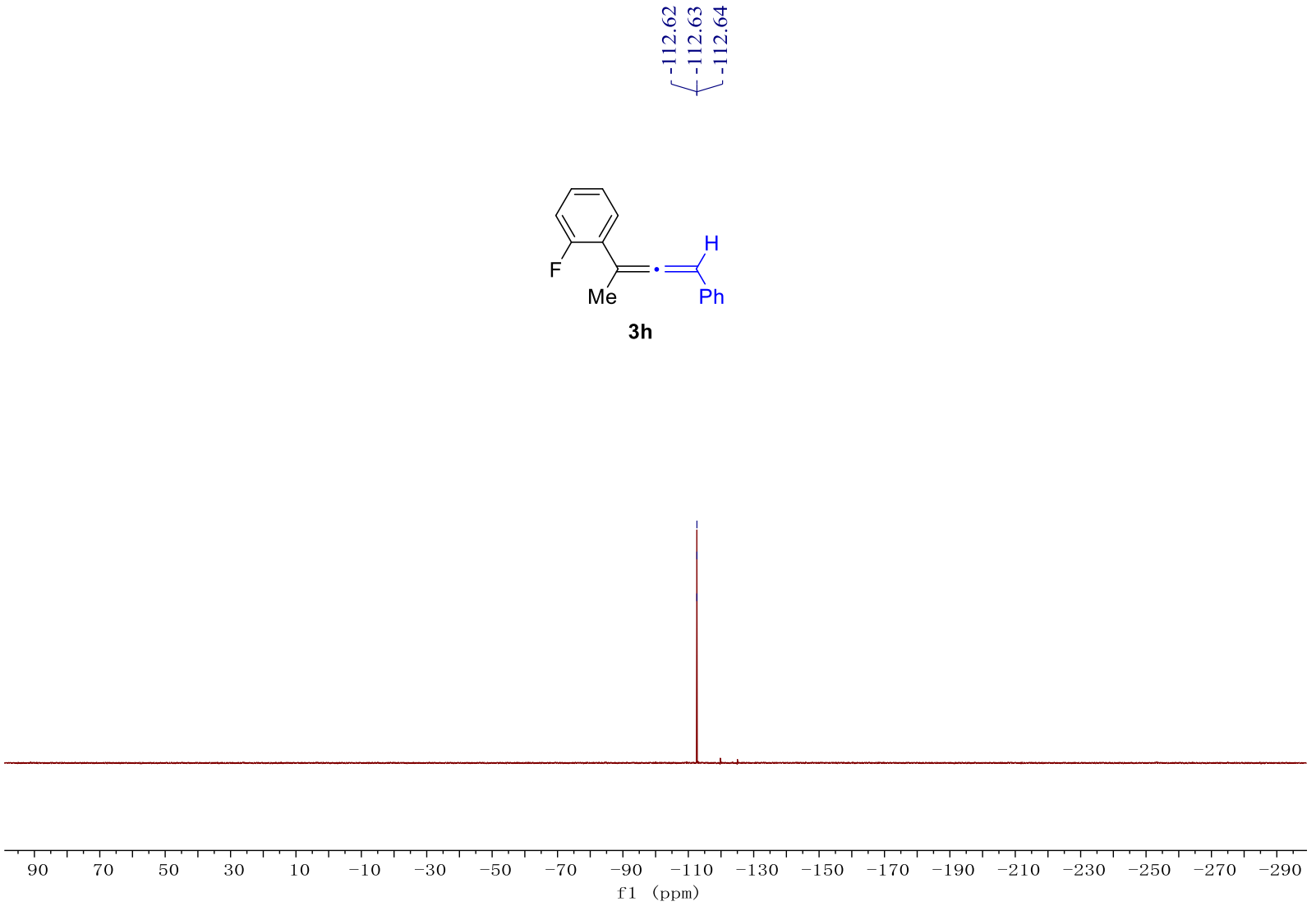
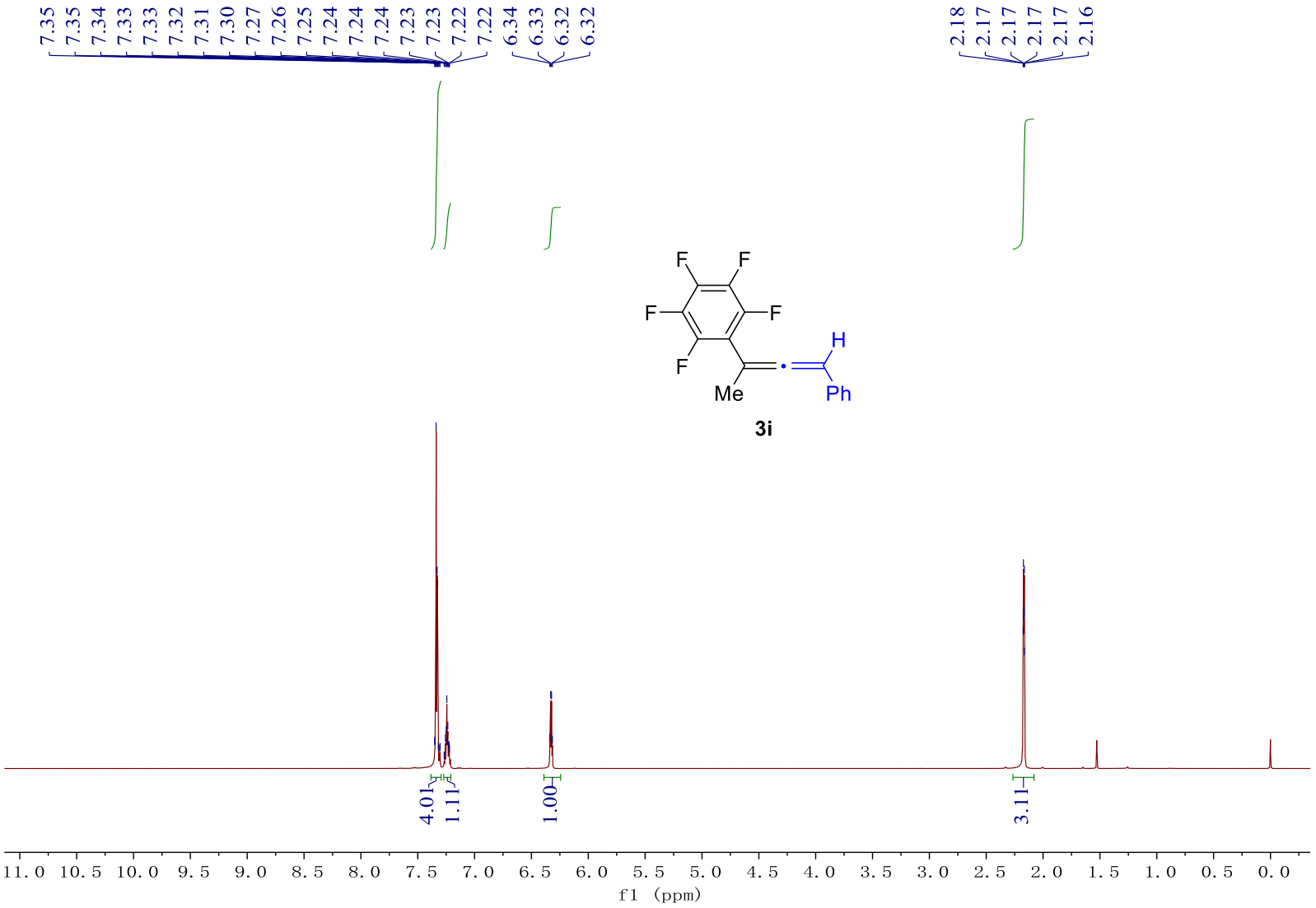
127.04
126.99
125.18
124.05
124.02
116.24
116.02
100.18
94.66
94.64
18.87
18.84
F
Me
H
Ph
3h
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-112.62
-112.63
-112.64
F
Me
H
Ph
90
70
50
30
10
−10
−30
−50
−70
−90
−110
−130
−150
−170
−190
−210
−230
−250
−270
−290
f1 (ppm)

3h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7.35
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7.33
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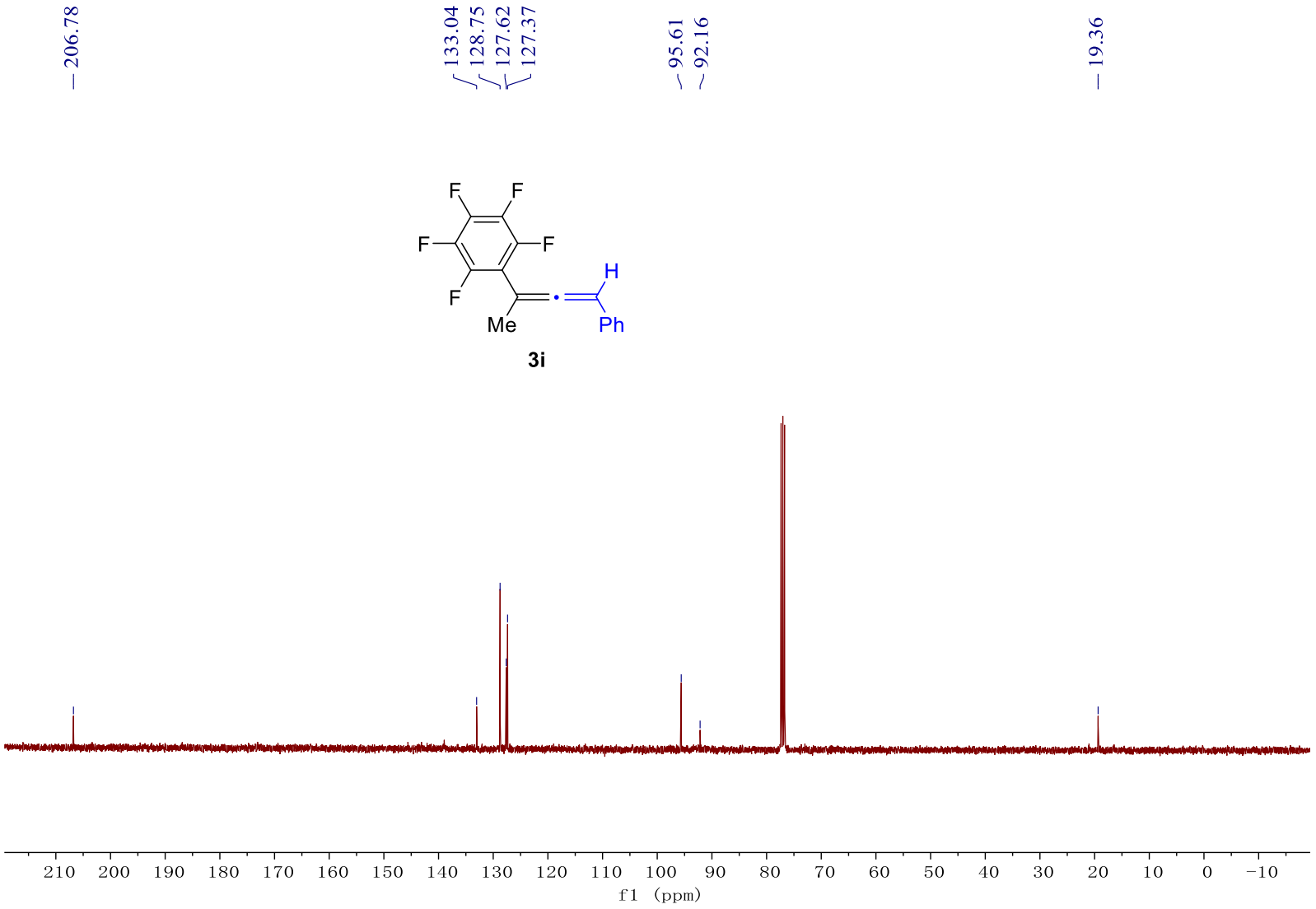
7.32
7.31
7.30
7.27
7.26
7.25
7.24
7.24
7.24
7.23
7.23
7.22
7.22
6.34
6.33
6.32
6.32
2.18
2.17
2.17
2.17
2.17
2.16
F
F
F
F
F
Me
H
Ph
3i
4.01
1.11
1.00
3.11
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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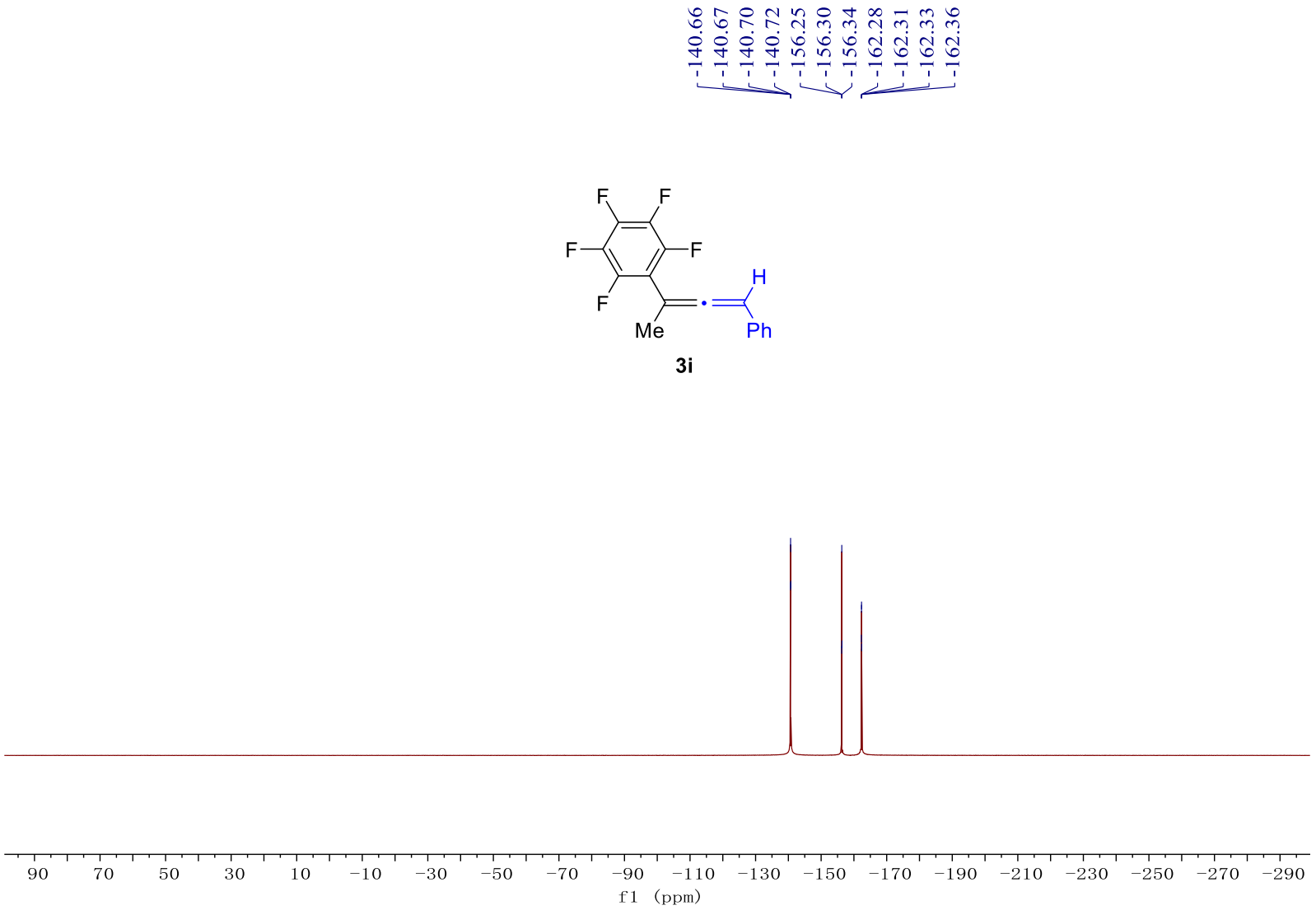
127.37
95.61
92.16
19.36
F
F
F
F
F
H
Me
Ph
3i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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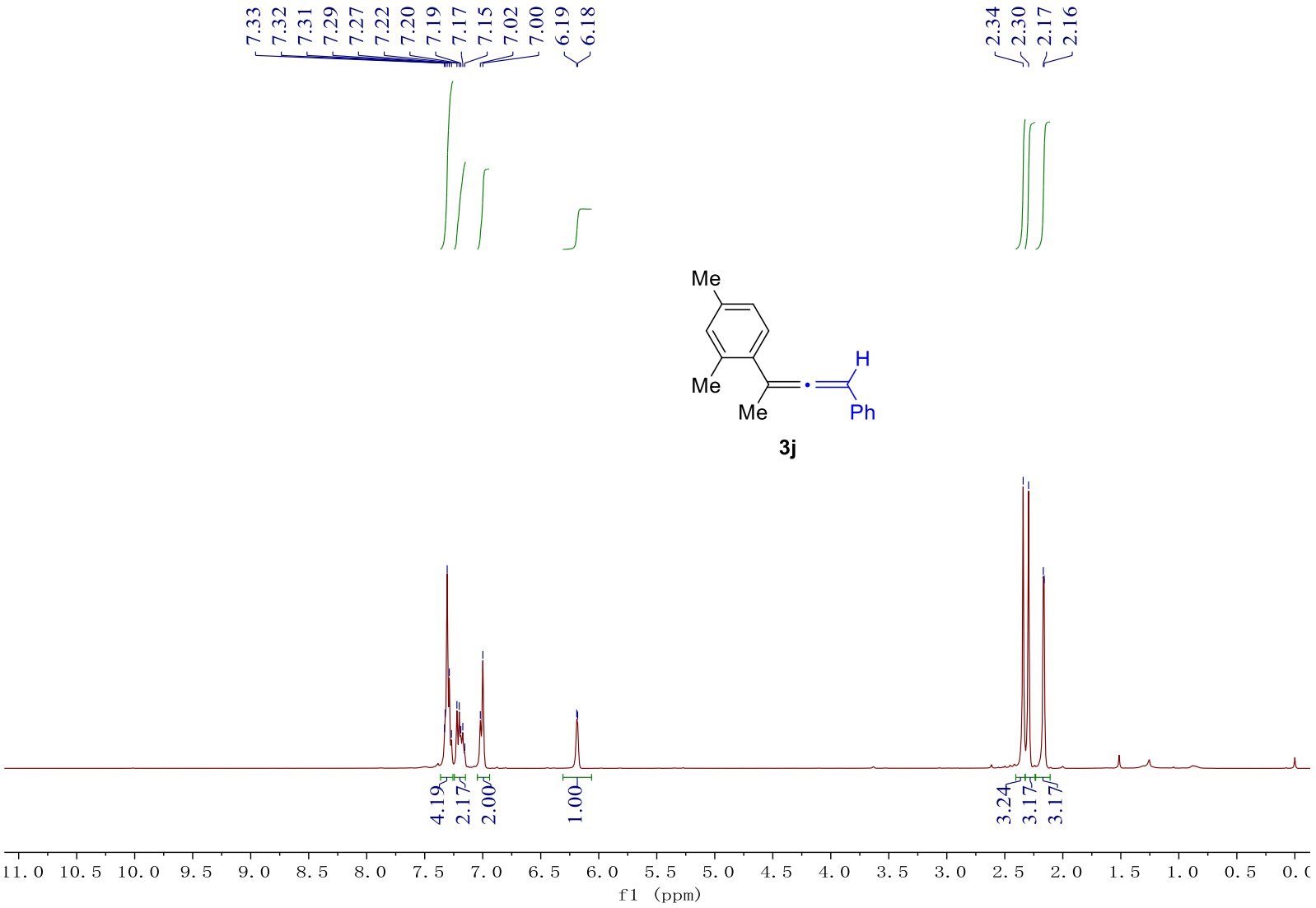
-140.67
-140.70
-140.72
-156.25
-156.30
-156.34
-162.28
-162.31
-162.33
-162.36
F
F
F
F
F
Me
H
Ph
90
70
50
30
10
−10
−30
−50
−70
−90
−110
−130
−150
−170
−190
−210
−230
−250
−270
−290
f1 (ppm)

3i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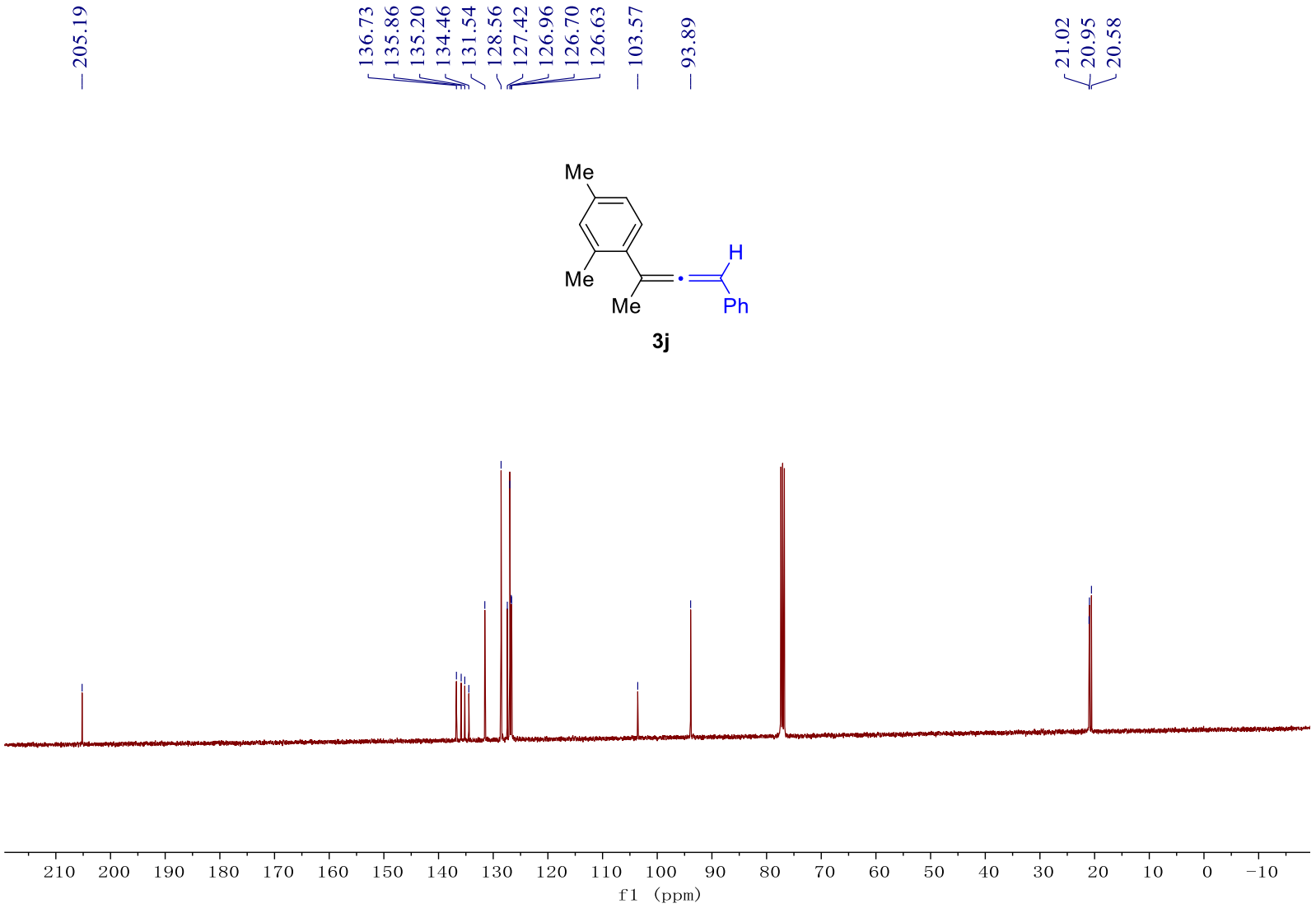
7.22
7.20
7.19
7.17
7.15
7.02
7.00
6.19
6.18
2.34
2.30
2.17
2.16
Me
Me
Me
H
Ph
3j
4.19
2.17
2.00
1.00
3.24
3.17
3.17
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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126.70
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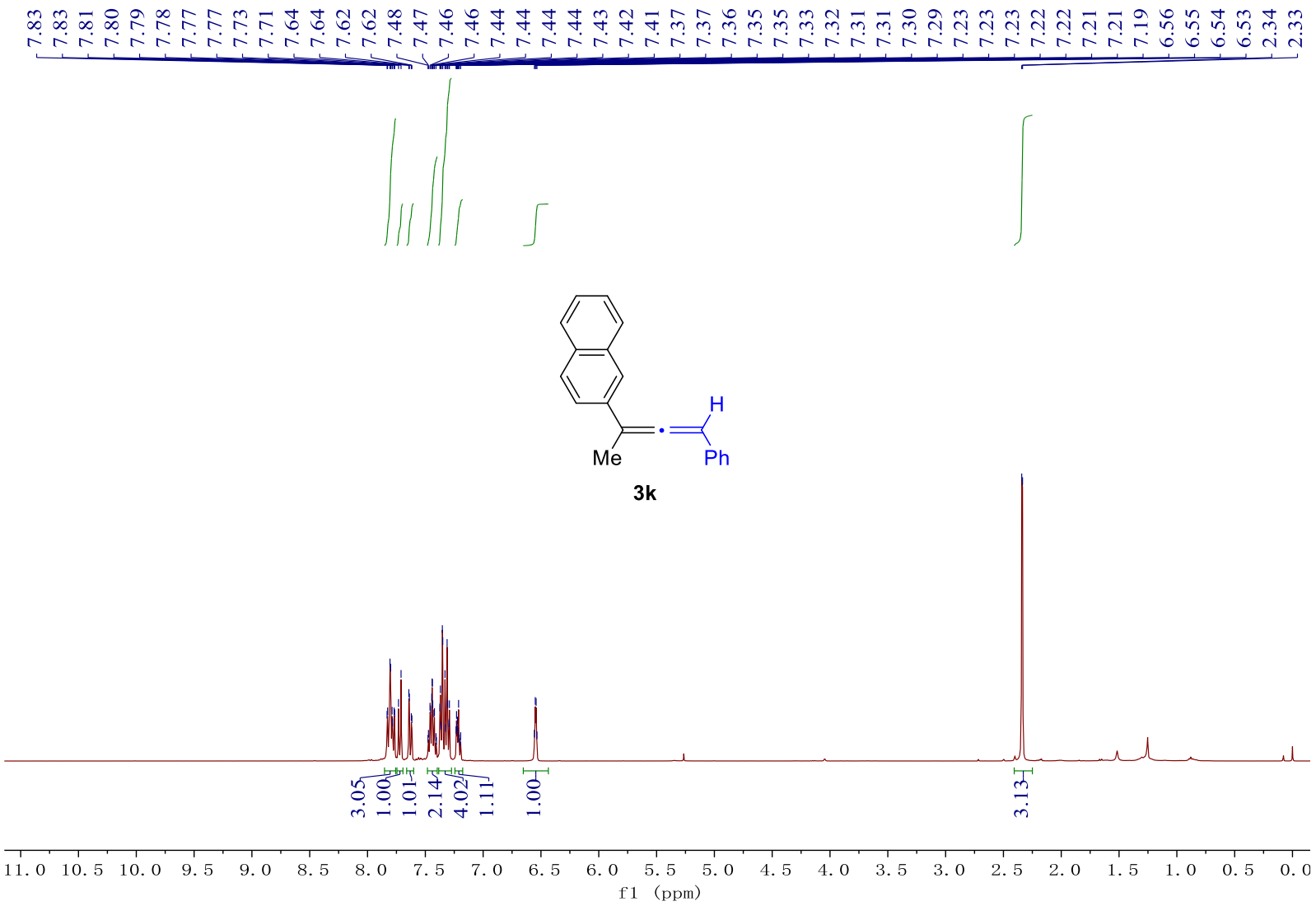
103.57
93.89
21.02
20.95
20.58
Me
Me
Me
H
Ph
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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7.21
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6.56
6.55
6.54
6.53
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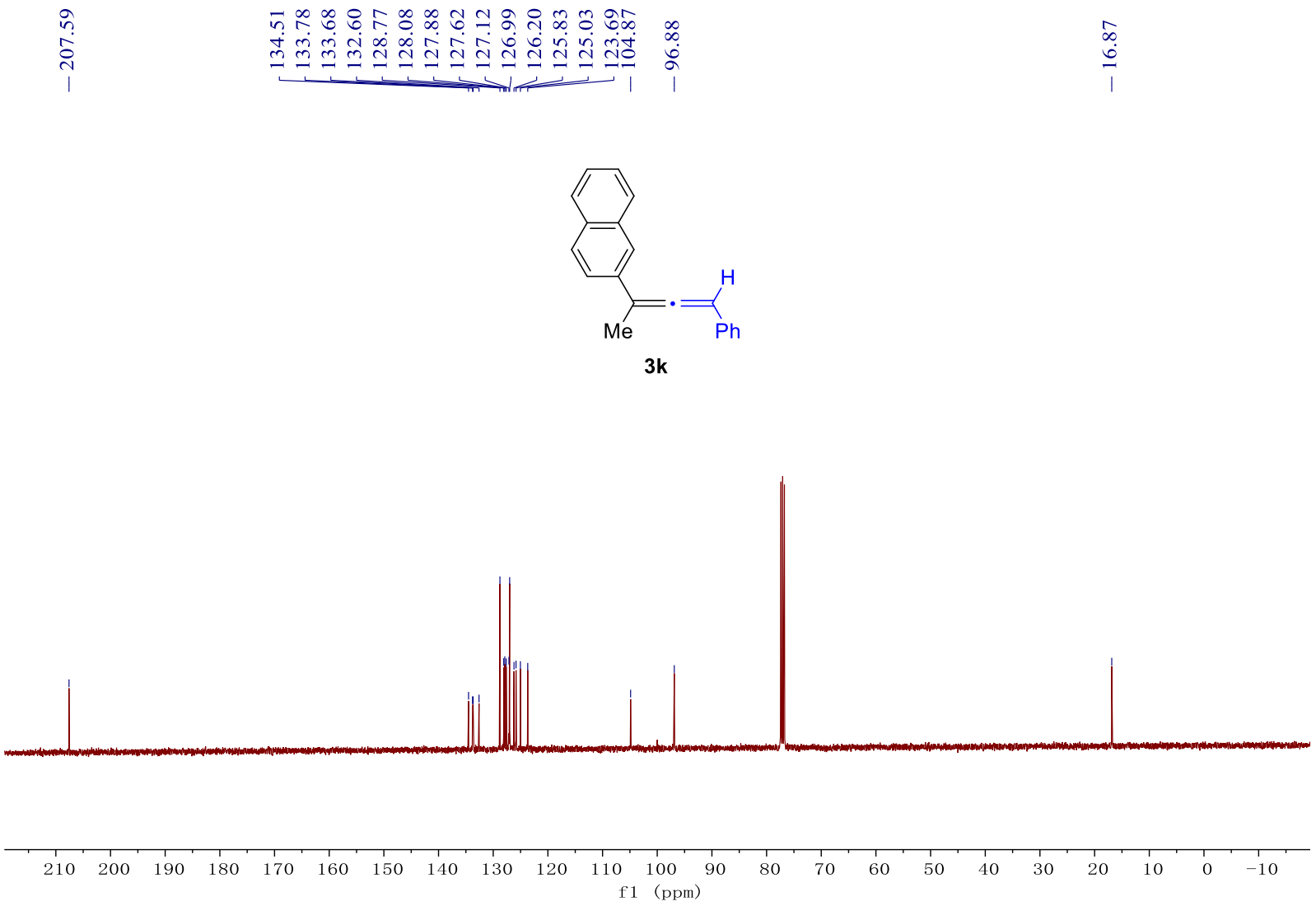
2.33
H
Me
Ph
3k
3.05
1.00
1.01
2.14
4.02
1.11
1.00
3.13
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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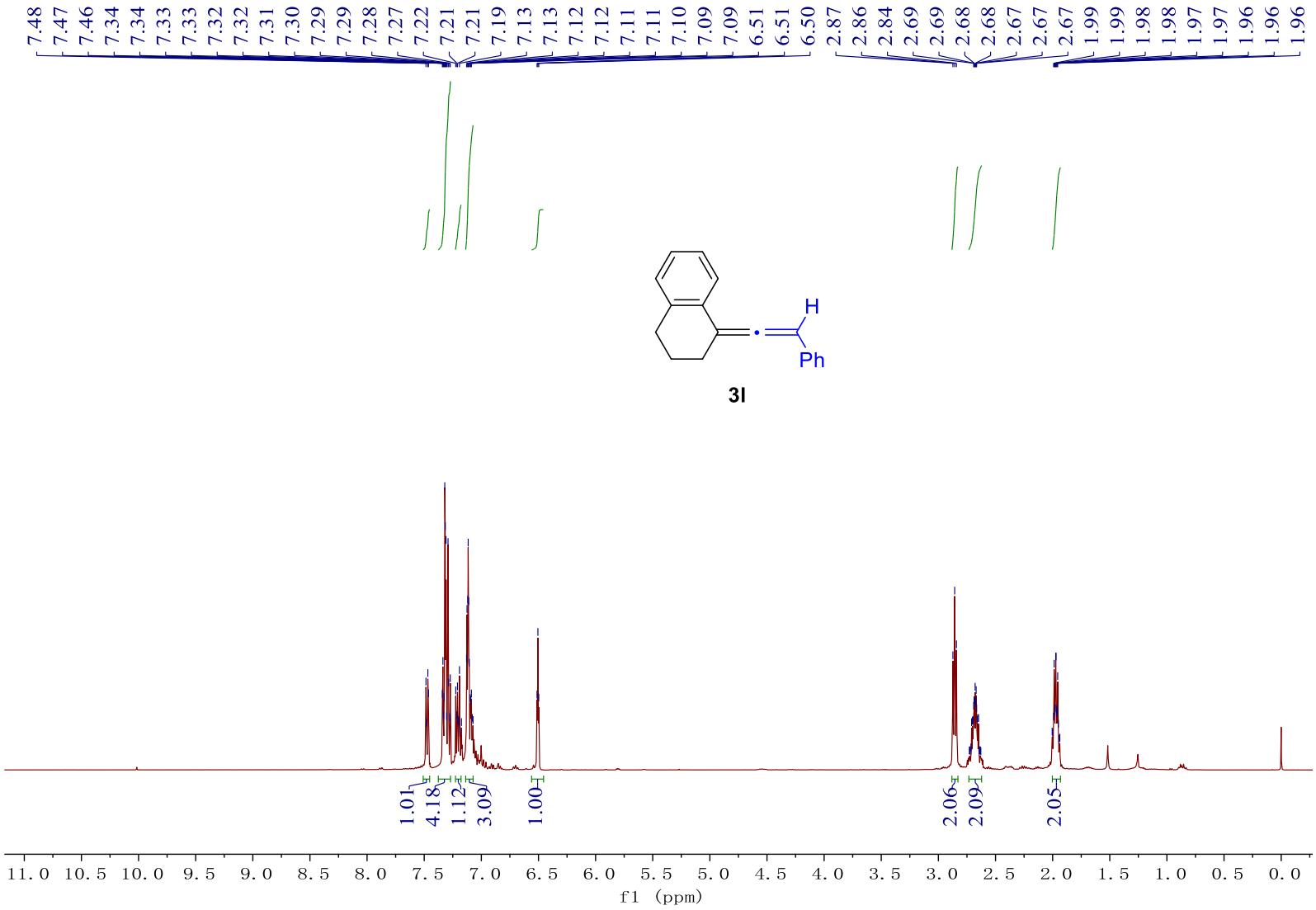
126.99
126.20
125.83
125.03
123.69
104.87
96.88
16.87
H
Me
Ph
3k
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)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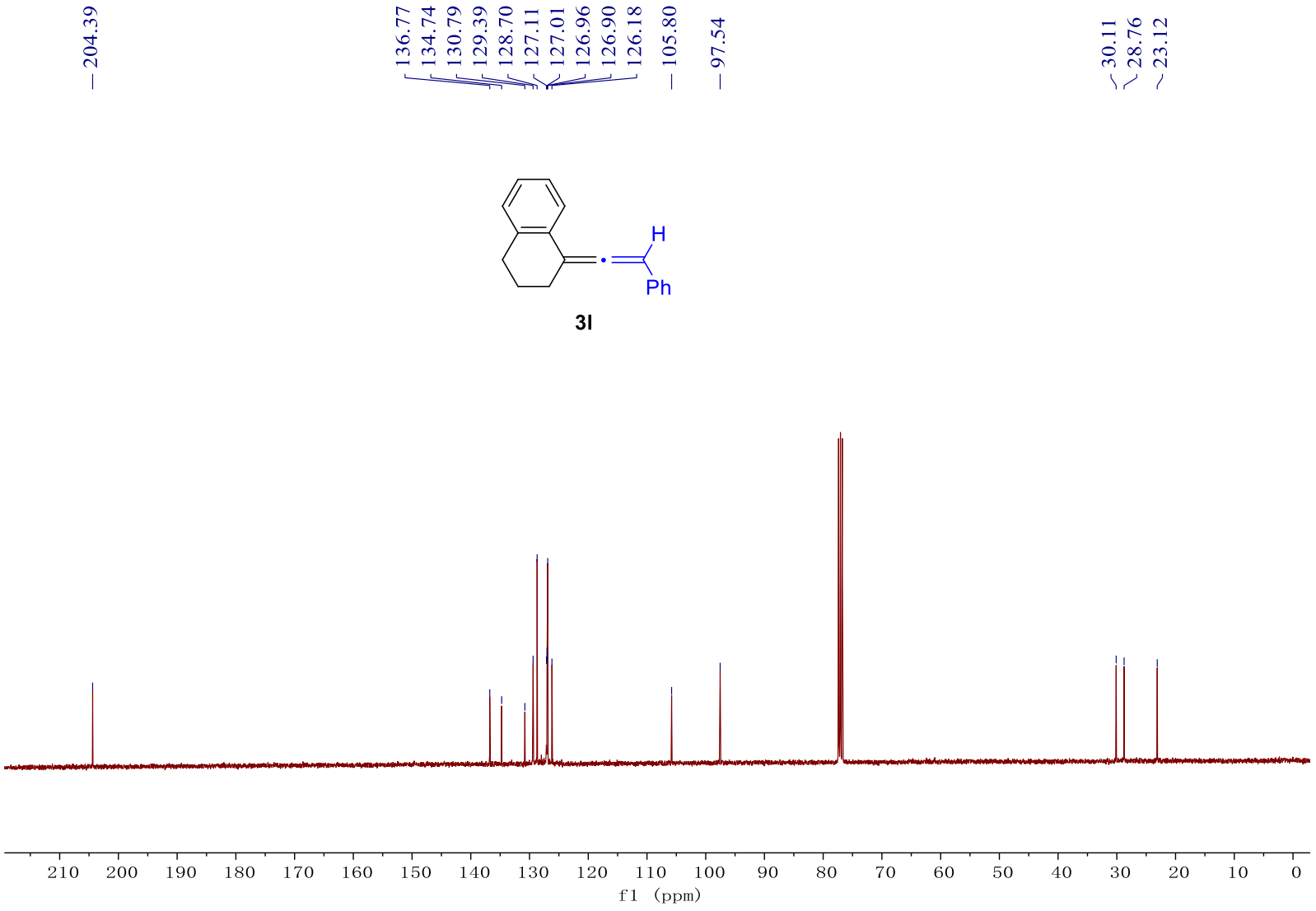
2.87 2.86 2.84 2.69 2.69 2.68 2.68 2.67 2.67 2.67 1.99 1.99 1.98 1.98 1.97 1.97 1.96 1.96 1.96
H
Ph
3l
1.01 4.18 1.12 3.09 1.00 2.06 2.09 2.05
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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126.18
105.80
97.54
30.11
28.76
23.12
H
Ph
210
200
190
180
170
160
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140
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110
100
90
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70
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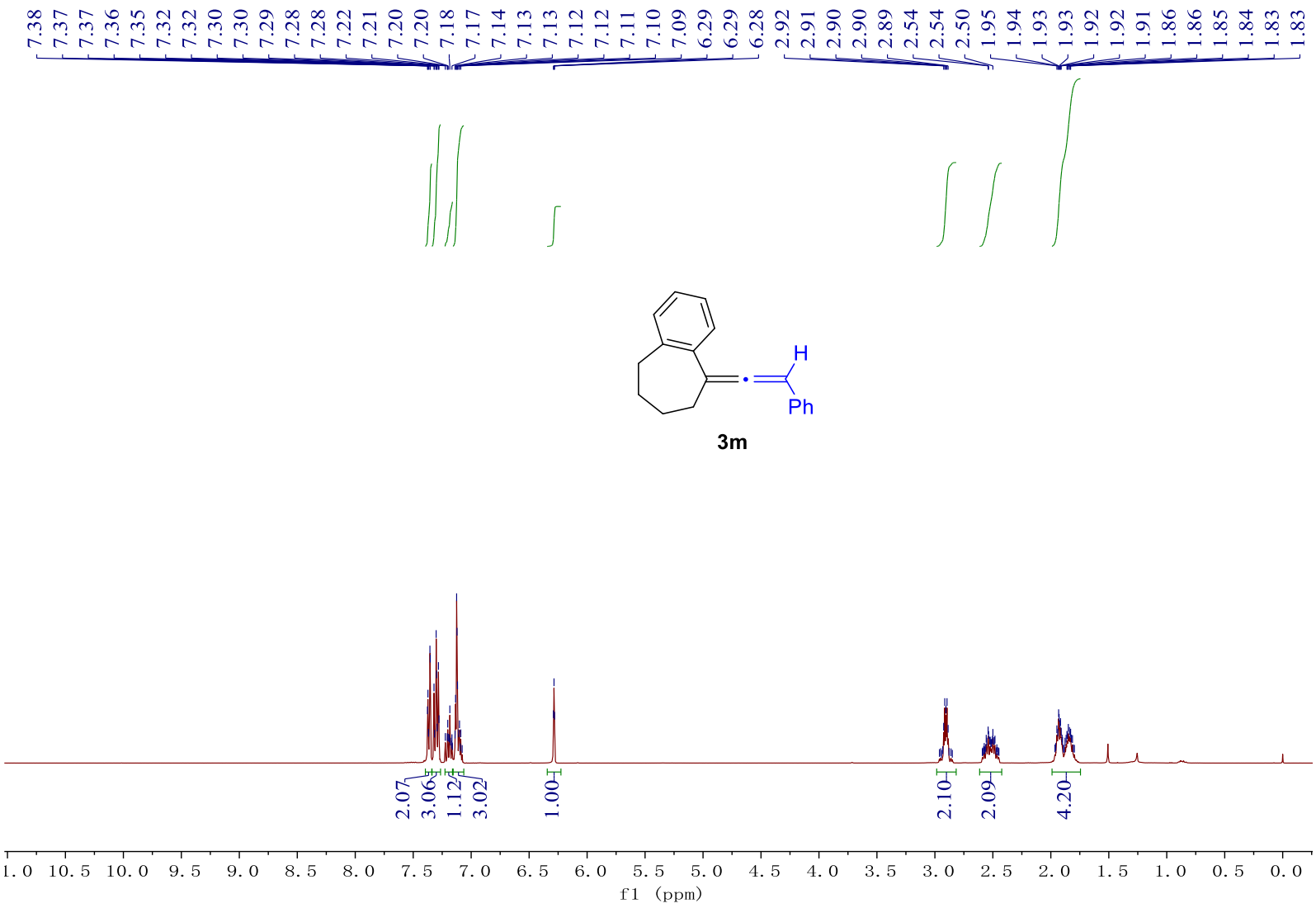
50
40
30
20
10
0
f1 (ppm)

3l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7.09
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6.28
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1.94
1.93
1.93
1.92
1.92
1.91
1.86
1.86
1.85
1.84
1.83
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H
Ph
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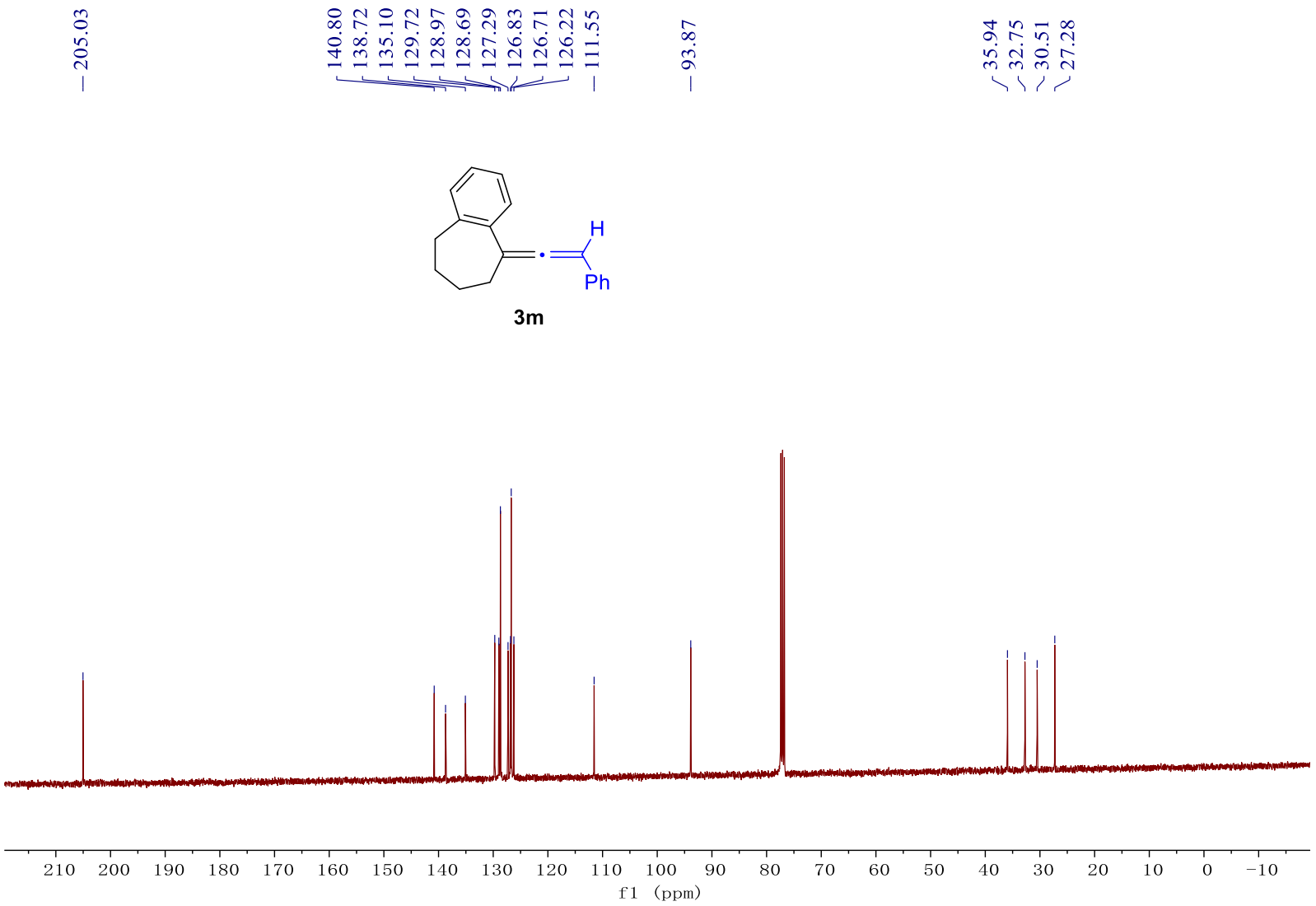
3.06
1.12
3.02
1.00
2.10
2.09
4.20
f1 (ppm)

3m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27.28
H
Ph
3m
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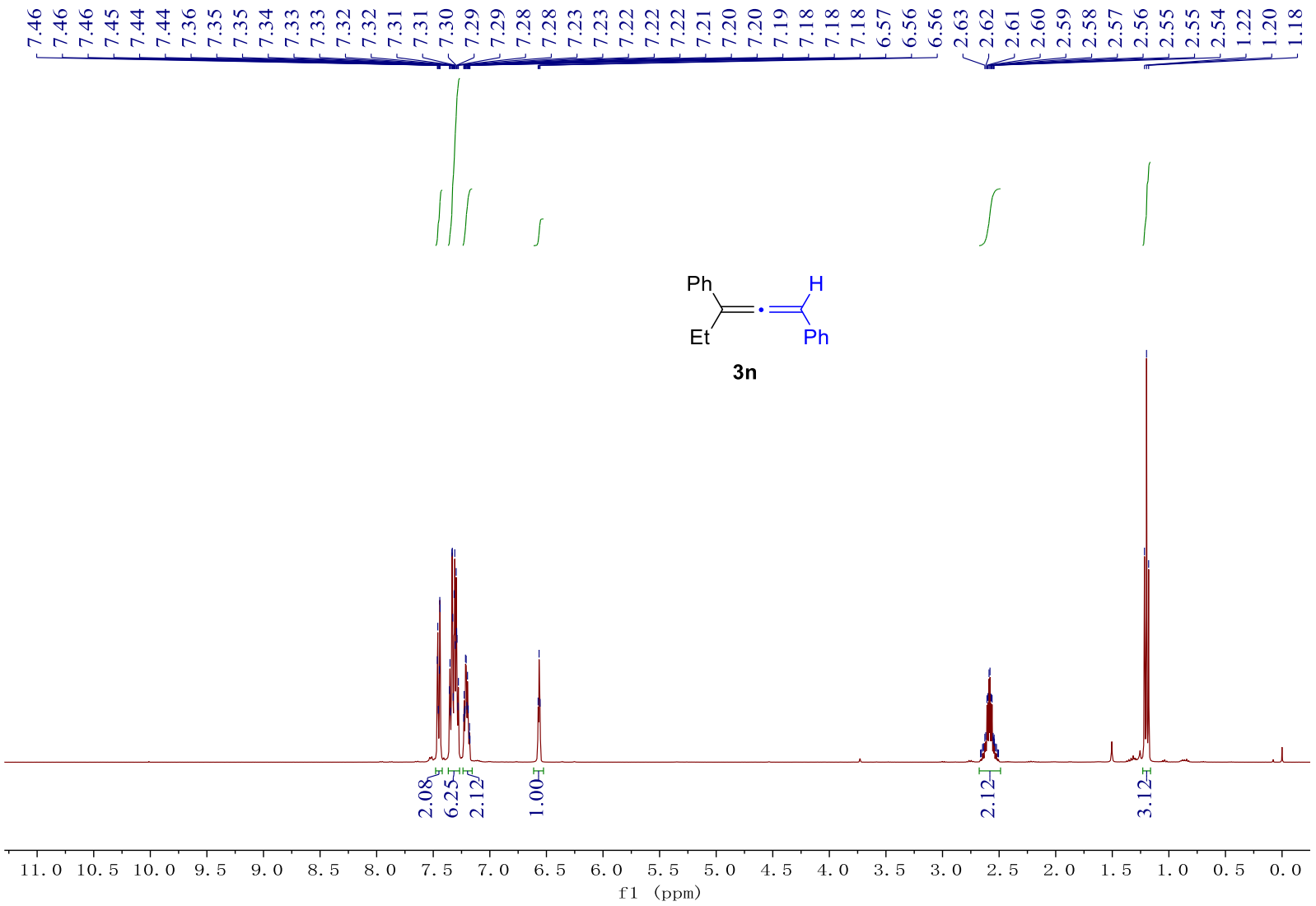
f1 (ppm)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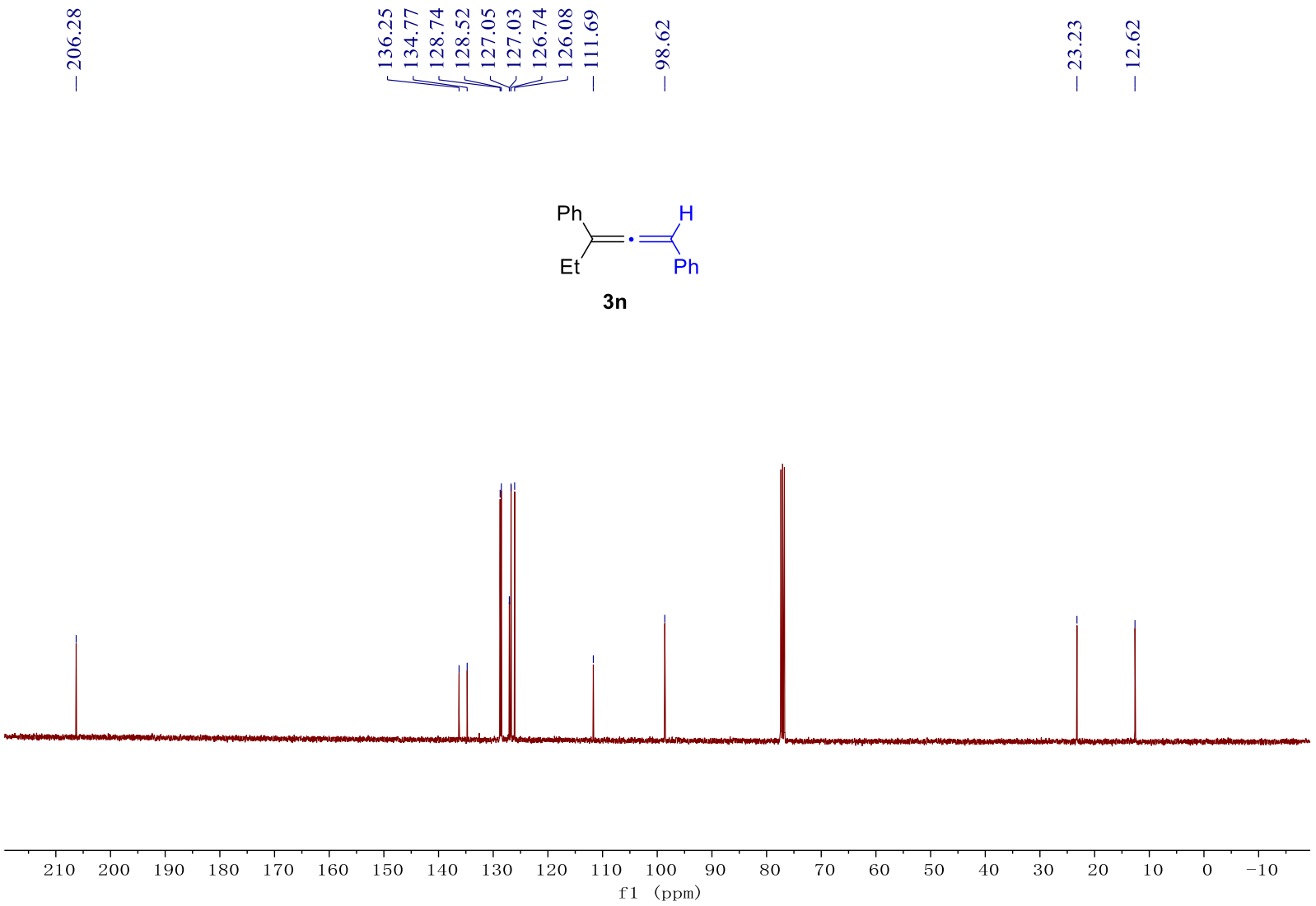
2.63 2.62 2.61 2.60 2.59 2.58 2.57 2.56 2.55 2.55 2.54 1.22 1.20 1.18
Ph
H
Et
Ph
3n
2.08
6.25
2.12
1.00
2.12
3.12
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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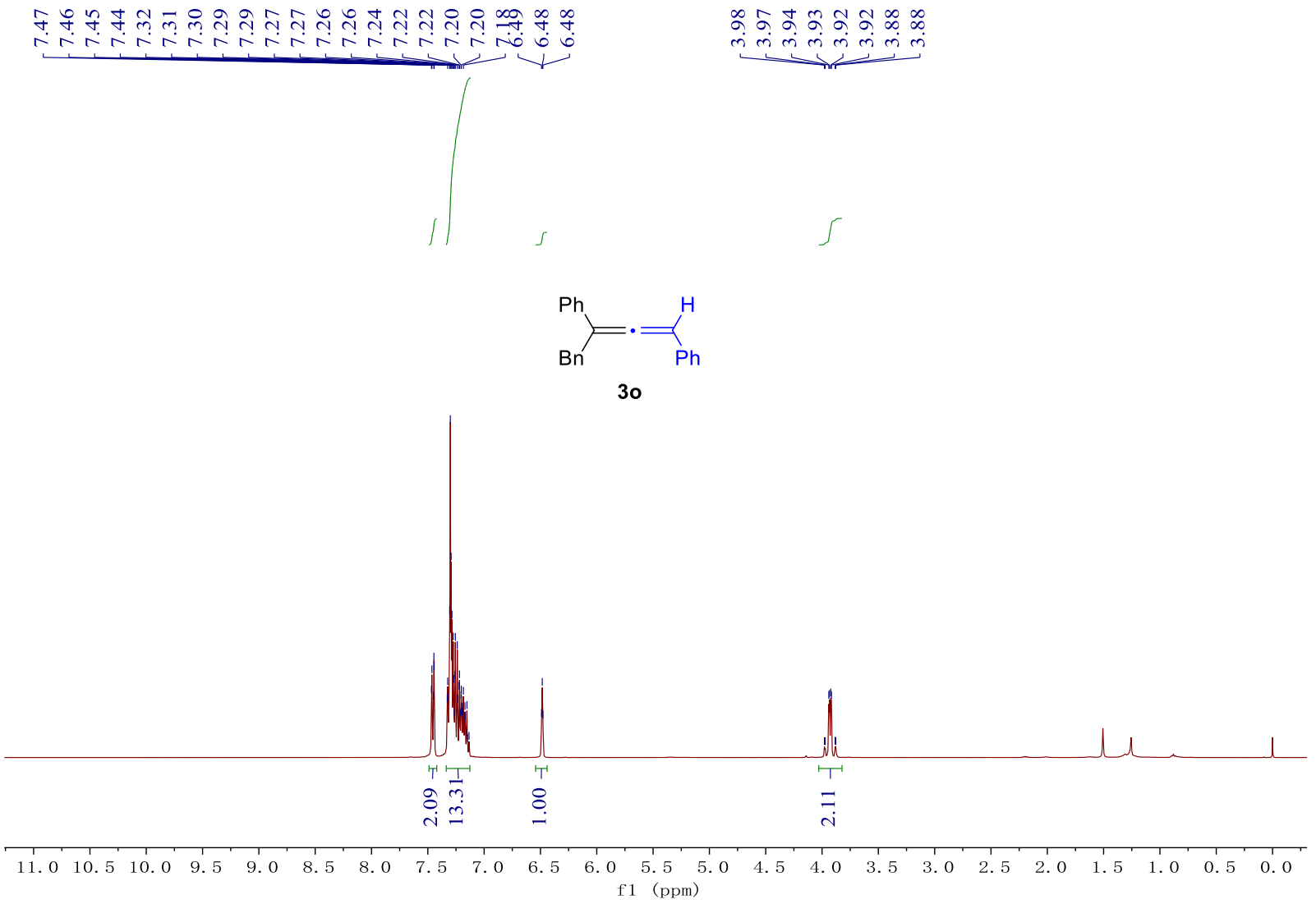
126.74
126.08
111.69
98.62
23.23
12.62
Ph
H
Et
Ph
3n
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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6.48
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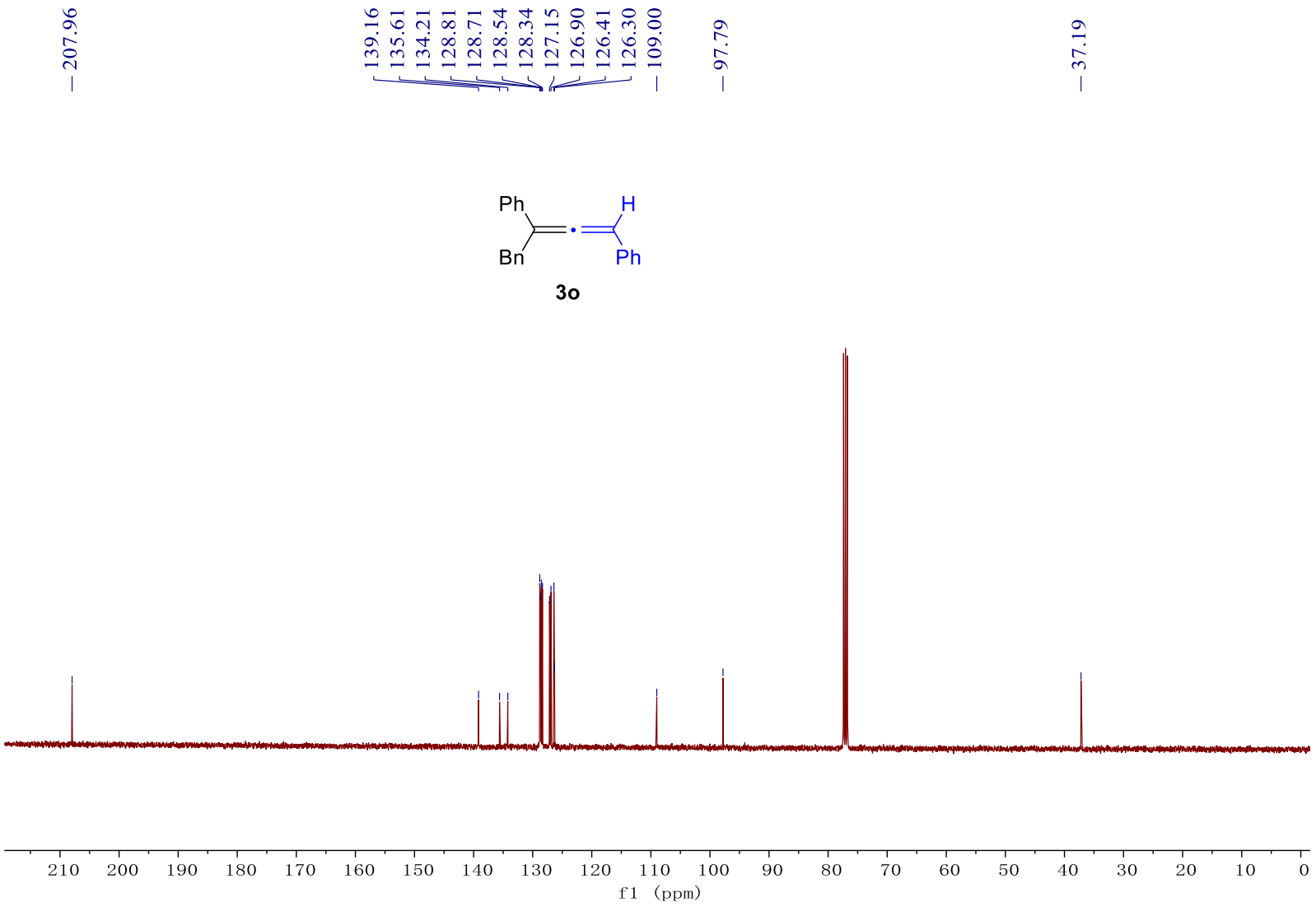
3.97
3.94
3.93
3.92
3.92
3.88
3.88
Ph
H
Bn
Ph
3o
2.09
13.31
1.00
2.11
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.34
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126.90
126.41
126.30
109.00
97.79
37.19
Ph
H
Bn
Ph
3o
210
200
190
180
170
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150
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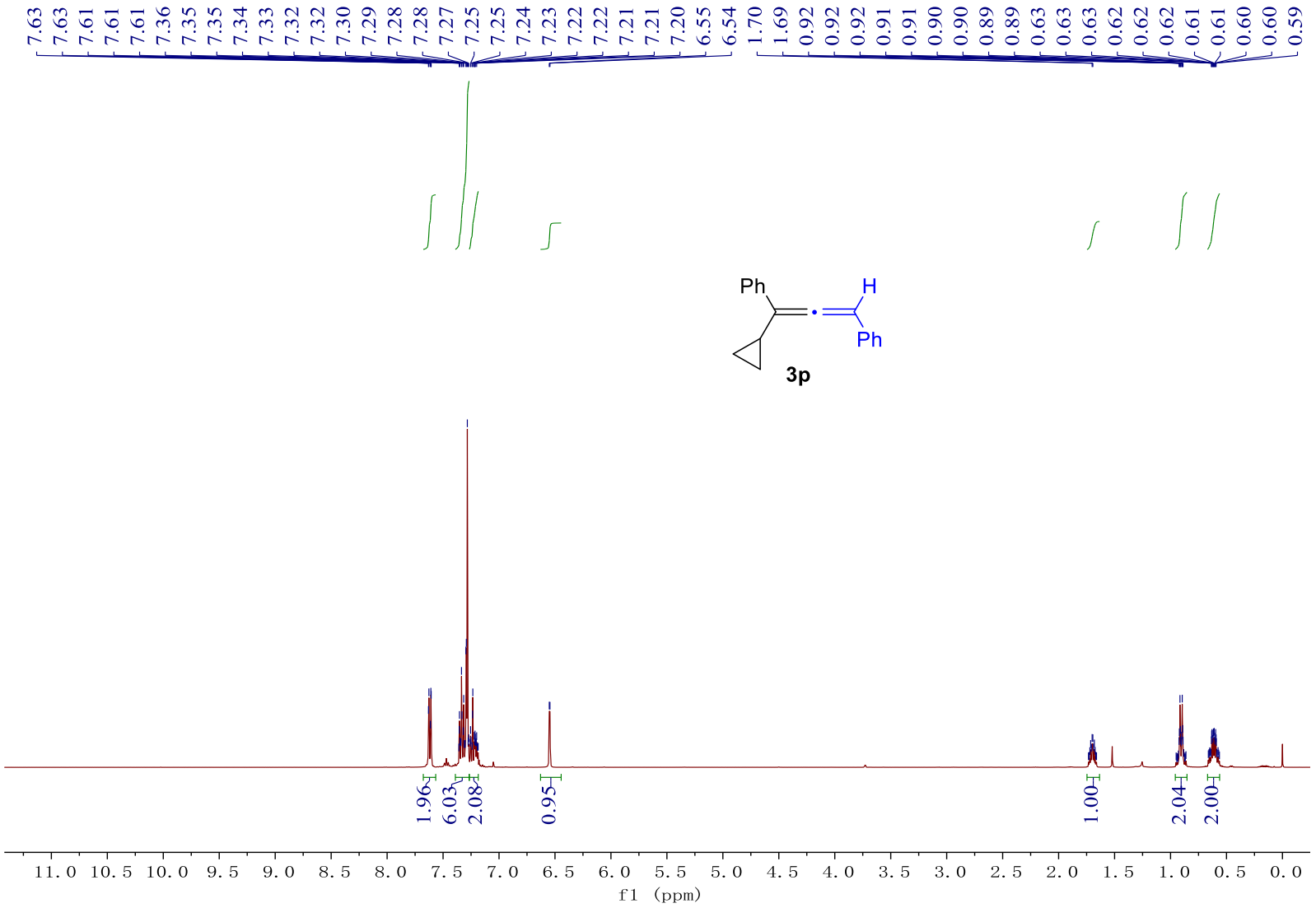
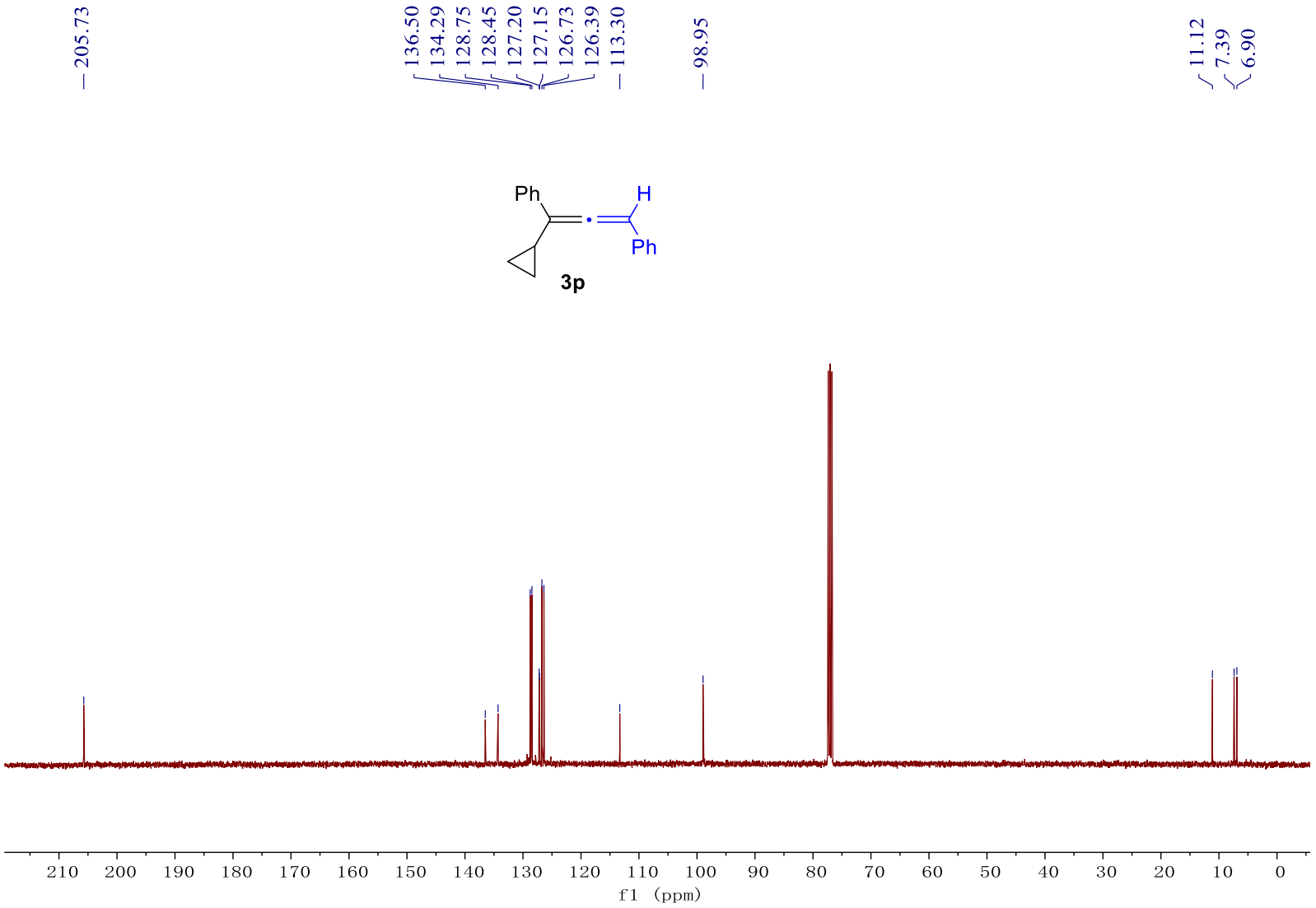
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.63 7.63 7.61 7.61 7.61 7.36 7.35 7.35 7.34 7.33 7.32 7.32 7.30 7.29 7.28 7.28 7.27 7.25 7.25 7.24 7.23 7.22 7.22 7.21 7.21 7.20 6.55 6.54
1.70 1.69 0.92 0.92 0.92 0.91 0.91 0.90 0.90 0.89 0.89 0.63 0.63 0.63 0.62 0.62 0.62 0.61 0.61 0.60 0.60 0.59
Ph
H
Ph
3p
1.96 6.03 2.08 0.95 1.00 2.04 2.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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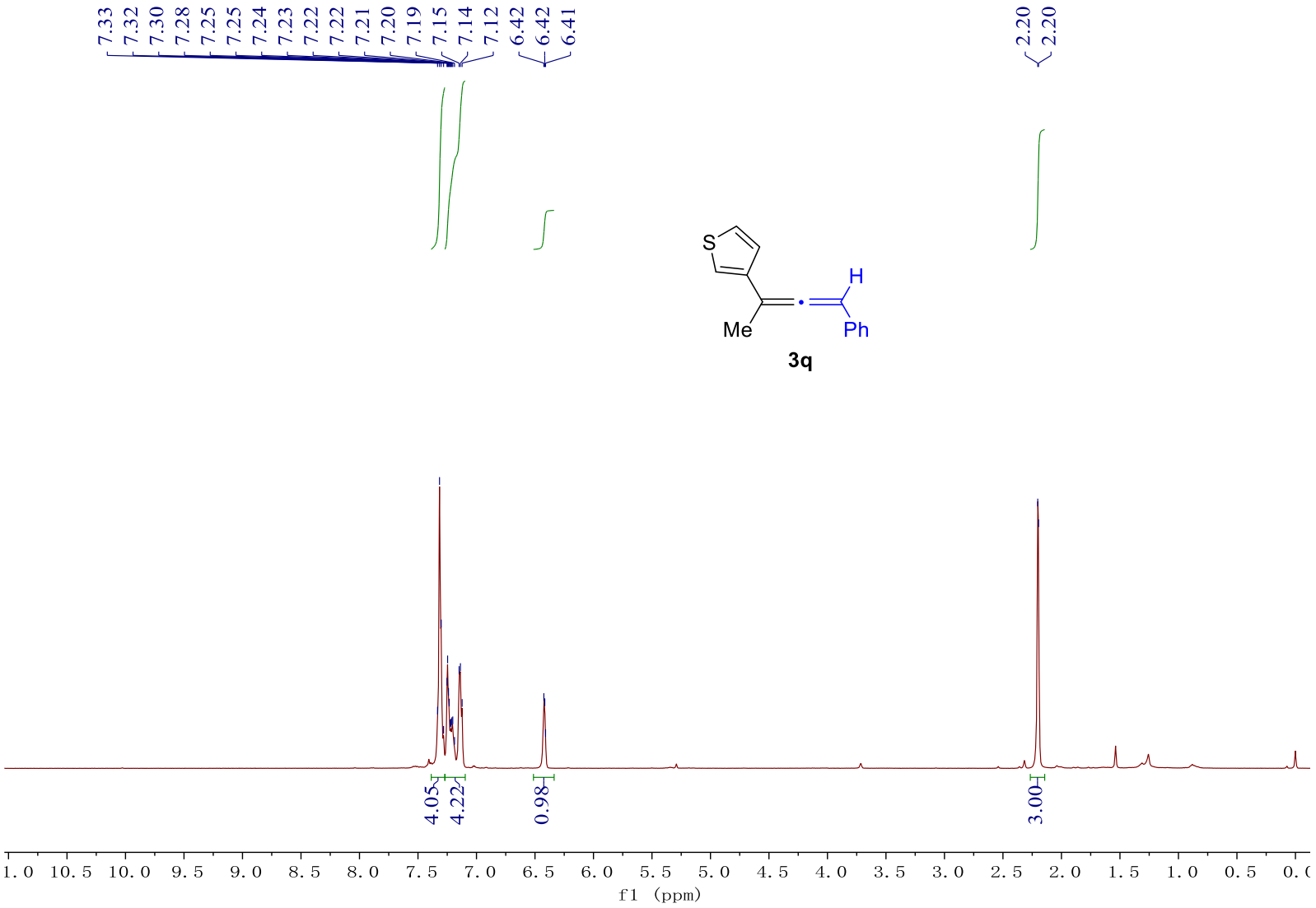
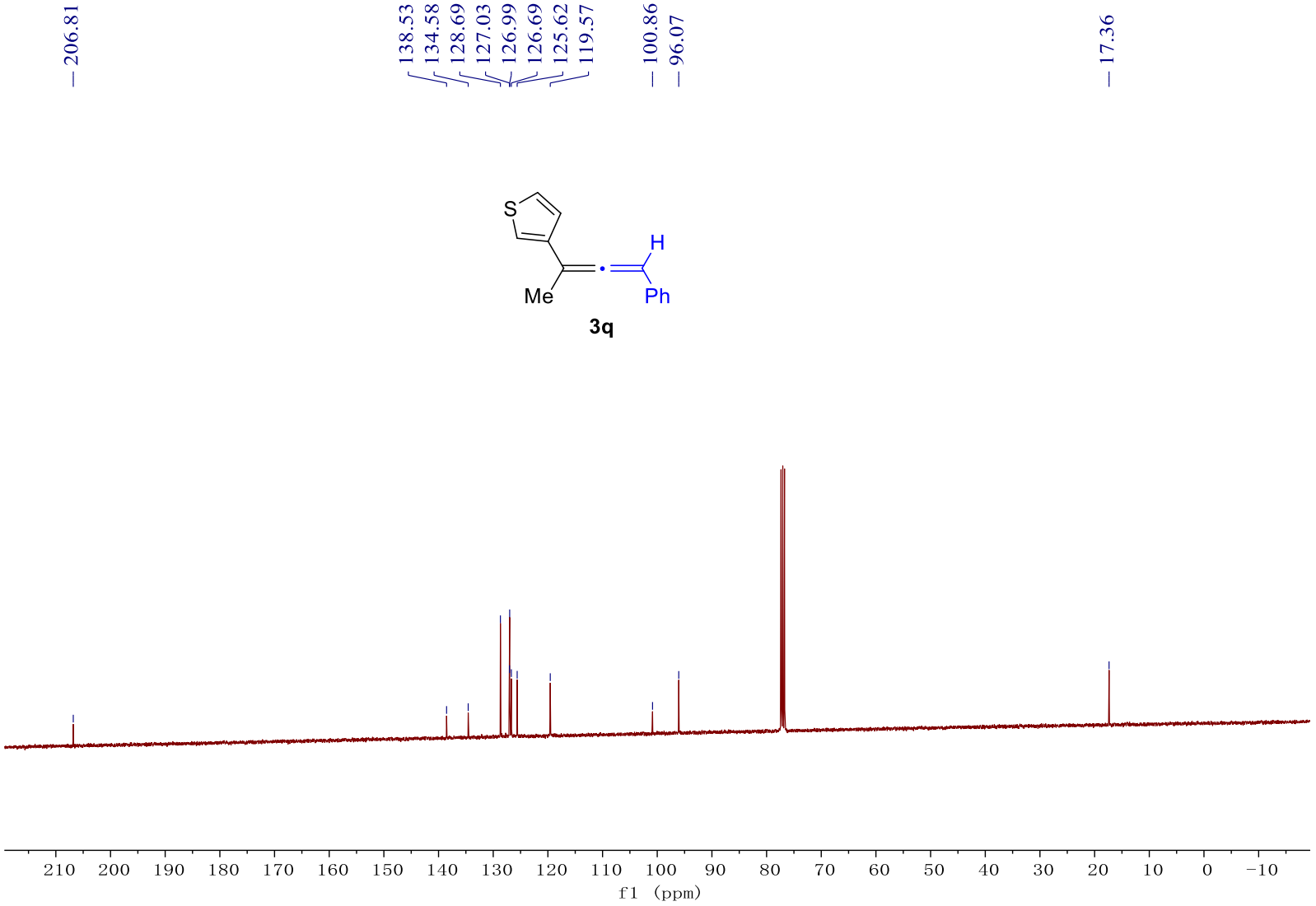
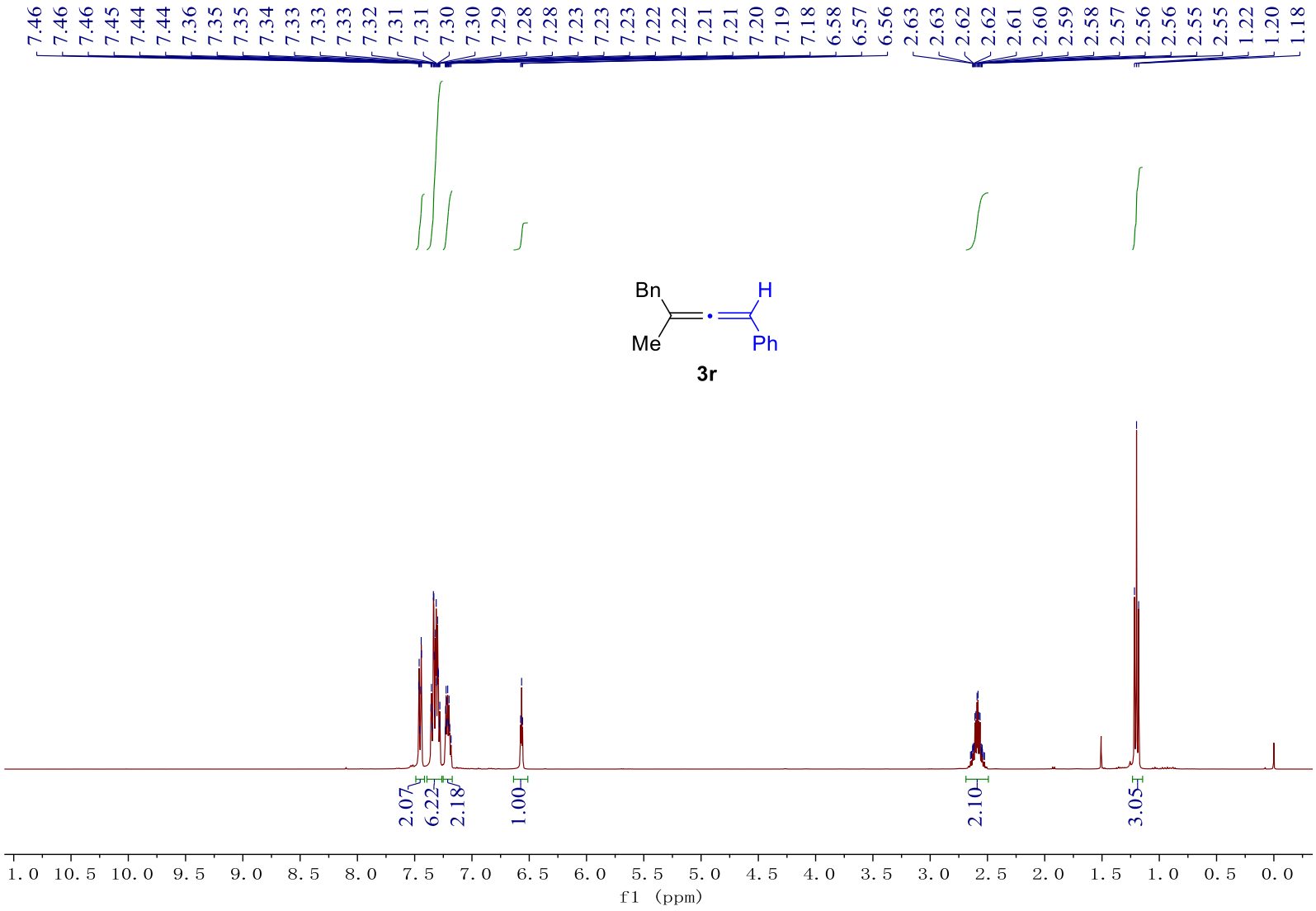
136.50
134.29
128.75
128.45
127.20
127.15
126.73
126.39
113.30
98.95
11.12
7.39
6.90
Ph
H
Ph
3p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.33
7.32
7.30
7.28
7.25
7.25
7.24
7.23
7.22
7.22
7.21
7.20
7.19
7.15
7.14
7.12
6.42
6.42
6.41
2.20
2.20
S
H
Me
Ph
3q
4.05
4.22
0.98
3.00
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


206.81
138.53
134.58
128.69
127.03
126.99
126.69
125.62
119.57
100.86
96.07
17.36
S
H
Me
Ph
3q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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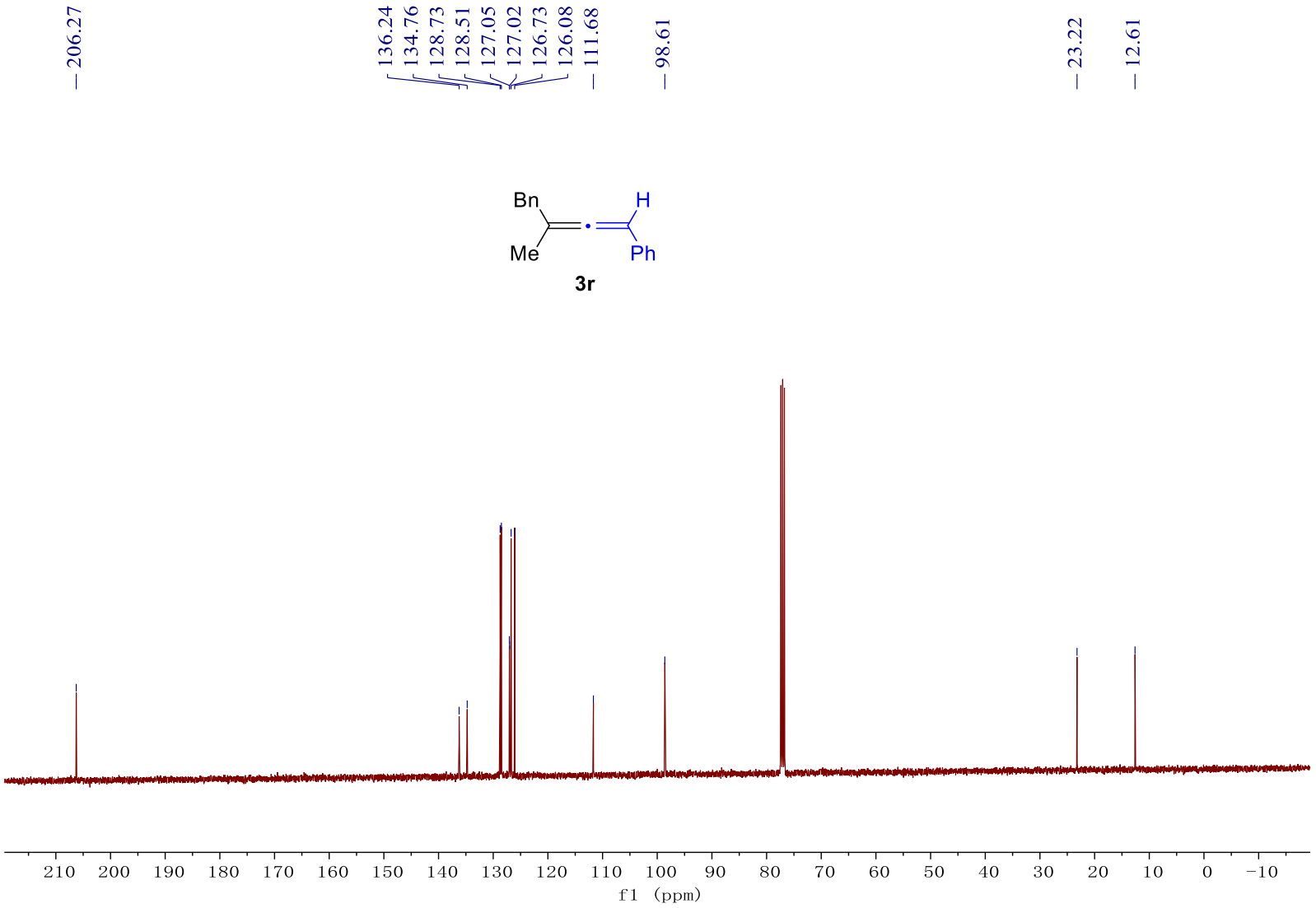
2.63 2.63 2.62 2.62 2.61 2.60 2.59 2.58 2.57 2.56 2.56 2.55 2.55 1.22 1.20 1.18
Bn
H
Me
Ph
3r
2.07
6.22
2.18
1.00
2.10
3.05
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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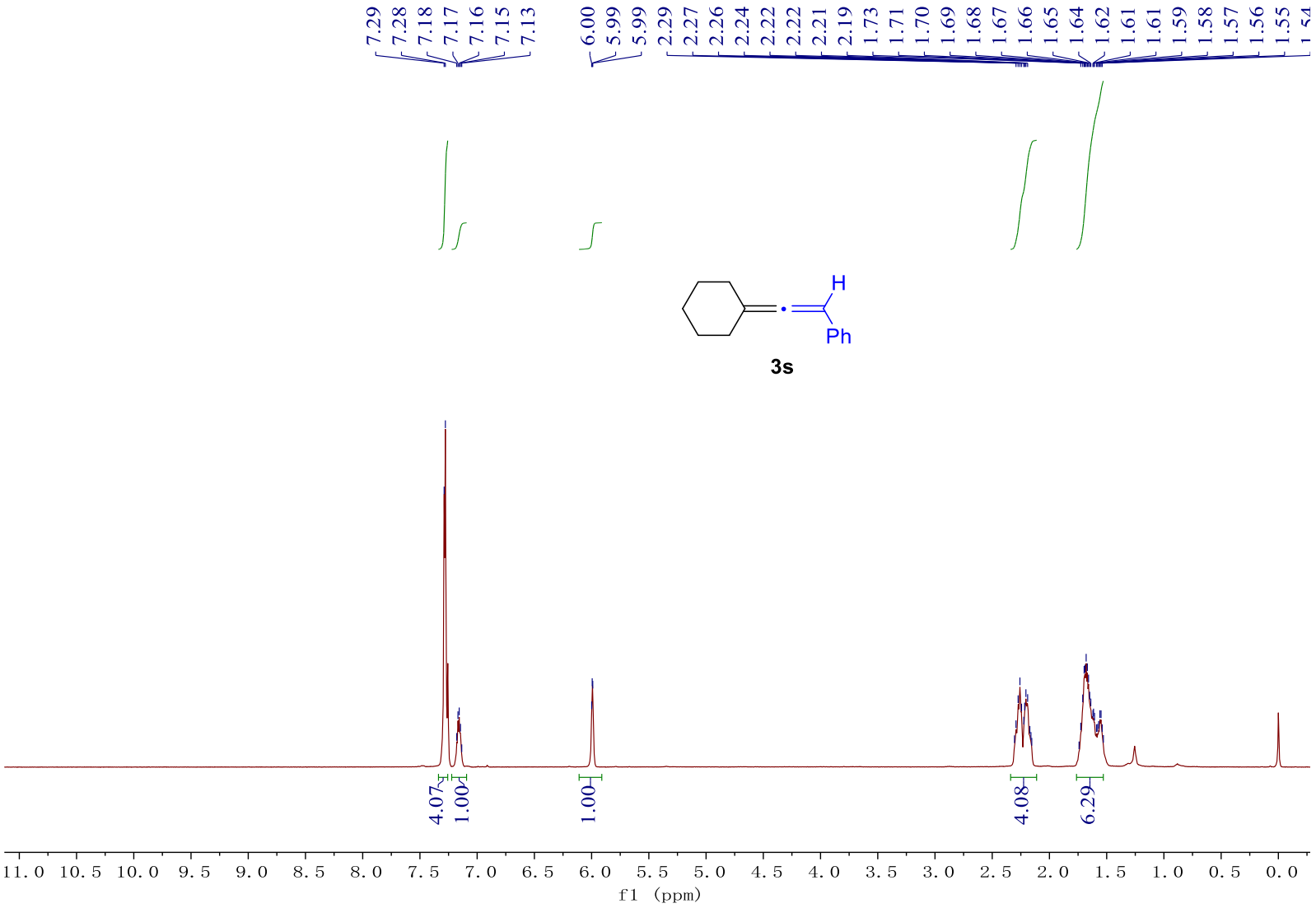
136.24
134.76
128.73
128.51
127.05
127.02
126.73
126.08
111.68
98.61
23.22
12.61
Bn
H
Me
Ph
3r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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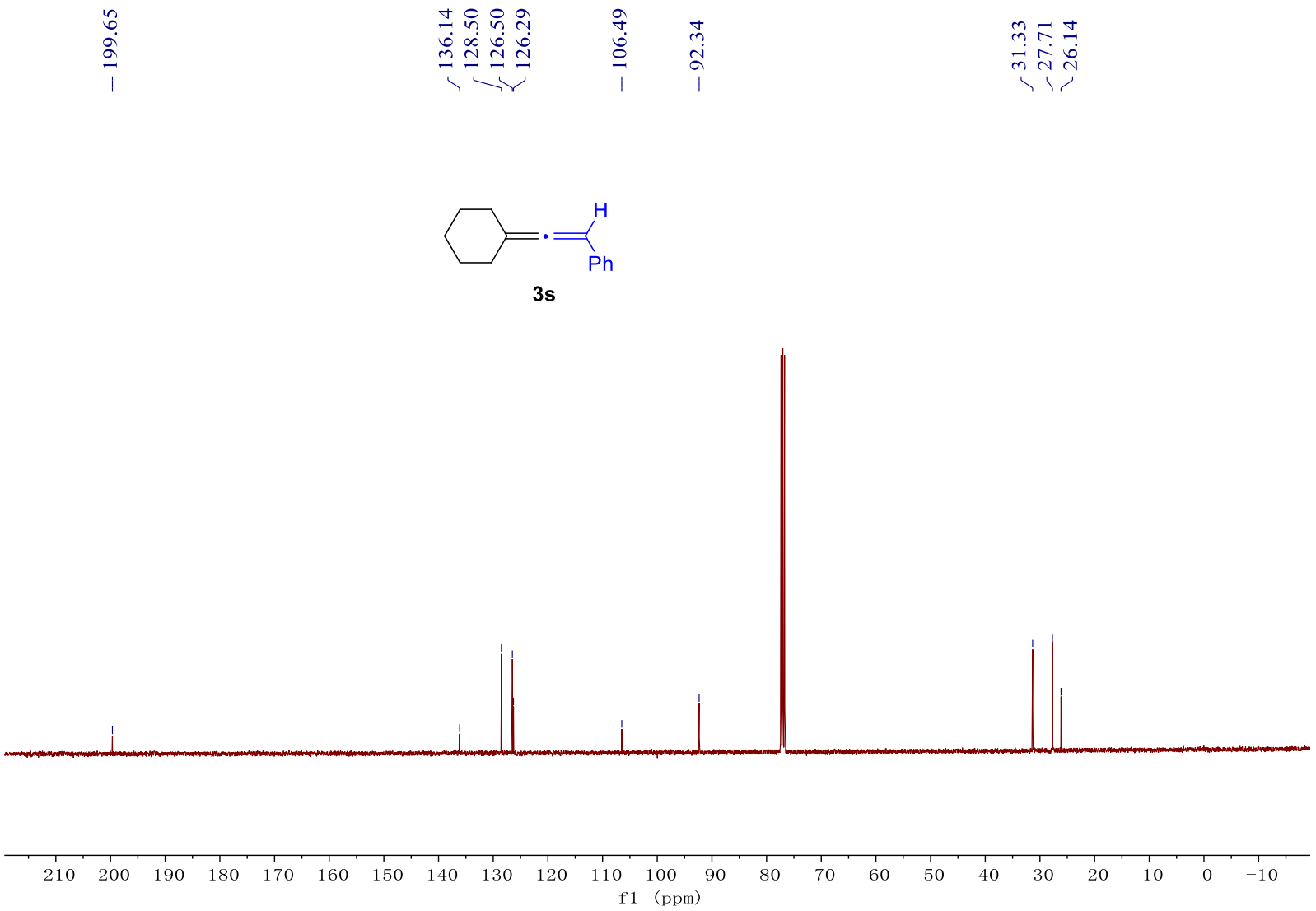
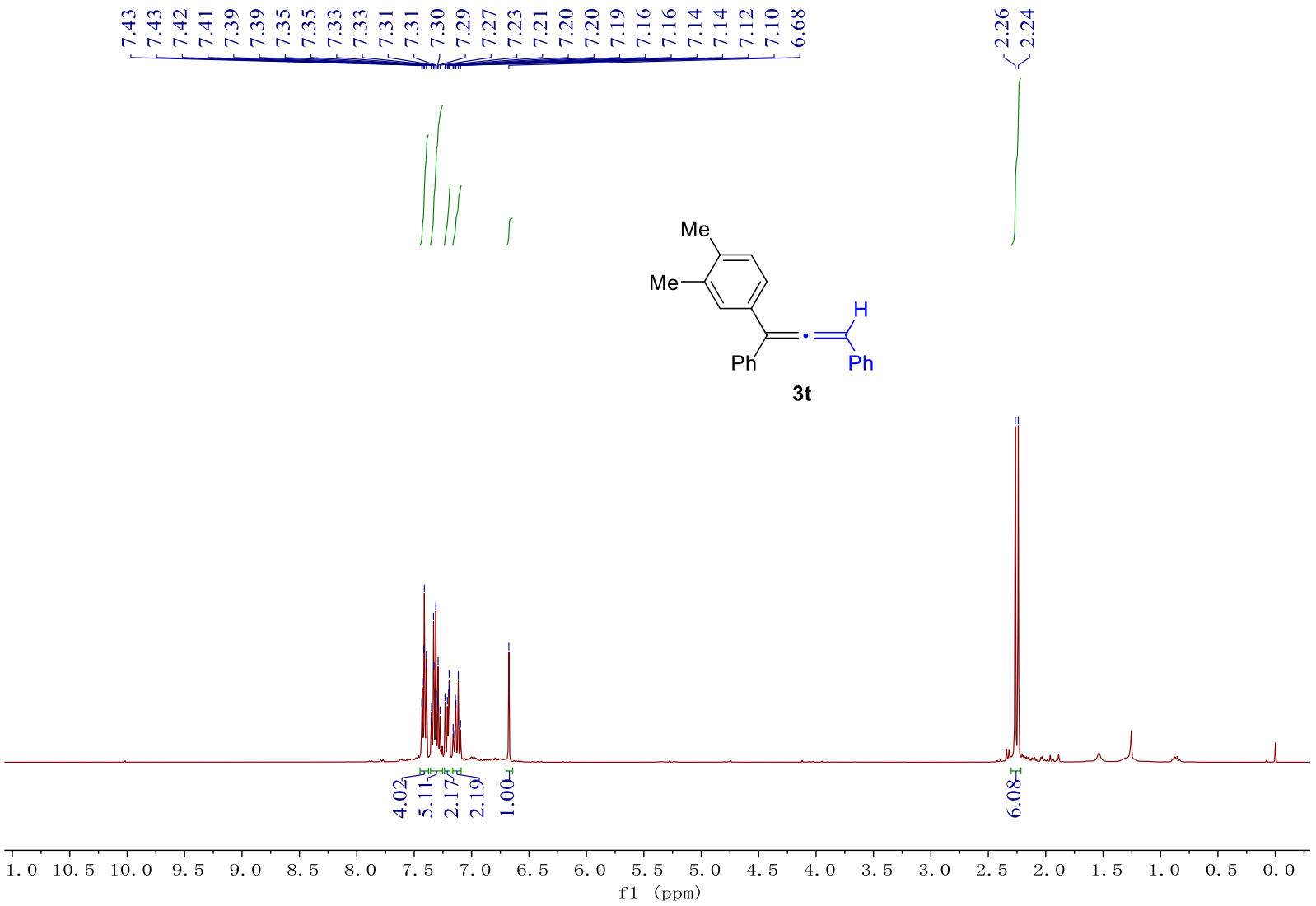
7.28
7.18
7.17
7.16
7.15
7.13
6.00
5.99
5.99
2.29
2.27
2.26
2.24
2.22
2.22
2.21
2.19
1.73
1.71
1.70
1.69
1.68
1.67
1.66
1.65
1.64
1.62
1.61
1.61
1.59
1.58
1.57
1.56
1.55
H
Ph
3s
4.07
1.00
1.00
4.08
6.29
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


199.65
136.14
128.50
126.50
126.29
106.49
92.34
31.33
27.71
26.14
H
Ph
3s
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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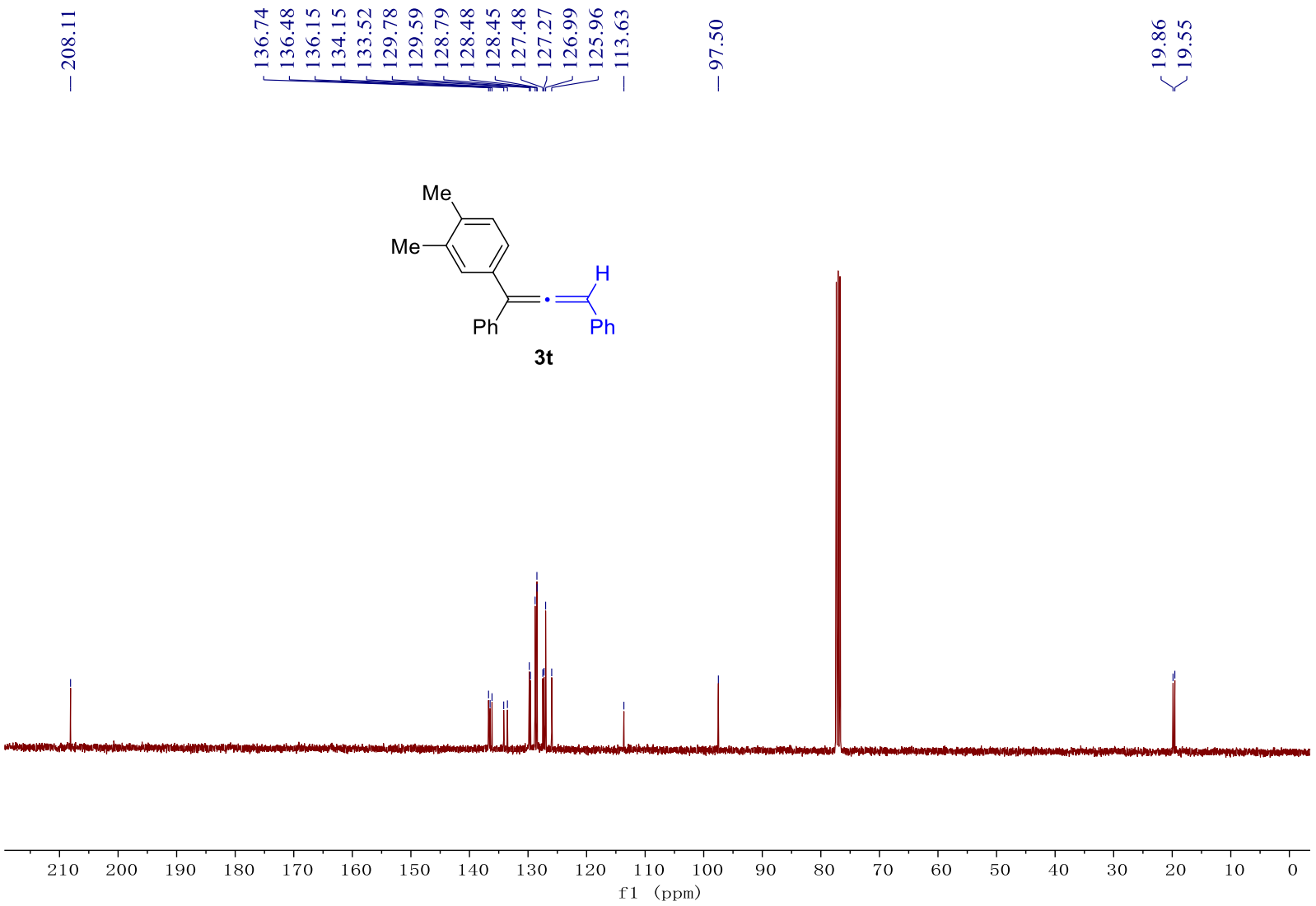
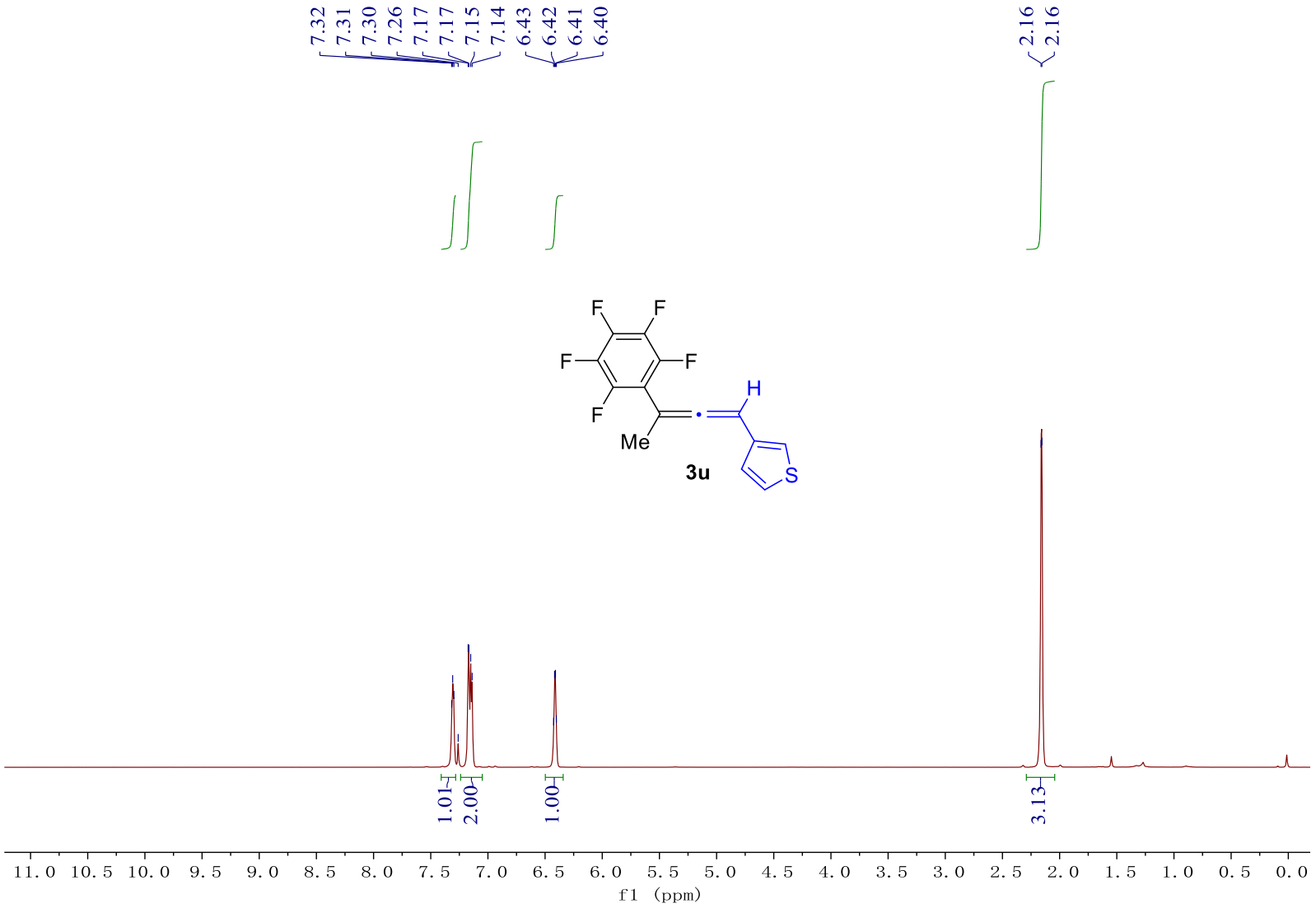
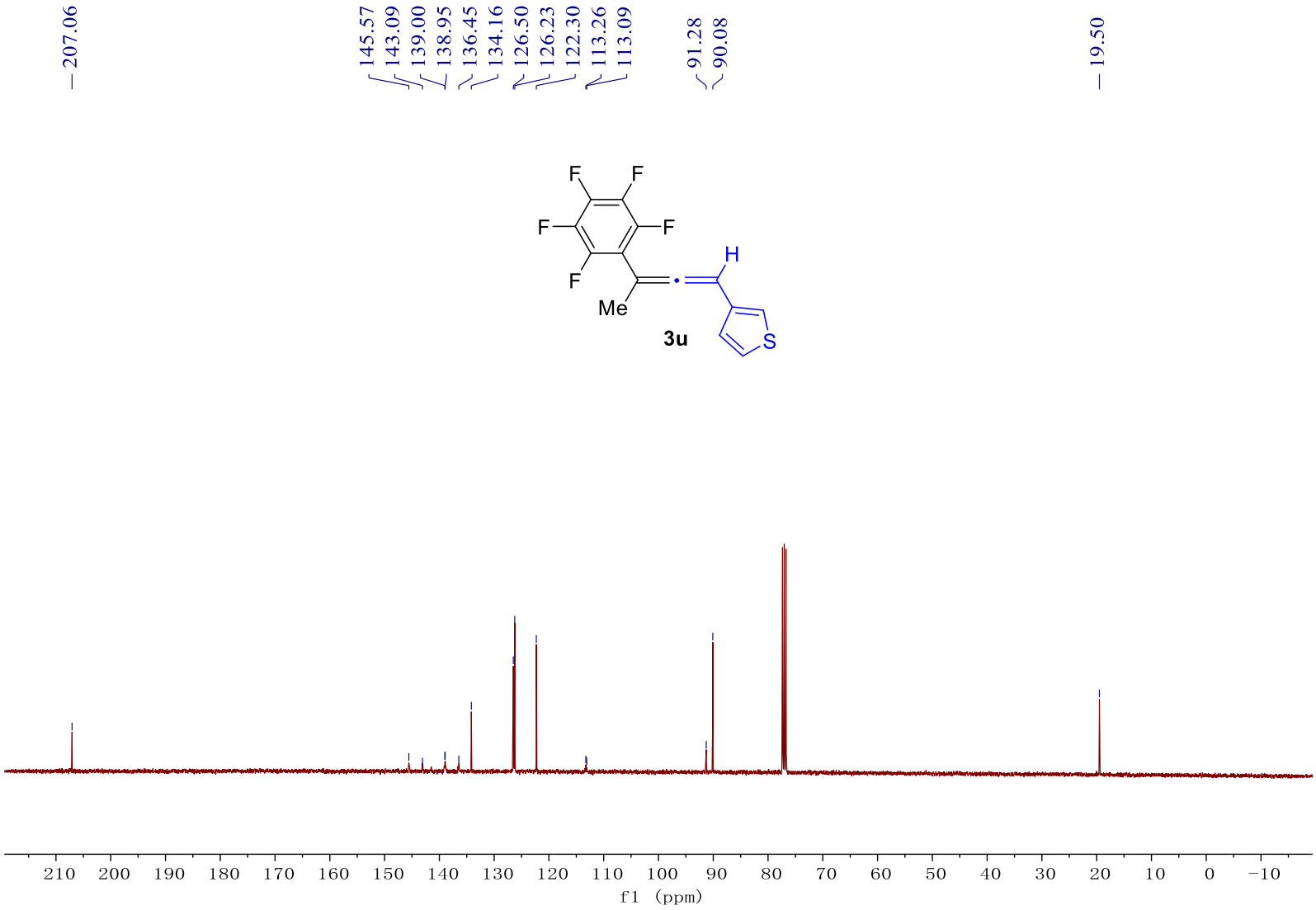
7.43
7.42
7.41
7.39
7.39
7.35
7.35
7.33
7.33
7.31
7.31
7.30
7.29
7.27
7.23
7.21
7.20
7.20
7.19
7.16
7.16
7.14
7.14
7.12
7.10
6.68
2.26
2.24
Me
Me
H
Ph
Ph
3t
4.02
5.11
2.17
2.19
1.00
6.08
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


208.11
136.74
136.48
136.15
134.15
133.52
129.78
129.59
128.79
128.48
128.45
127.48
127.27
126.99
125.96
113.63
97.50
19.86
19.55
Me
Me
Ph
H
Ph
3t
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.32
7.31
7.30
7.26
7.17
7.17
7.15
7.14
6.43
6.42
6.41
6.40
2.16
2.16
F
F
F
F
F
Me
H
S
3u
1.01
2.00
1.00
3.13
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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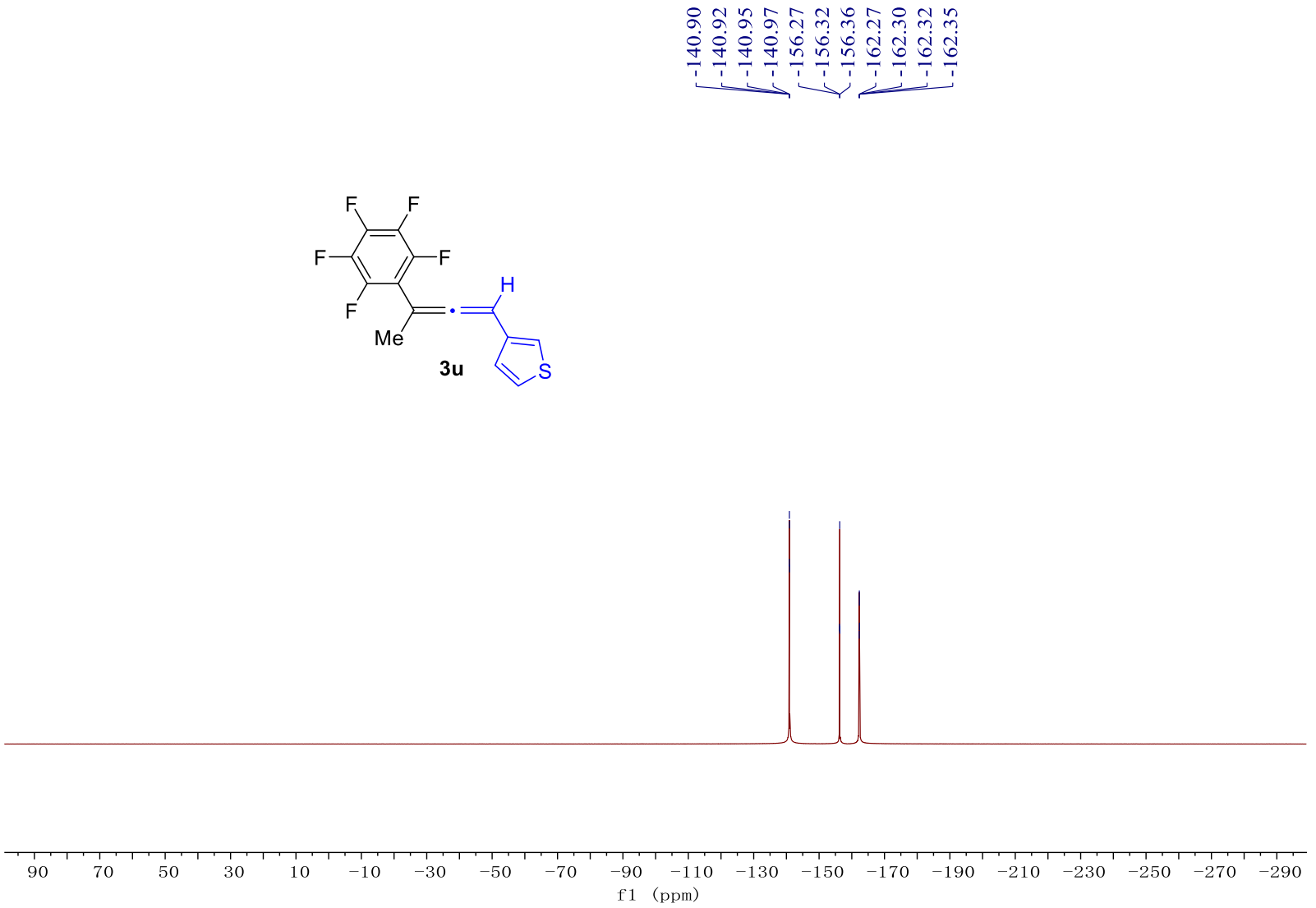
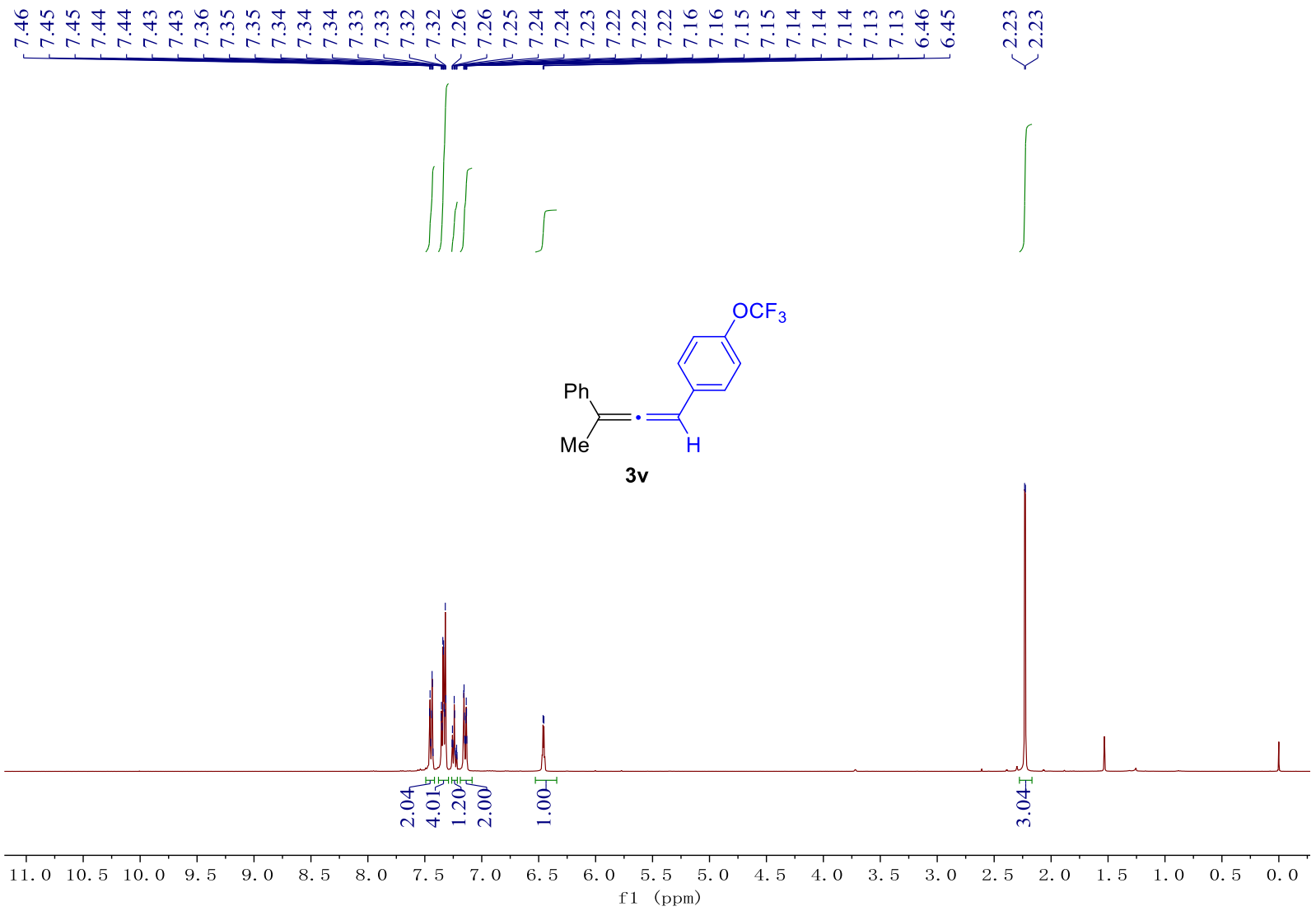
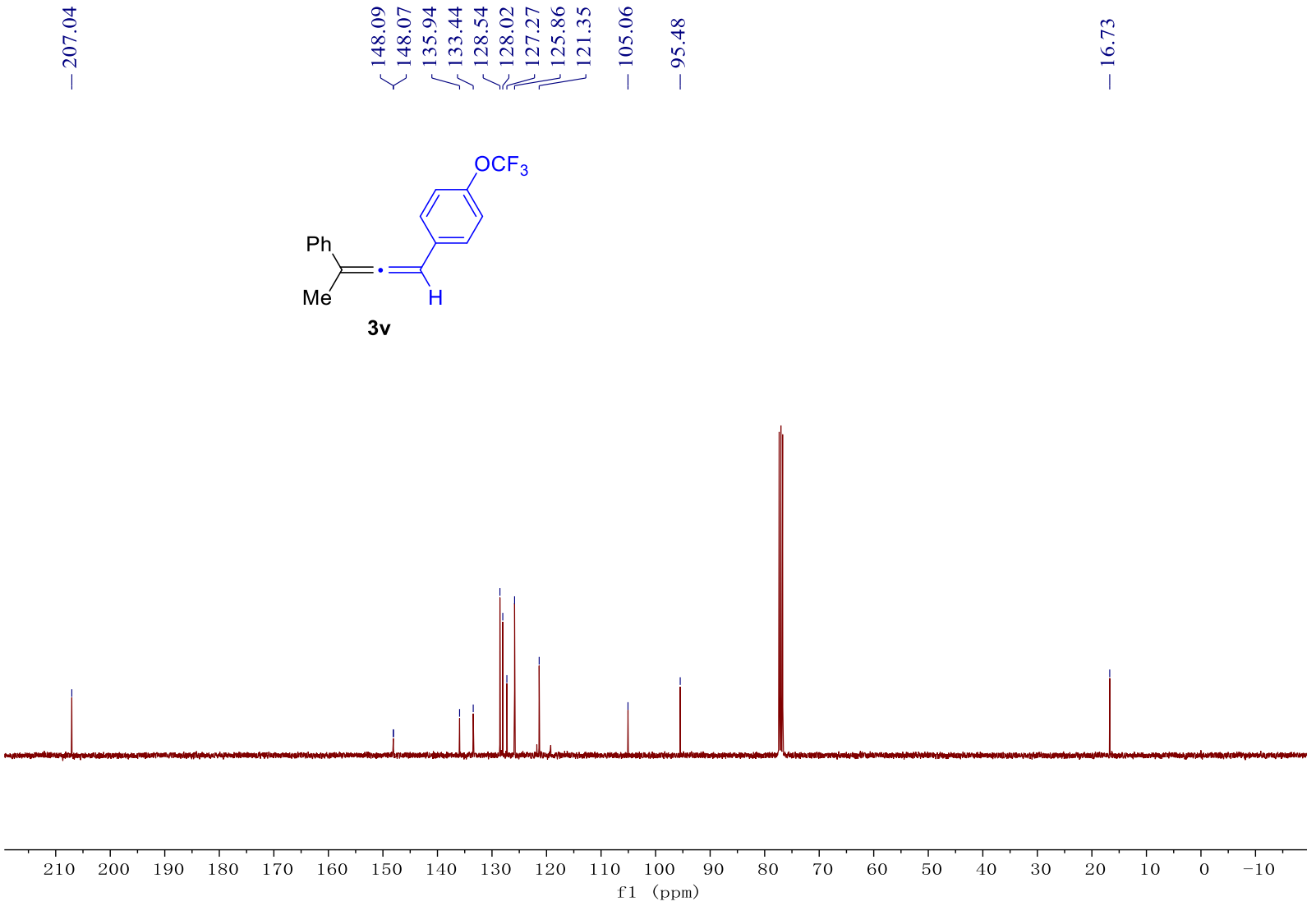
145.57
143.09
139.00
138.95
136.45
134.16
126.50
126.23
122.30
113.26
113.09
91.28
90.08
19.50
F
F
F
F
F
H
Me
S
3u
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


-140.90
-140.92
-140.95
-140.97
-156.27
-156.32
-156.36
-162.27
-162.30
-162.32
-162.35
F
F
F
F
F
Me
H
S
3u
90
70
50
30
10
−10
−30
−50
−70
−90
−110
−130
−150
−170
−190
−210
−230
−250
−270
−290
f1 (ppm)


7.46
7.45
7.45
7.44
7.44
7.43
7.43
7.36
7.35
7.35
7.34
7.34
7.34
7.33
7.33
7.32
7.32
7.26
7.26
7.25
7.24
7.24
7.23
7.22
7.22
7.22
7.16
7.16
7.15
7.15
7.14
7.14
7.14
7.13
7.13
6.46
6.45
2.23
2.23
OCF3
Ph
Me
H
3v
2.04
4.01
1.20
2.00
1.00
3.04
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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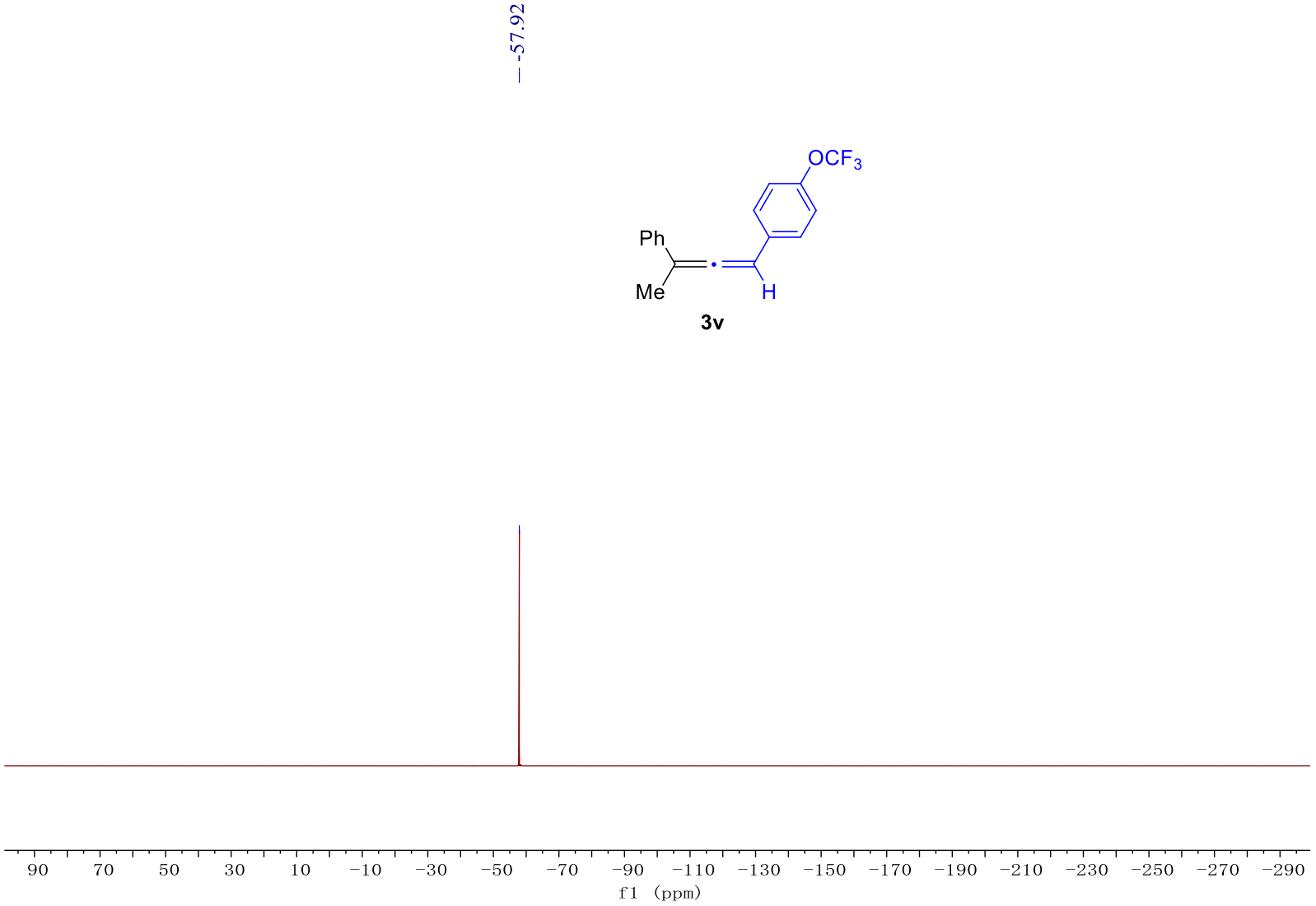
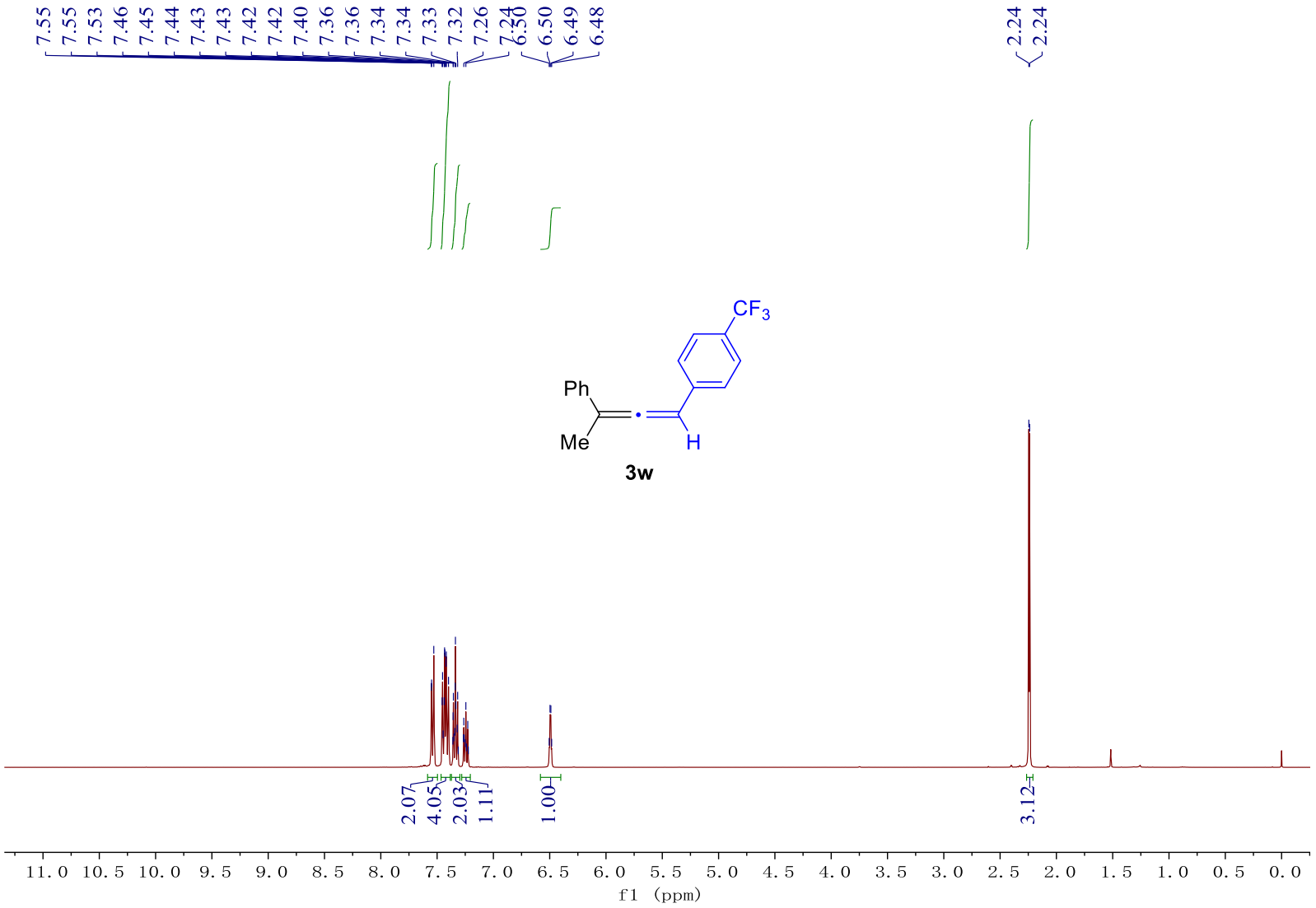
148.07
135.94
133.44
128.54
128.02
127.27
125.86
121.35
105.06
95.48
16.73
OCF3
Ph
Me
H
3v
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


-57.92
OCF3
Ph
Me
H
3v
90
70
50
30
10
−10
−30
−50
−70
−90
−110
−130
−150
−170
−190
−210
−230
−250
−270
−290
f1 (ppm)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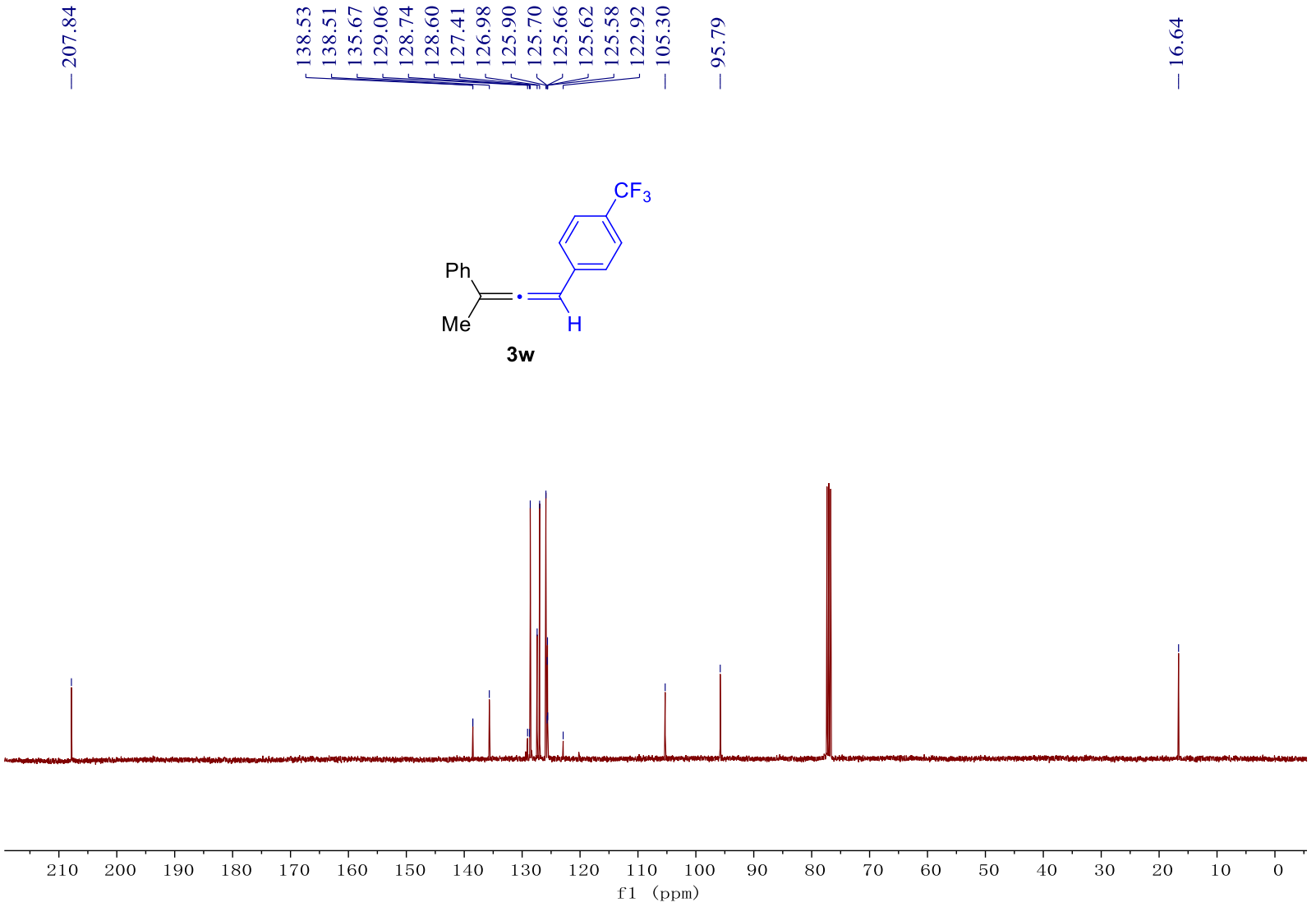
7.42
7.40
7.36
7.36
7.34
7.34
7.33
7.32
7.26
7.24
6.50
6.49
6.48
2.24
2.24
CF3
Ph
Me
H
3w
2.07
4.05
2.03
1.11
1.00
3.12
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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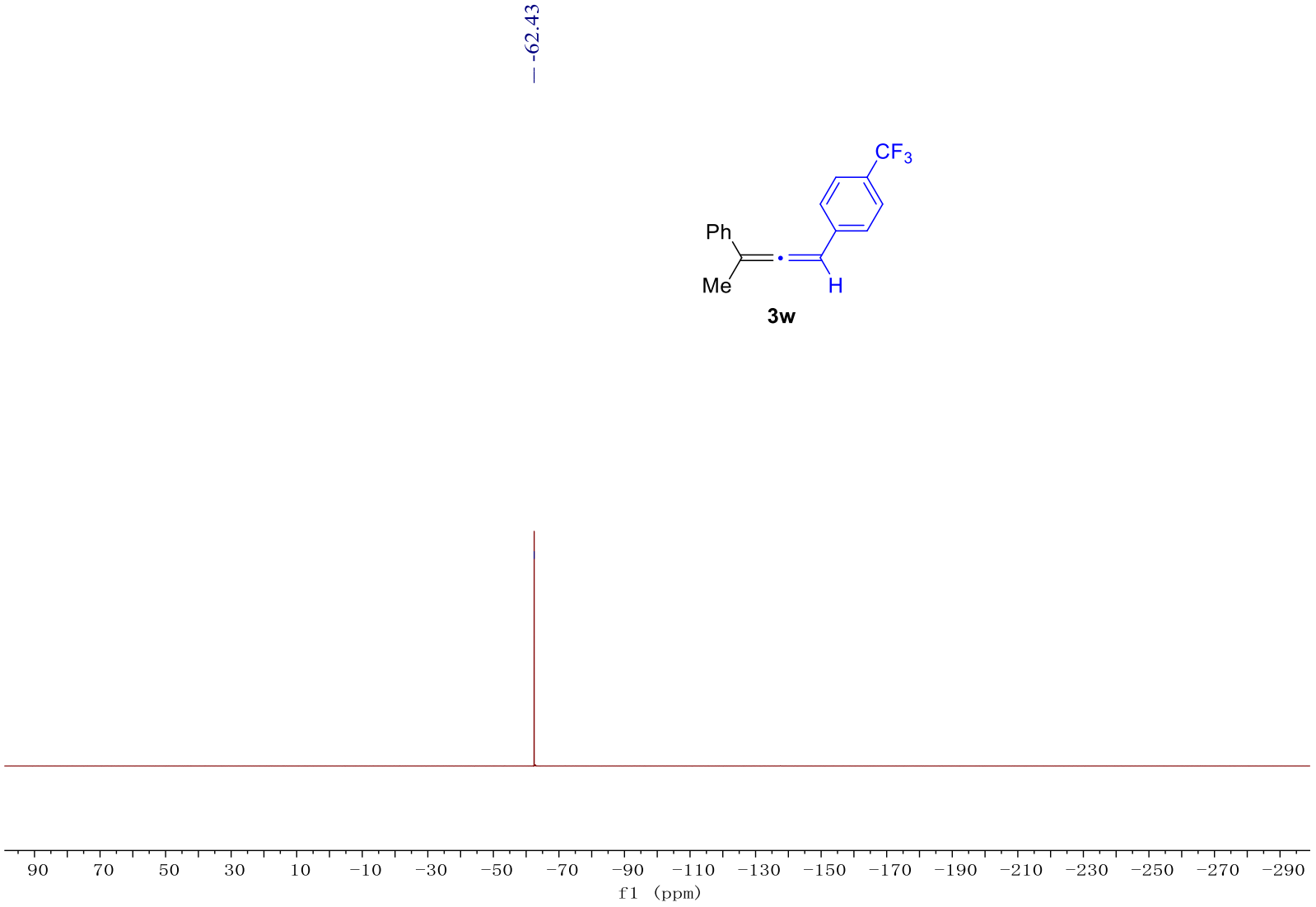
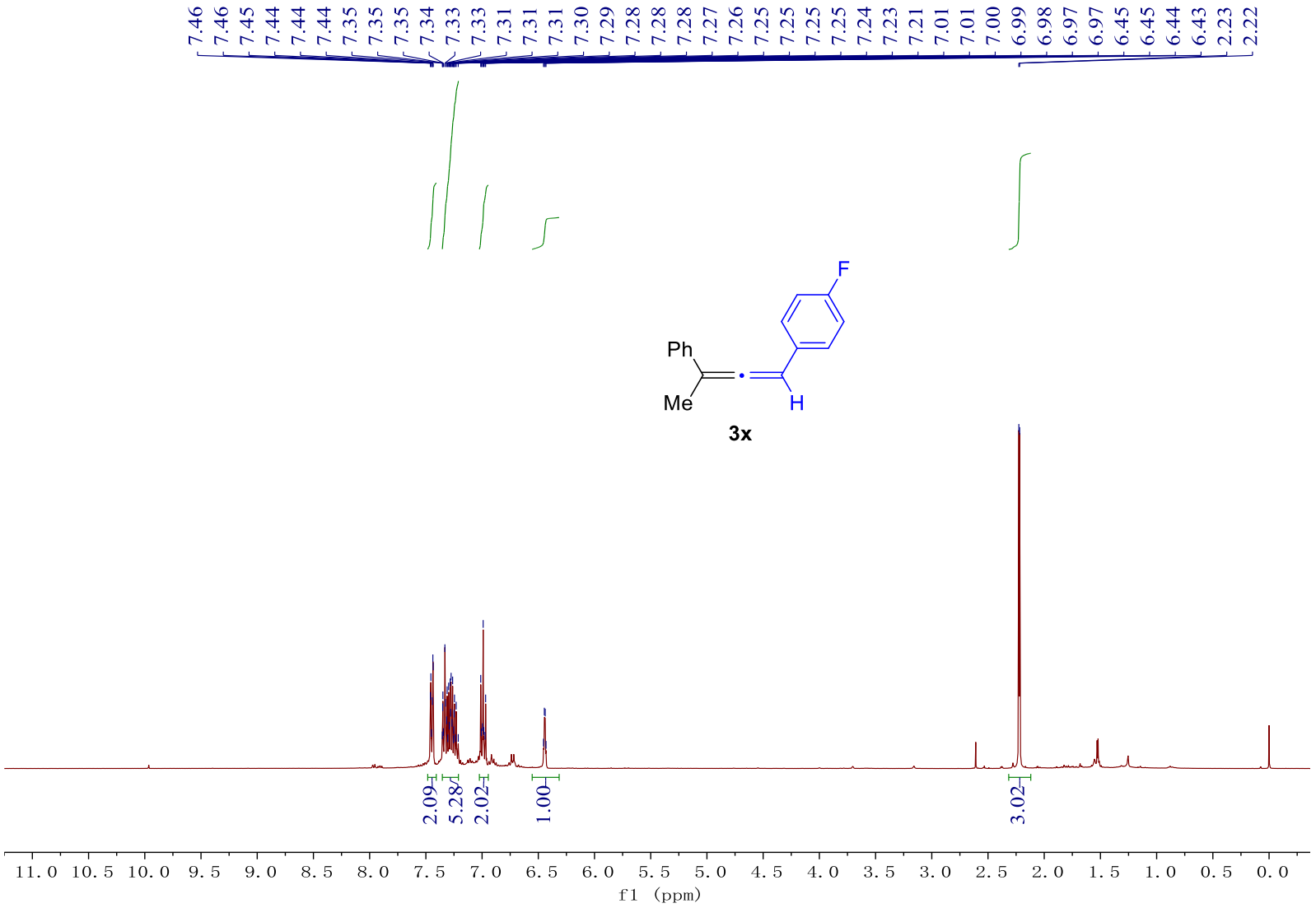
127.41
126.98
125.90
125.70
125.66
125.62
125.58
122.92
105.30
95.79
16.64
CF3
Ph
Me
H
3w
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-62.43
CF3
Ph
Me
H
3w
90
70
50
30
10
−10
−30
−50
−70
−90
−110
−130
−150
−170
−190
−210
−230
−250
−270
−290
f1 (ppm)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7.46
7.45
7.44
7.44
7.44
7.35
7.35
7.35
7.34
7.33
7.33
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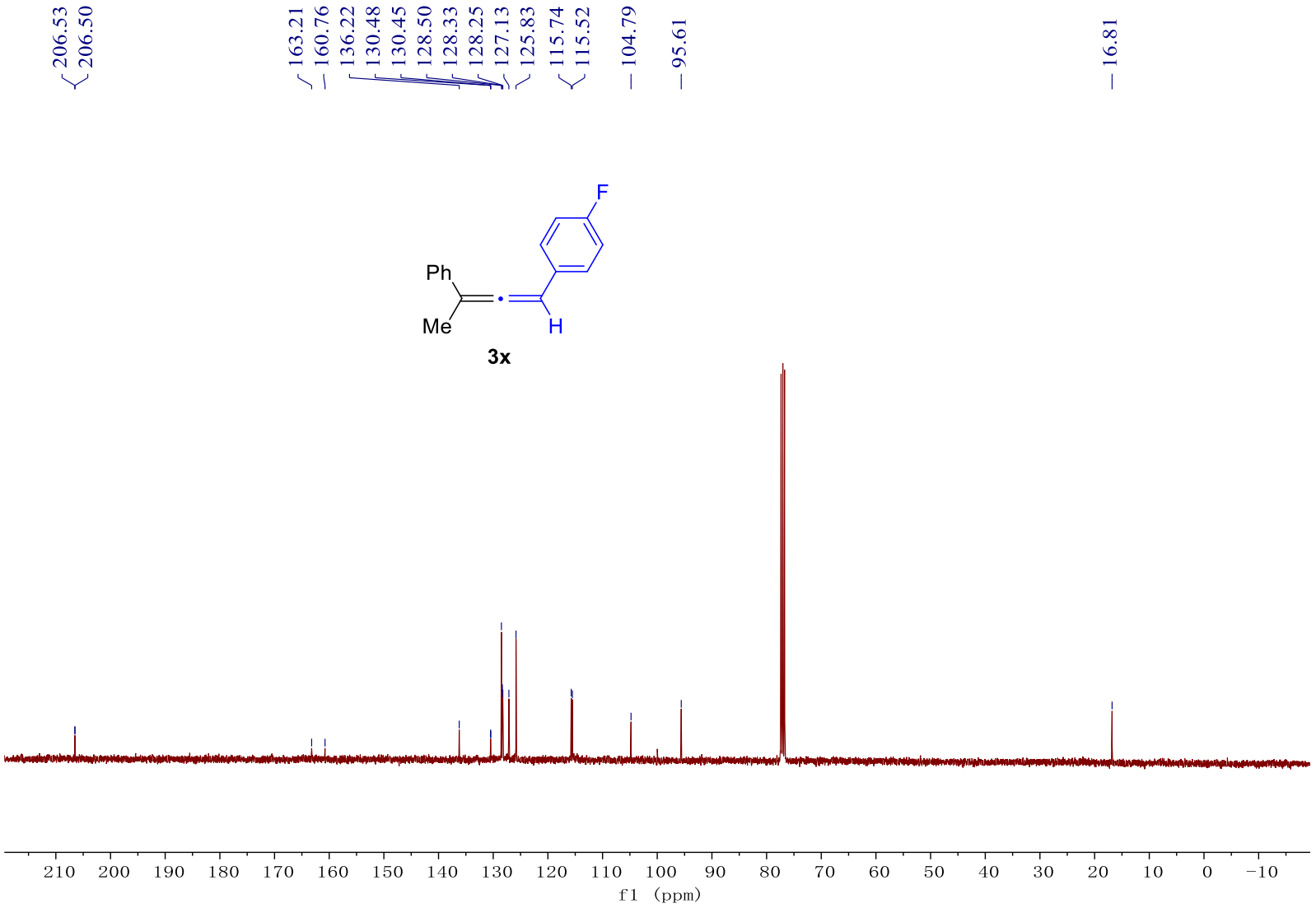
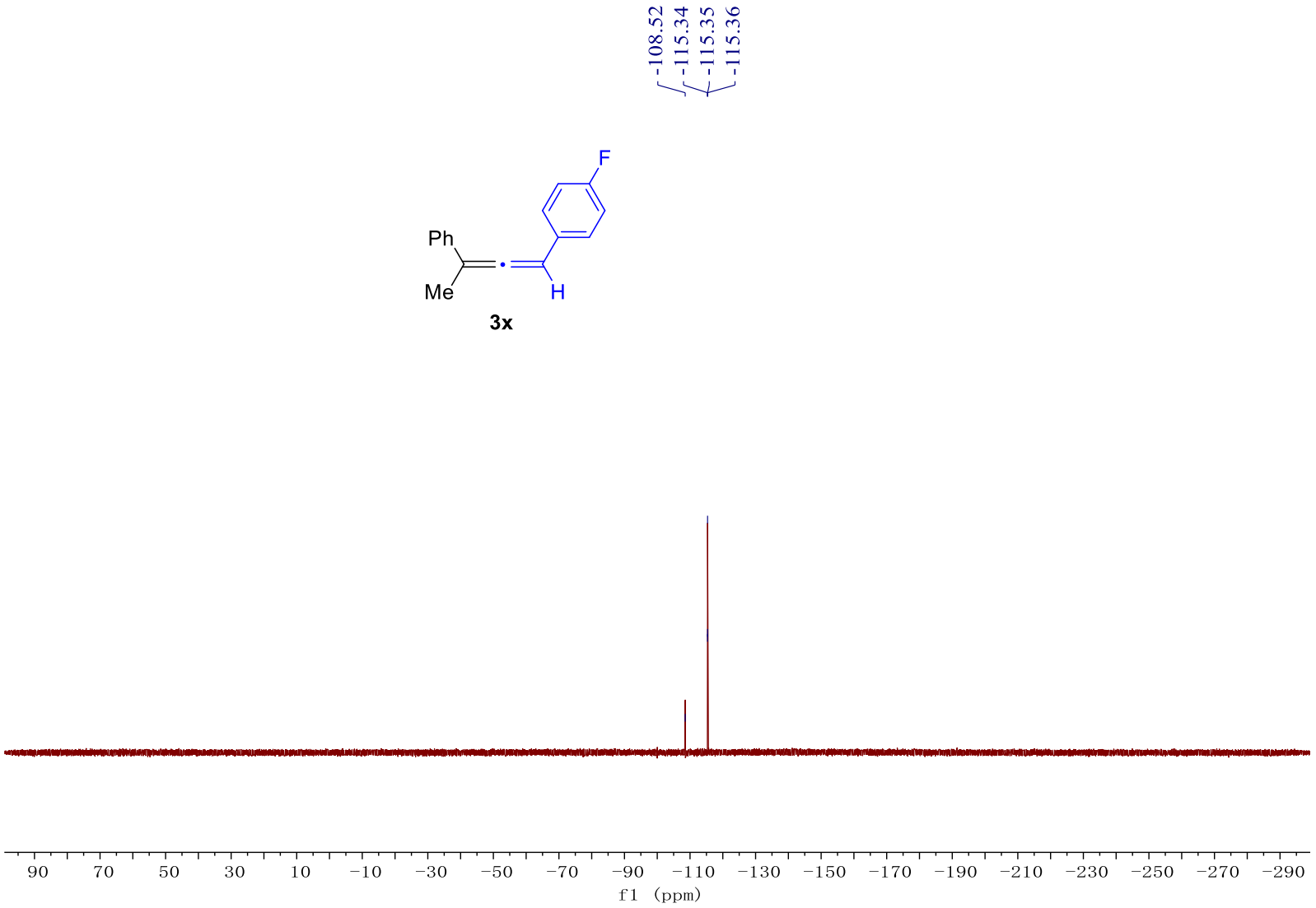
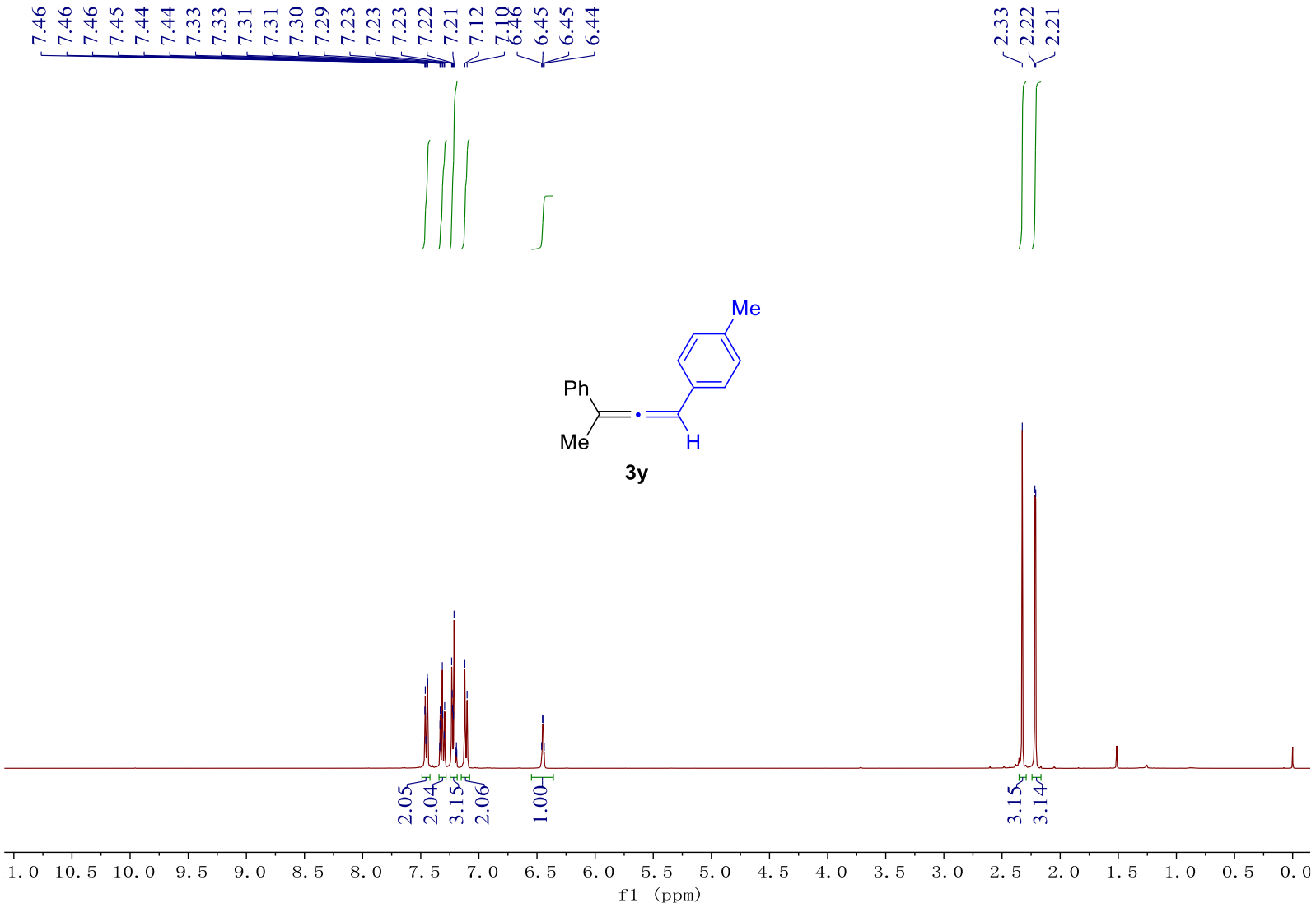
7.30
7.29
7.28
7.28
7.28
7.27
7.26
7.25
7.25
7.25
7.25
7.24
7.23
7.21
7.01
7.01
7.00
6.99
6.98
6.97
6.97
6.45
6.45
6.44
6.43
2.23
2.22
F
Ph
Me
H
3x
2.09
5.28
2.02
1.00
3.02
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


206.53
206.50
163.21
160.76
136.22
130.48
130.45
128.50
128.33
128.25
127.13
125.83
115.74
115.52
104.79
95.61
16.81
F
Ph
Me
H
3x
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


-108.52
-115.34
-115.35
-115.36
F
Ph
Me
H
3x
90
70
50
30
10
−10
−30
−50
−70
−90
−110
−130
−150
−170
−190
−210
−230
−250
−270
−290
f1 (ppm)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7.46
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7.45
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7.44
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7.33
7.31
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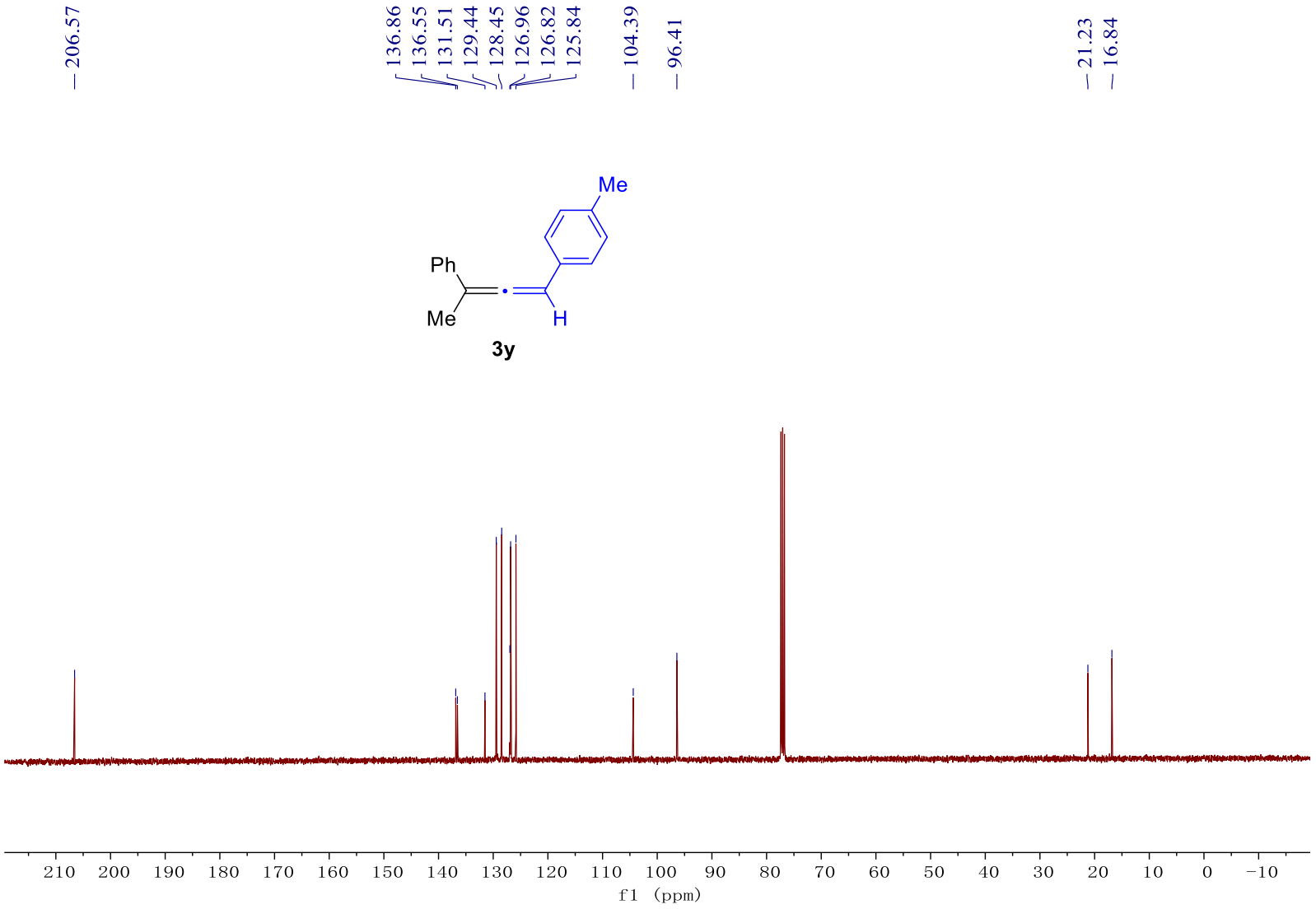
7.29
7.23
7.23
7.23
7.22
7.21
7.12
7.10
6.46
6.45
6.45
6.44
2.33
2.22
2.21
Me
Ph
Me
H
3y
2.05
2.04
3.15
2.06
1.00
3.15
3.14
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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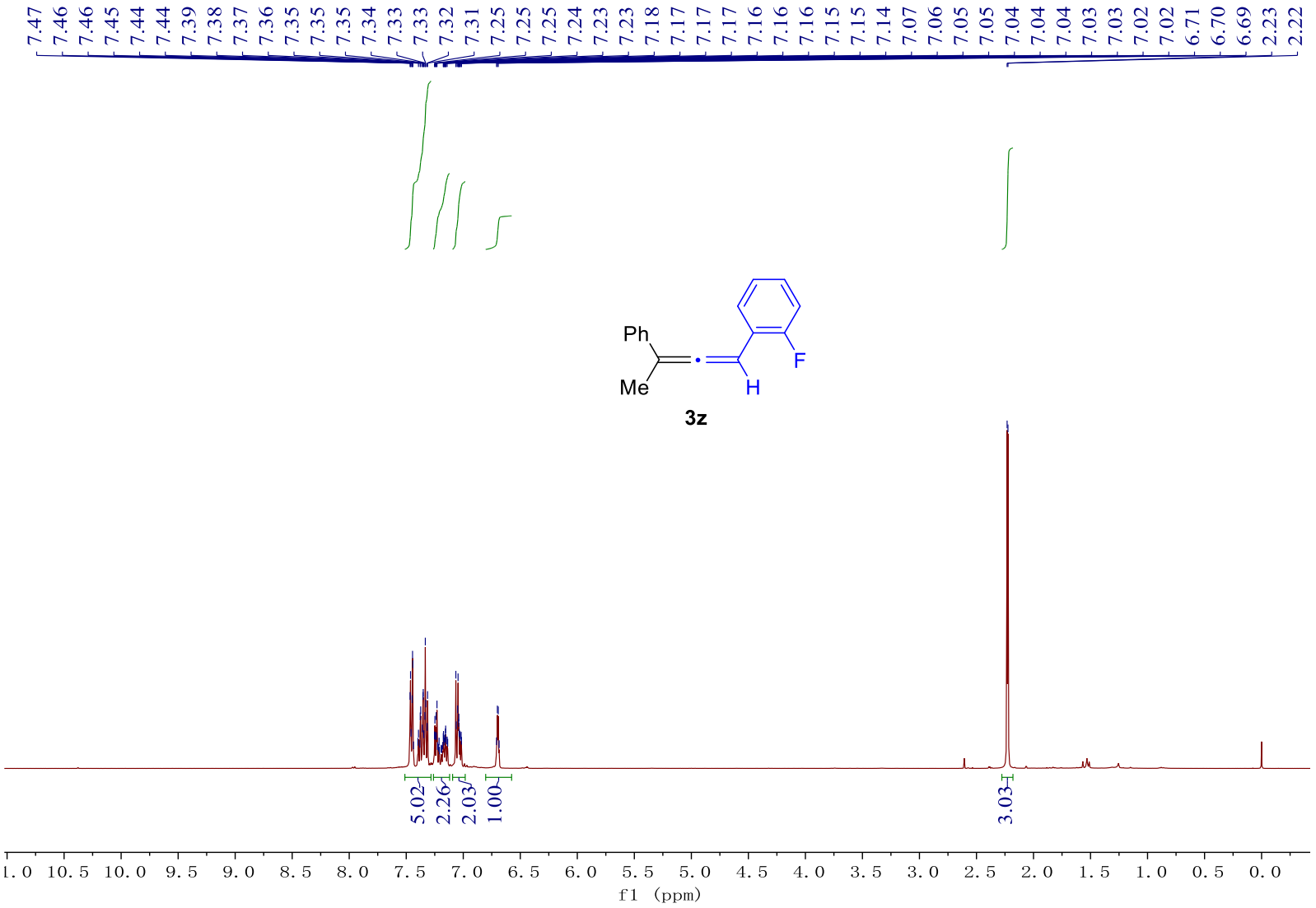
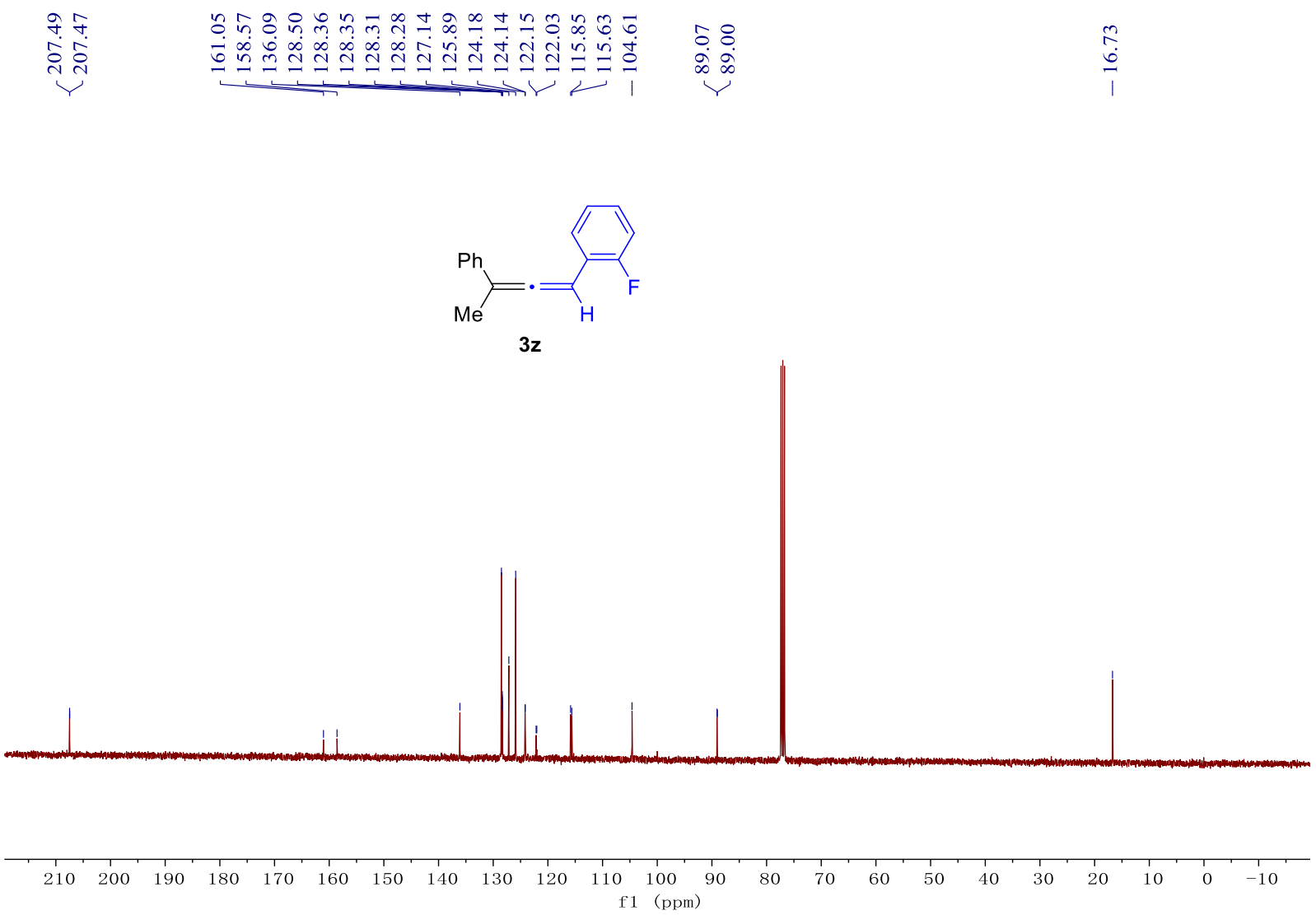
136.55
131.51
129.44
128.45
126.96
126.82
125.84
104.39
96.41
21.23
16.84
Me
Ph
Me
H
3y
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


7.47 7.46 7.46 7.45 7.44 7.44 7.39 7.38 7.37 7.36 7.35 7.35 7.35 7.34 7.33 7.33 7.32 7.31 7.25 7.25 7.25 7.24 7.23 7.23 7.18 7.17 7.17 7.17 7.16 7.16 7.16 7.15 7.15 7.14 7.07 7.06 7.05 7.05 7.04 7.04 7.04 7.03 7.03 7.02 7.02 6.71 6.70 6.69 2.23 2.22
Ph
Me
F
H
3z
5.02
2.26
2.03
1.00
3.03
f1 (ppm)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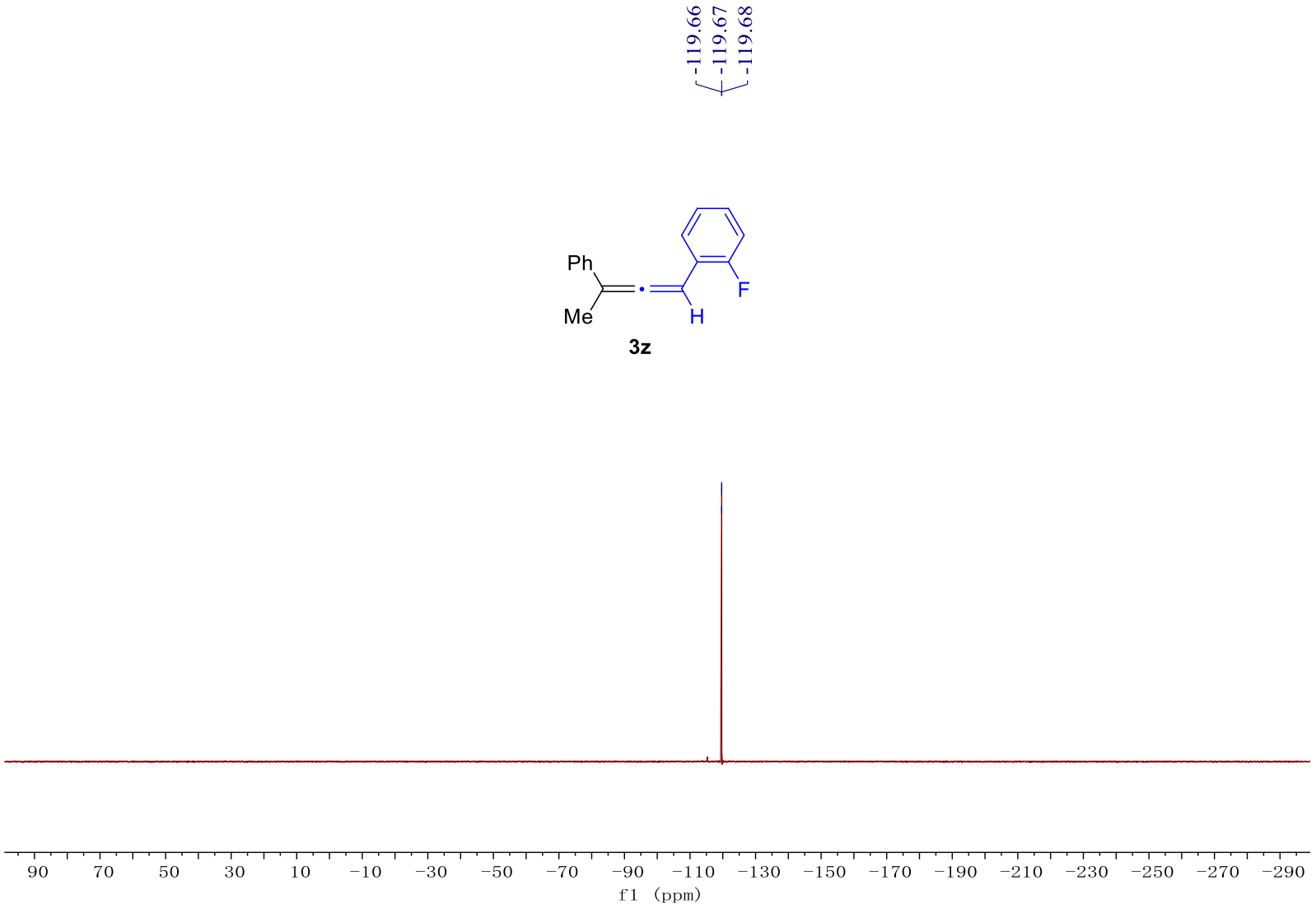
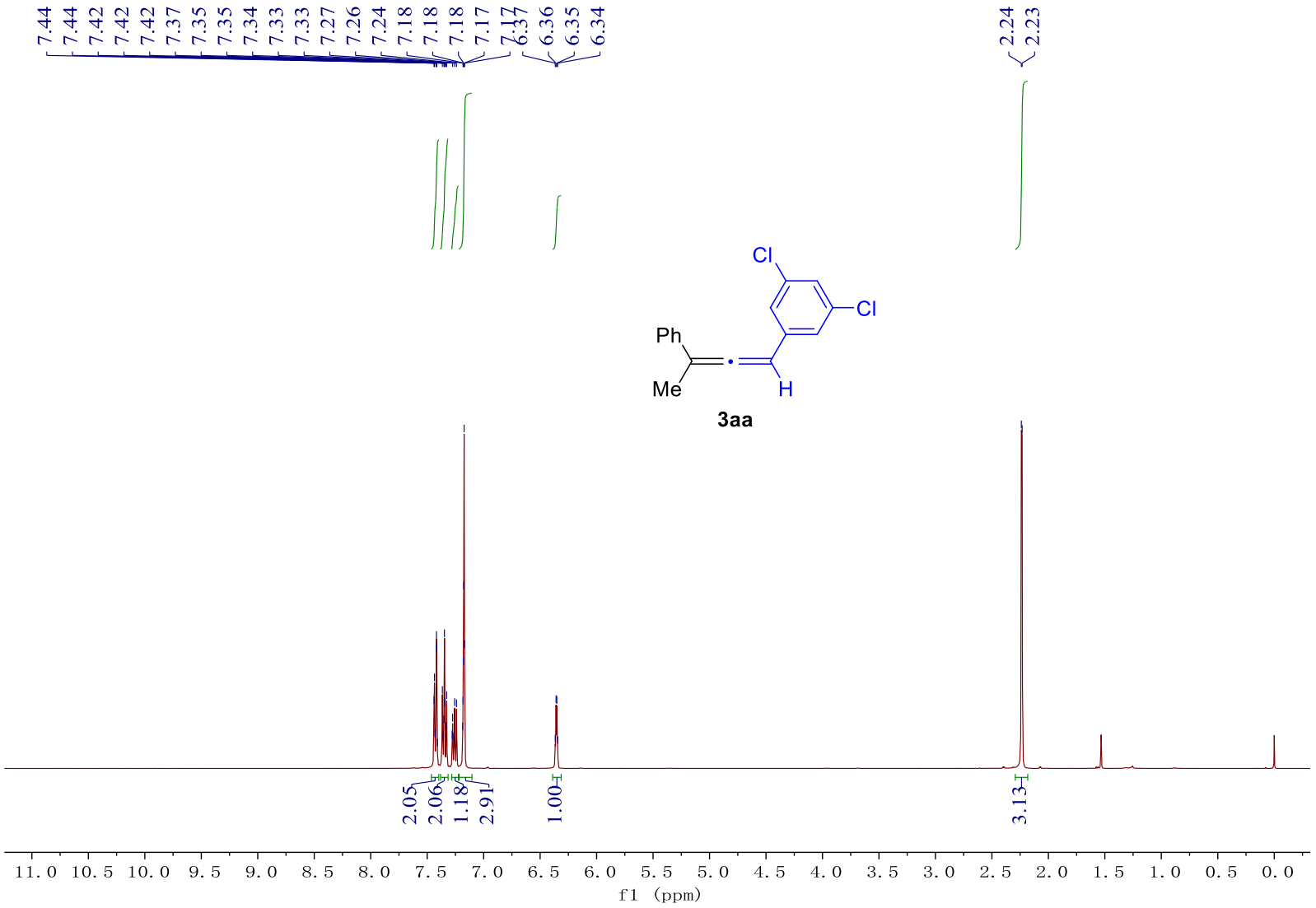
161.05
158.57
136.09
128.50
128.36
128.35
128.31
128.28
127.14
125.89
124.18
124.14
122.15
122.03
115.85
115.63
104.61
89.07
89.00
16.73
Ph
Me
F
H
3z
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


-119.66
-119.67
-119.68
Ph
Me
H
F
3z
90
70
50
30
10
−10
−30
−50
−70
−90
−110
−130
−150
−170
−190
−210
−230
−250
−270
−290
f1 (ppm)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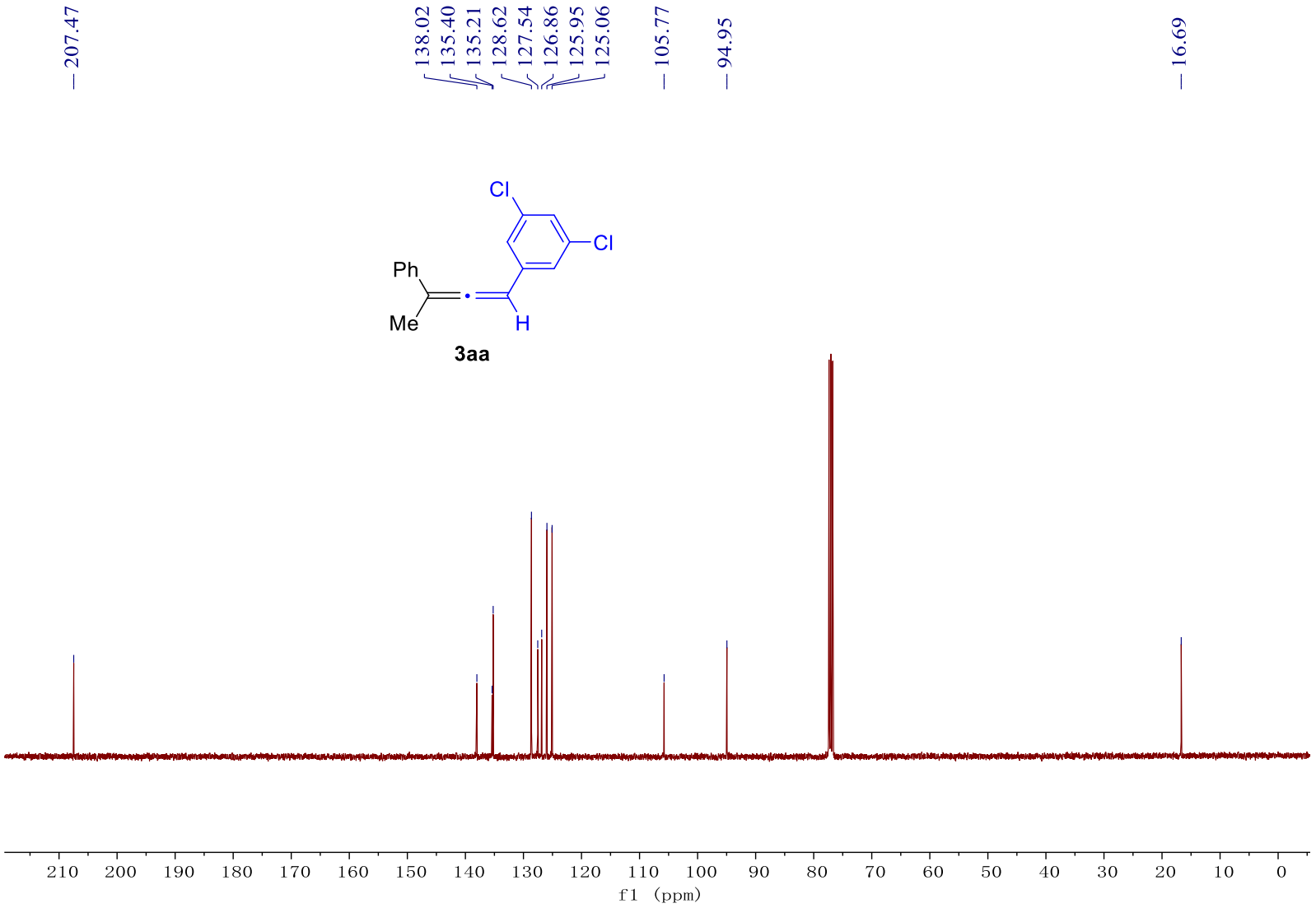
7.35
7.35
7.34
7.33
7.33
7.27
7.26
7.24
7.18
7.18
7.18
7.17
7.17
6.37
6.36
6.35
6.34
2.24
2.23
Cl
Cl
Ph
Me
H
3aa
2.05
2.06
1.18
2.91
1.00
3.13
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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138.02
135.40
135.21
128.62
127.54
126.86
125.95
125.06
105.77
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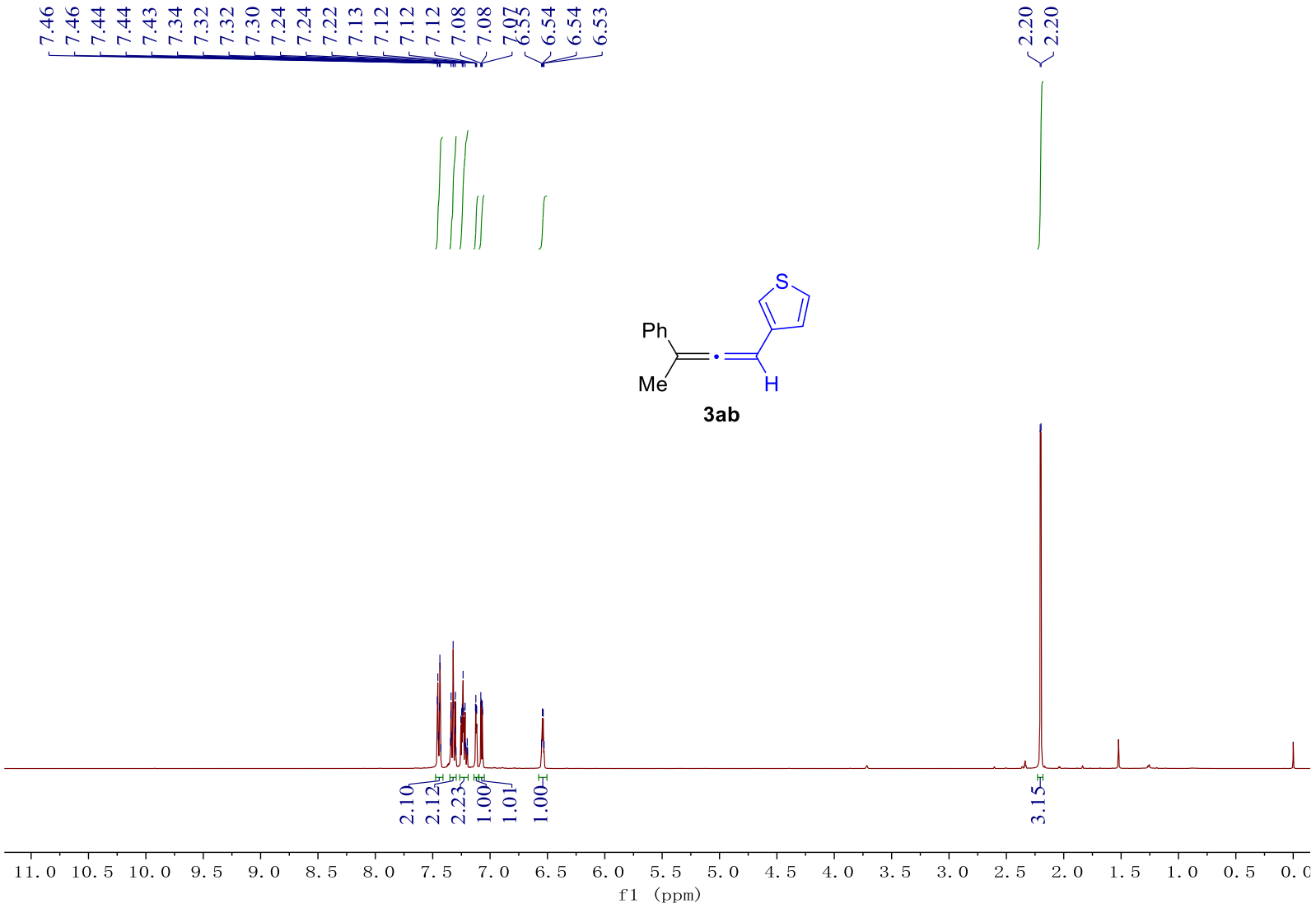
Cl
Cl
Ph
Me
H
3aa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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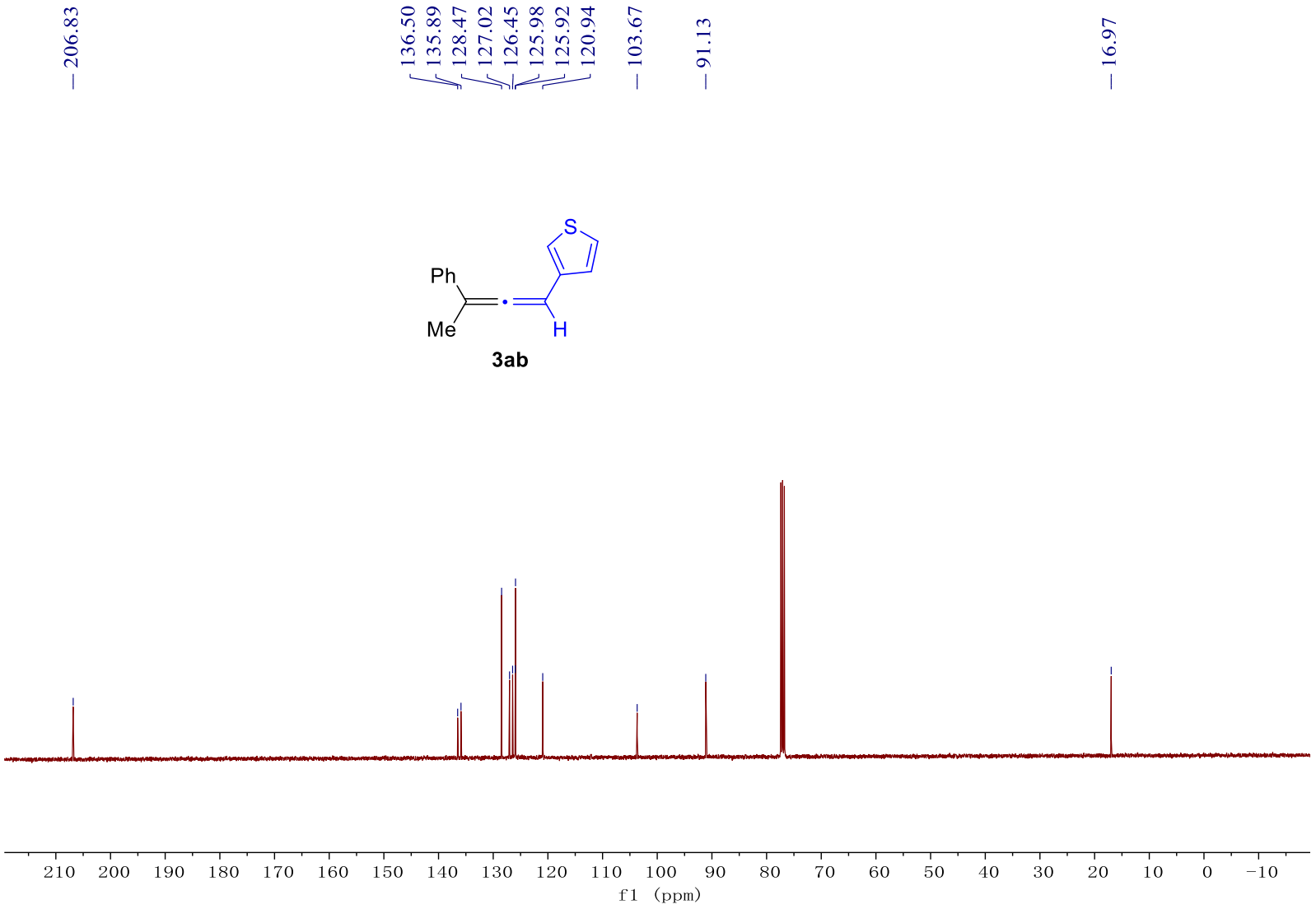
7.32
7.32
7.30
7.24
7.24
7.22
7.13
7.12
7.12
7.12
7.08
7.08
7.07
6.55
6.54
6.54
6.53
2.20
2.20
S
Ph
Me
H
3ab
2.10
2.12
2.23
1.00
1.01
1.00
3.15
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


206.83
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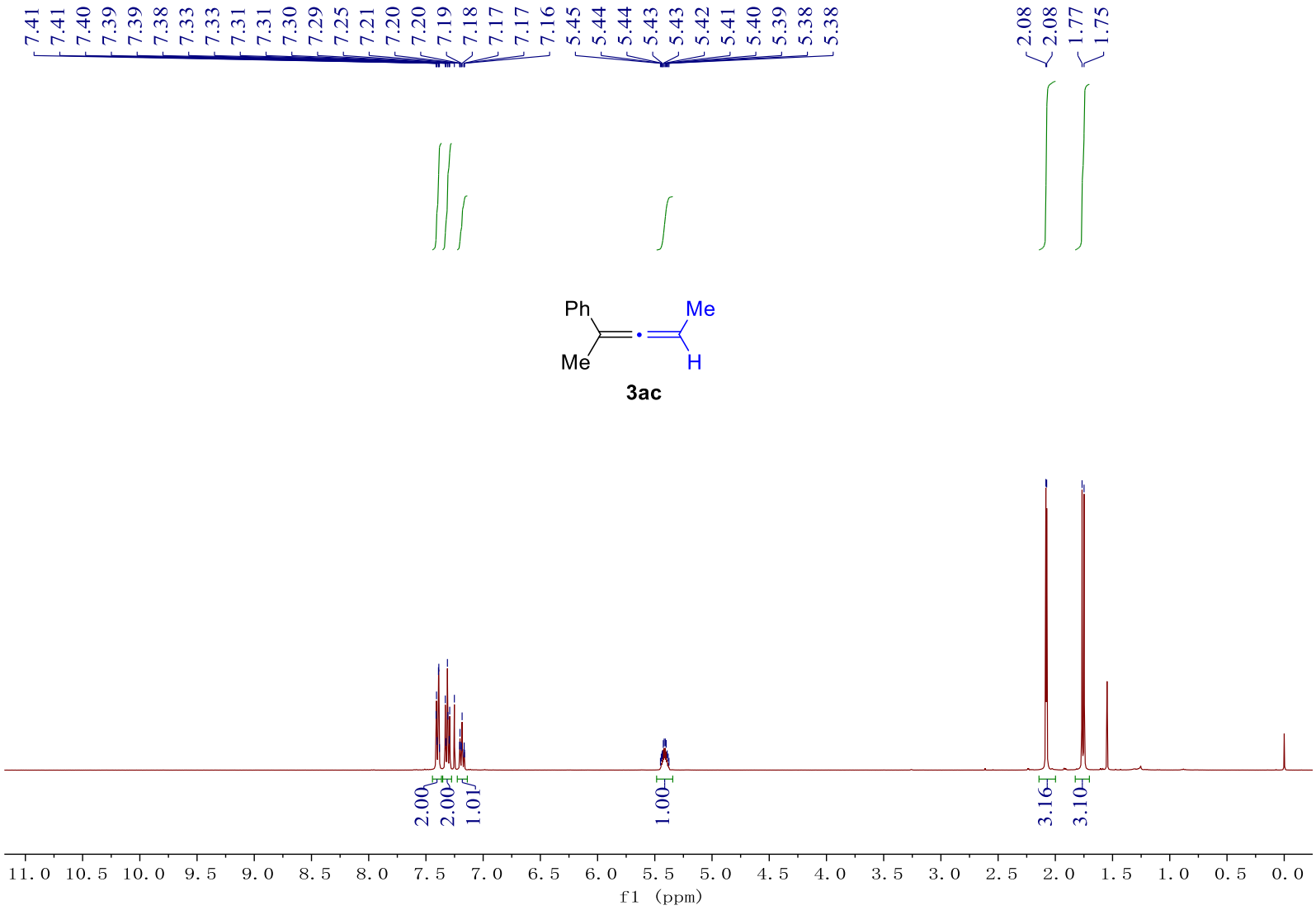
128.47
127.02
126.45
125.98
125.92
120.94
103.67
91.13
16.97
S
Ph
Me
H
3ab
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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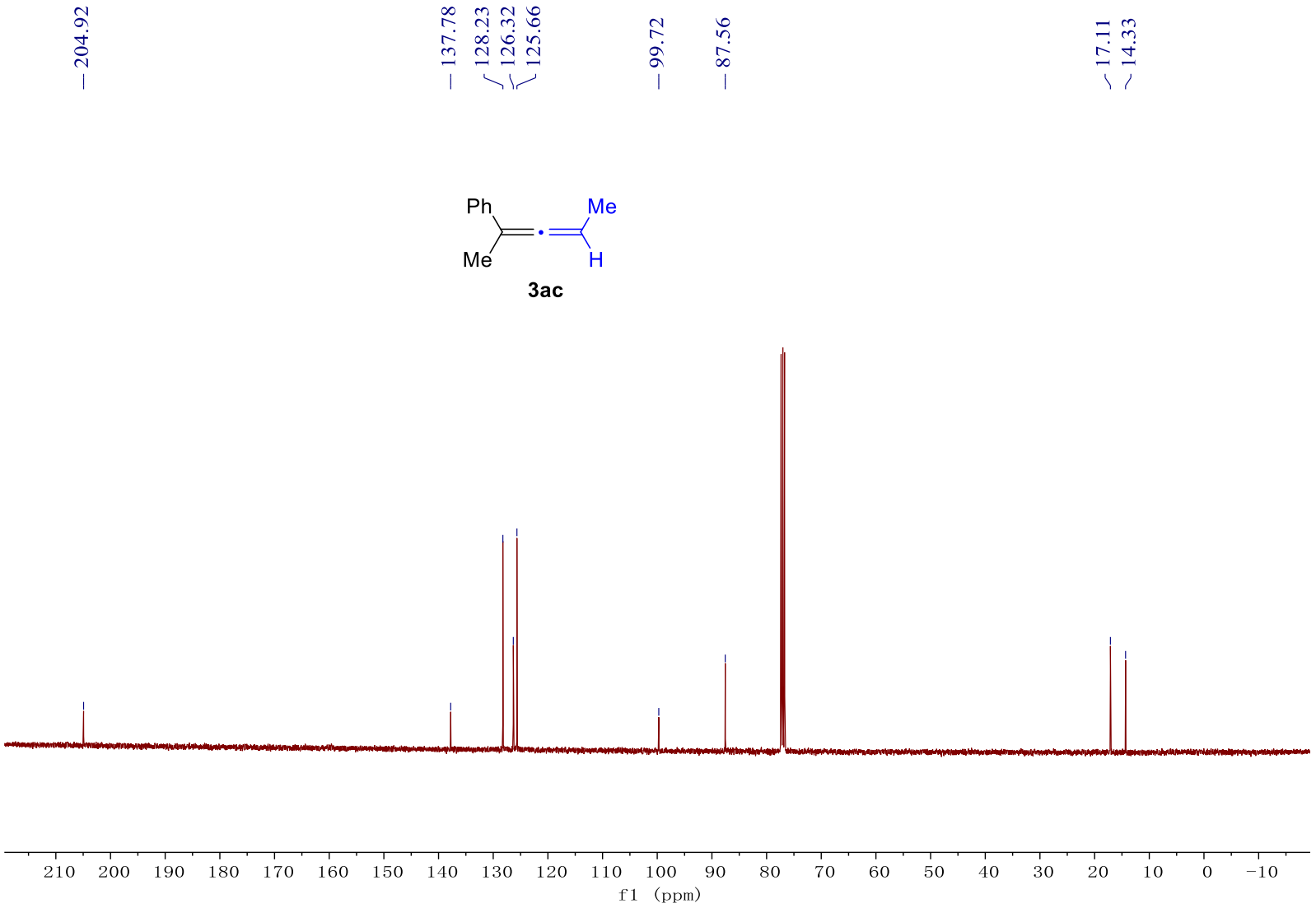
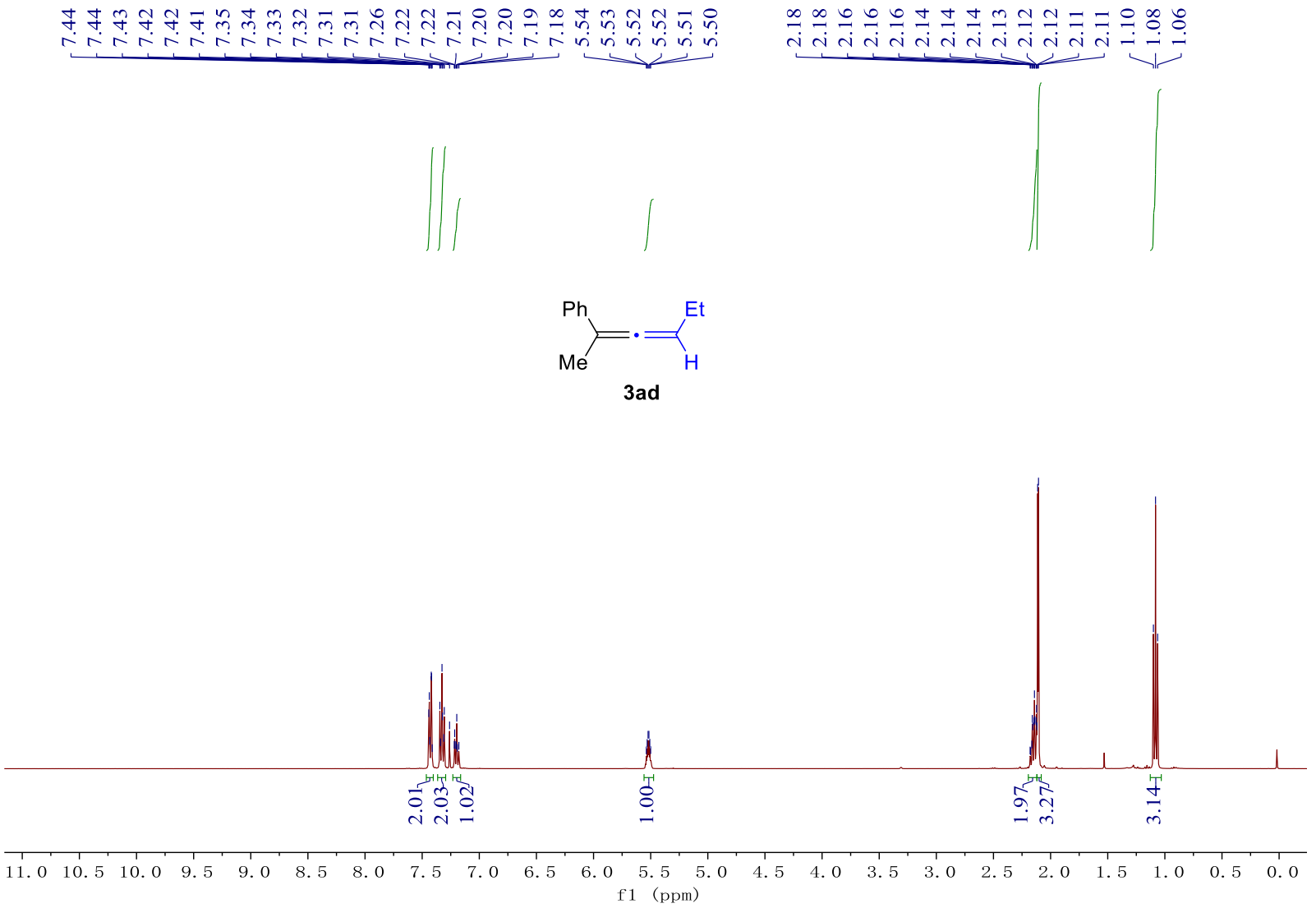
7.38
7.33
7.33
7.31
7.31
7.30
7.29
7.25
7.21
7.20
7.20
7.19
7.18
7.17
7.17
7.16
5.45
5.44
5.44
5.43
5.43
5.42
5.41
5.40
5.39
5.38
5.38
2.08
2.08
1.77
1.75
Ph
Me
Me
H
3ac
2.00
2.00
1.01
1.00
3.16
3.10
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


204.92
137.78
128.23
126.32
125.66
99.72
87.56
17.11
14.33
Ph
Me
Me
H
3ac
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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7.44
7.43
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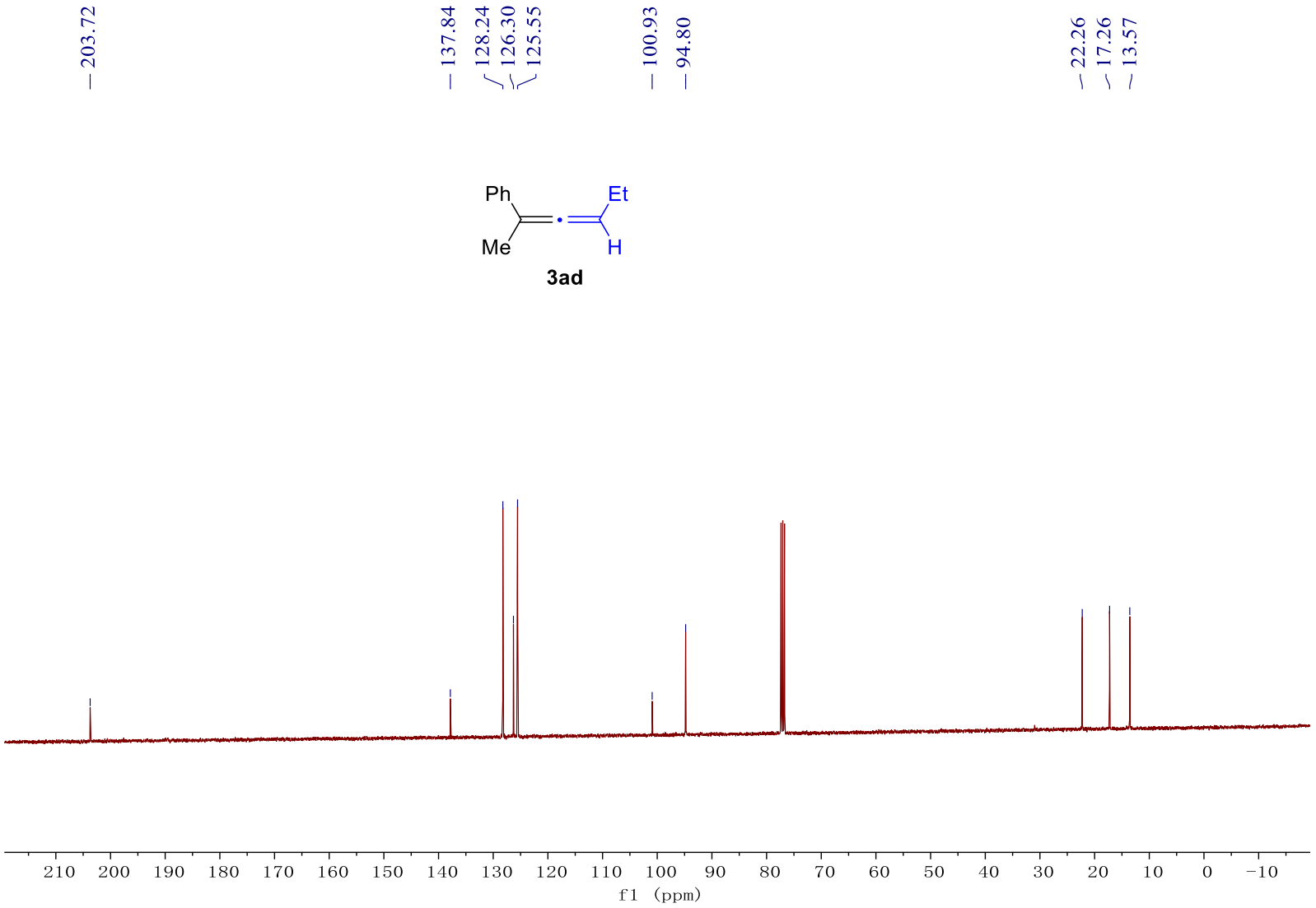
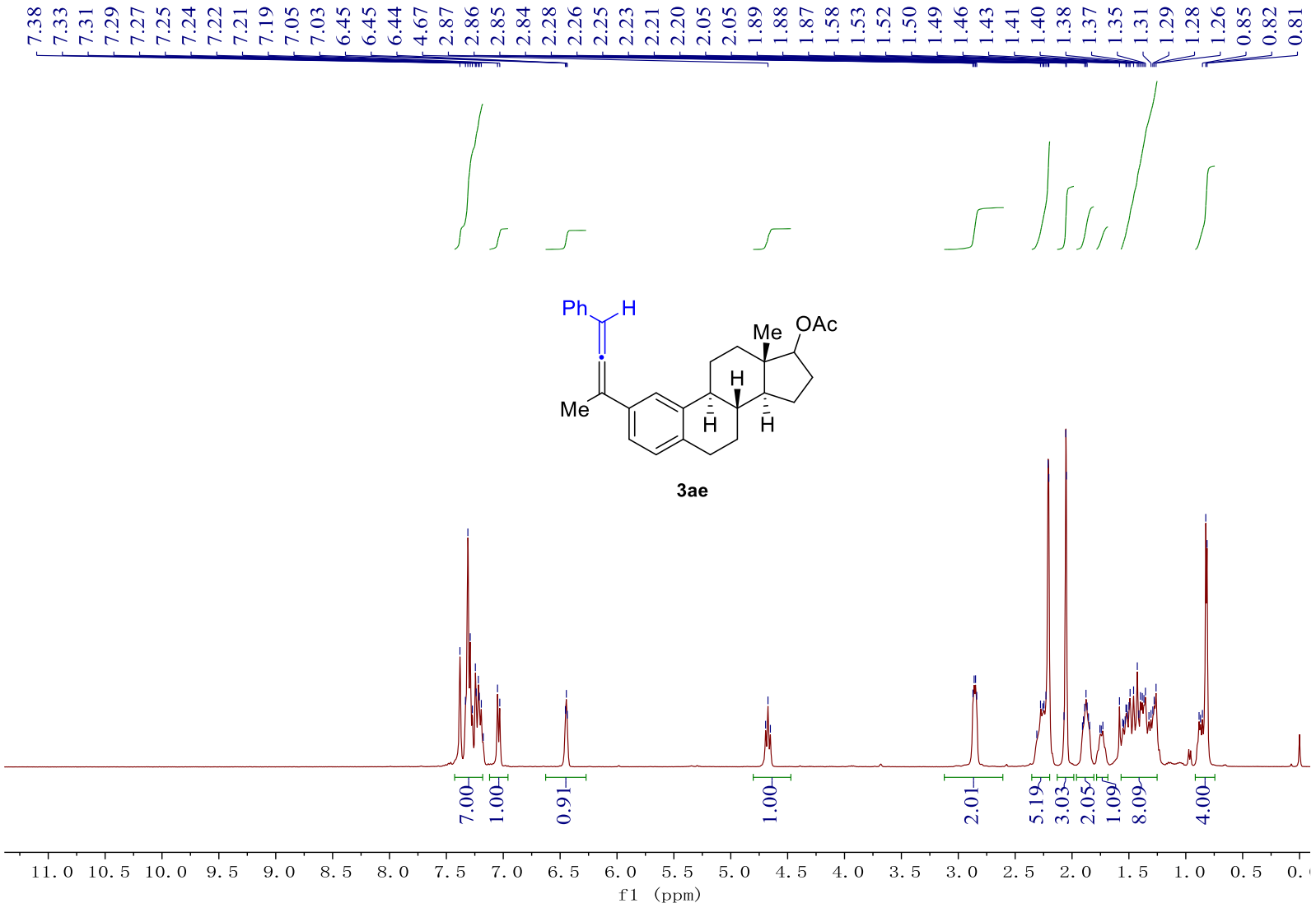
7.42
7.41
7.35
7.34
7.33
7.32
7.31
7.31
7.26
7.22
7.22
7.21
7.20
7.20
7.19
7.18
5.54
5.53
5.52
5.52
5.51
5.50
2.18
2.18
2.16
2.16
2.16
2.14
2.14
2.14
2.13
2.12
2.12
2.11
2.11
1.10
1.08
1.06
Ph
Et
Me
H
3ad
2.01
2.03
1.02
1.00
1.97
3.27
3.14
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


203.72
137.84
128.24
126.30
125.55
100.93
94.80
22.26
17.26
13.57
Ph
Et
Me
H
3ad
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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7.31
7.29
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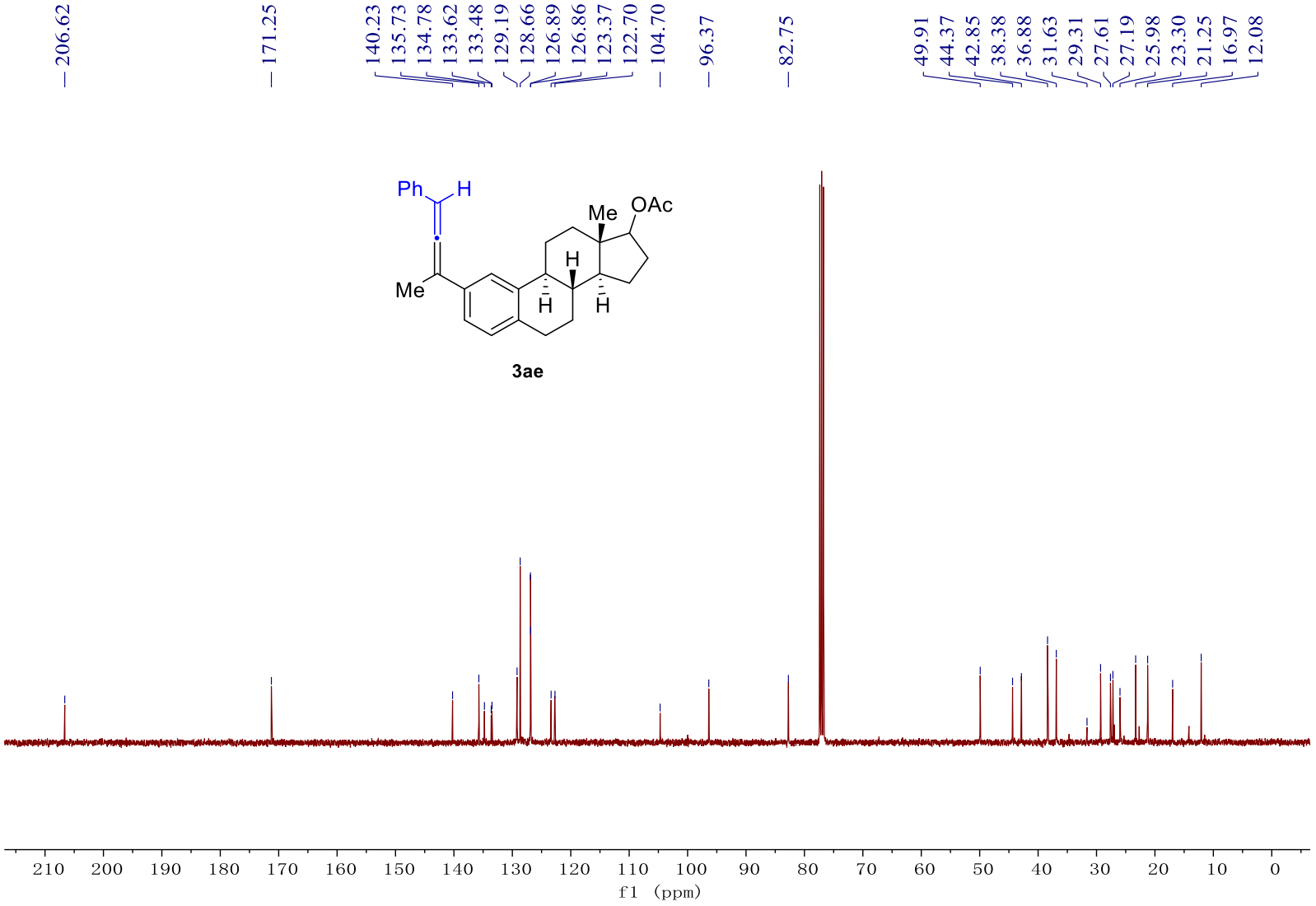
7.25
7.24
7.22
7.21
7.19
7.05
7.03
6.45
6.45
6.44
4.67
2.87
2.86
2.85
2.84
2.28
2.26
2.25
2.23
2.21
2.20
2.05
2.05
1.89
1.88
1.87
1.58
1.53
1.52
1.50
1.49
1.46
1.43
1.41
1.40
1.38
1.37
1.35
1.31
1.29
1.28
1.26
0.85
0.82
0.81
Ph
H
Me
OAc
H
Me
H
H
3ae
7.00
1.00
0.91
1.00
2.01
5.19
3.03
2.05
1.09
8.09
4.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)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140.23
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134.78
133.62
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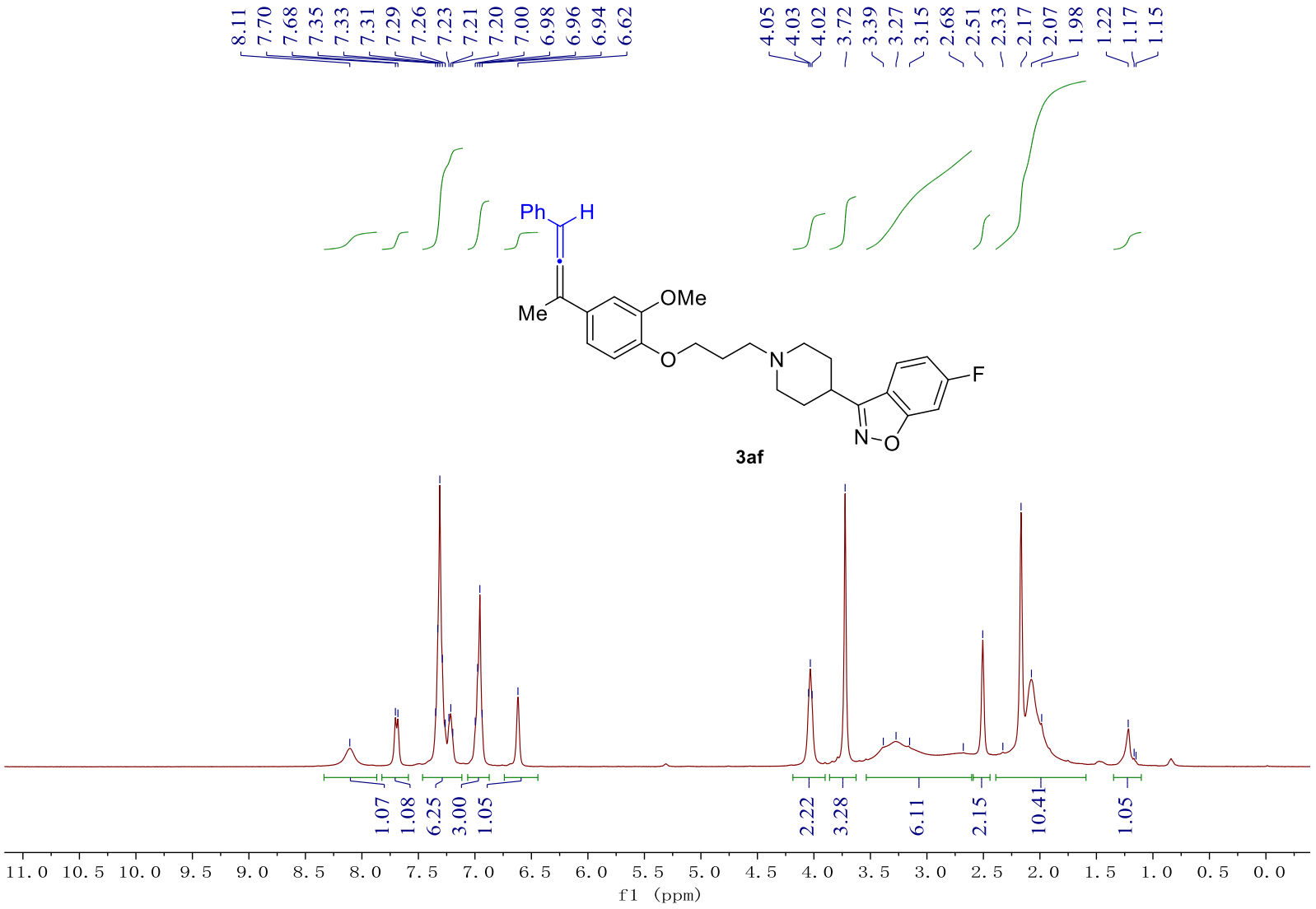
129.19
128.66
126.89
126.86
123.37
122.70
104.70
96.37
82.75
49.91
44.37
42.85
38.38
36.88
31.63
29.31
27.61
27.19
25.98
23.30
21.25
16.97
12.08
Ph
H
Me
OAc
H
Me
H
H
3ae
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.11
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7.68
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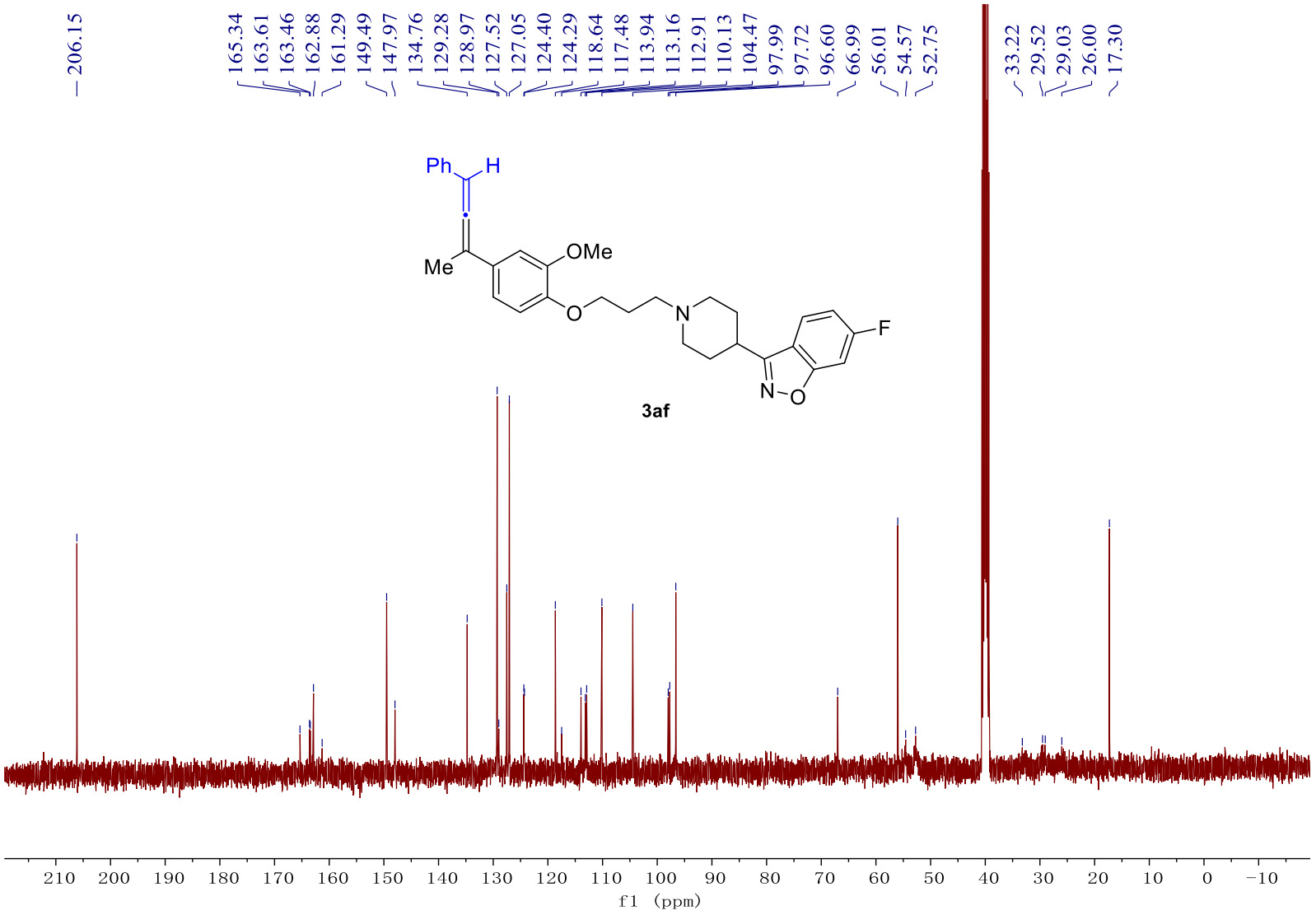
7.31
7.29
7.26
7.23
7.21
7.20
7.00
6.98
6.96
6.94
6.62
4.05
4.03
4.02
3.72
3.39
3.27
3.15
2.68
2.51
2.33
2.17
2.07
1.98
1.22
1.17
1.15
Ph
H
Me
OMe
O
N
F
N
O
3af
1.07
1.08
6.25
3.00
1.05
2.22
3.28
6.11
2.15
10.41
1.05
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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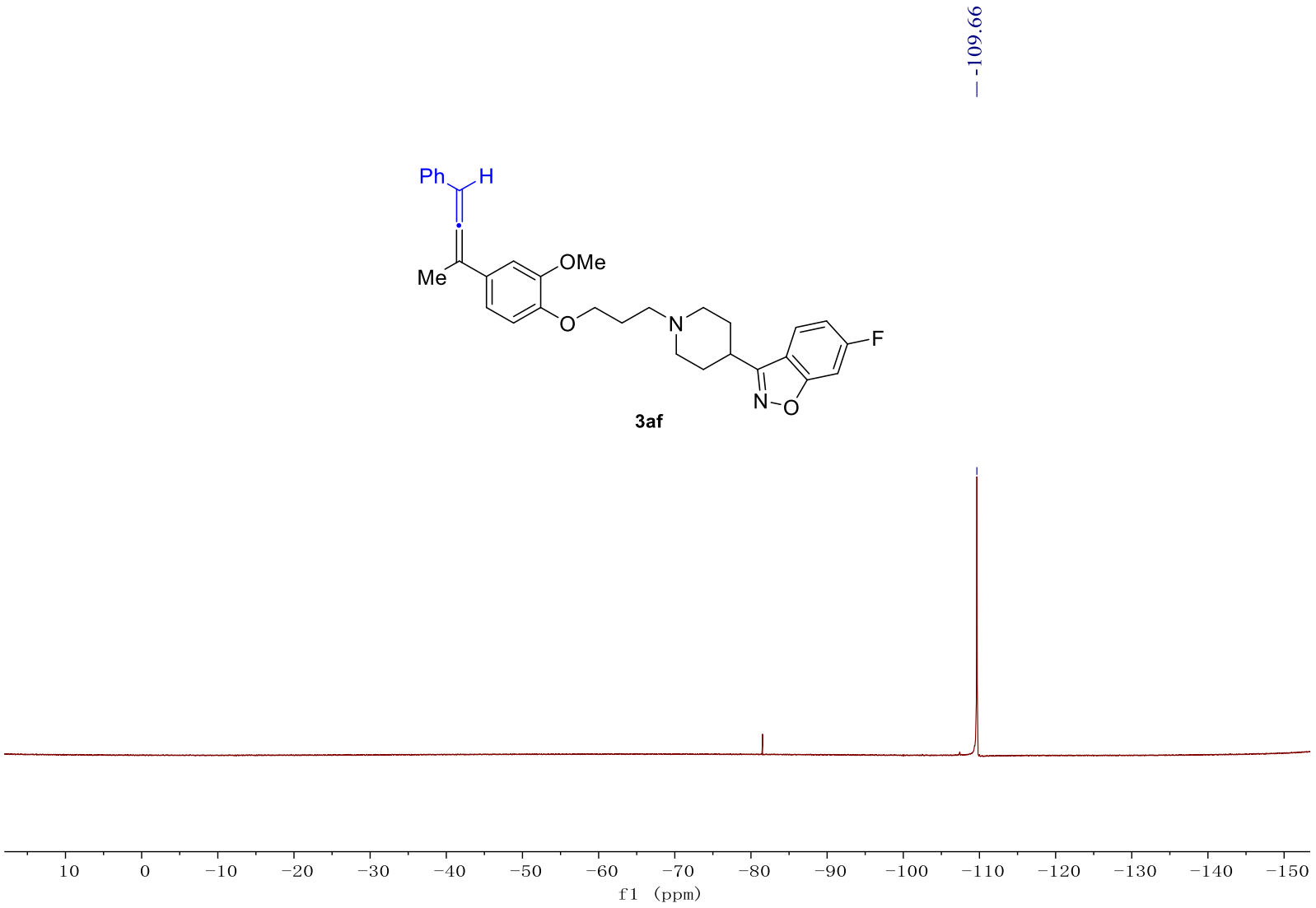
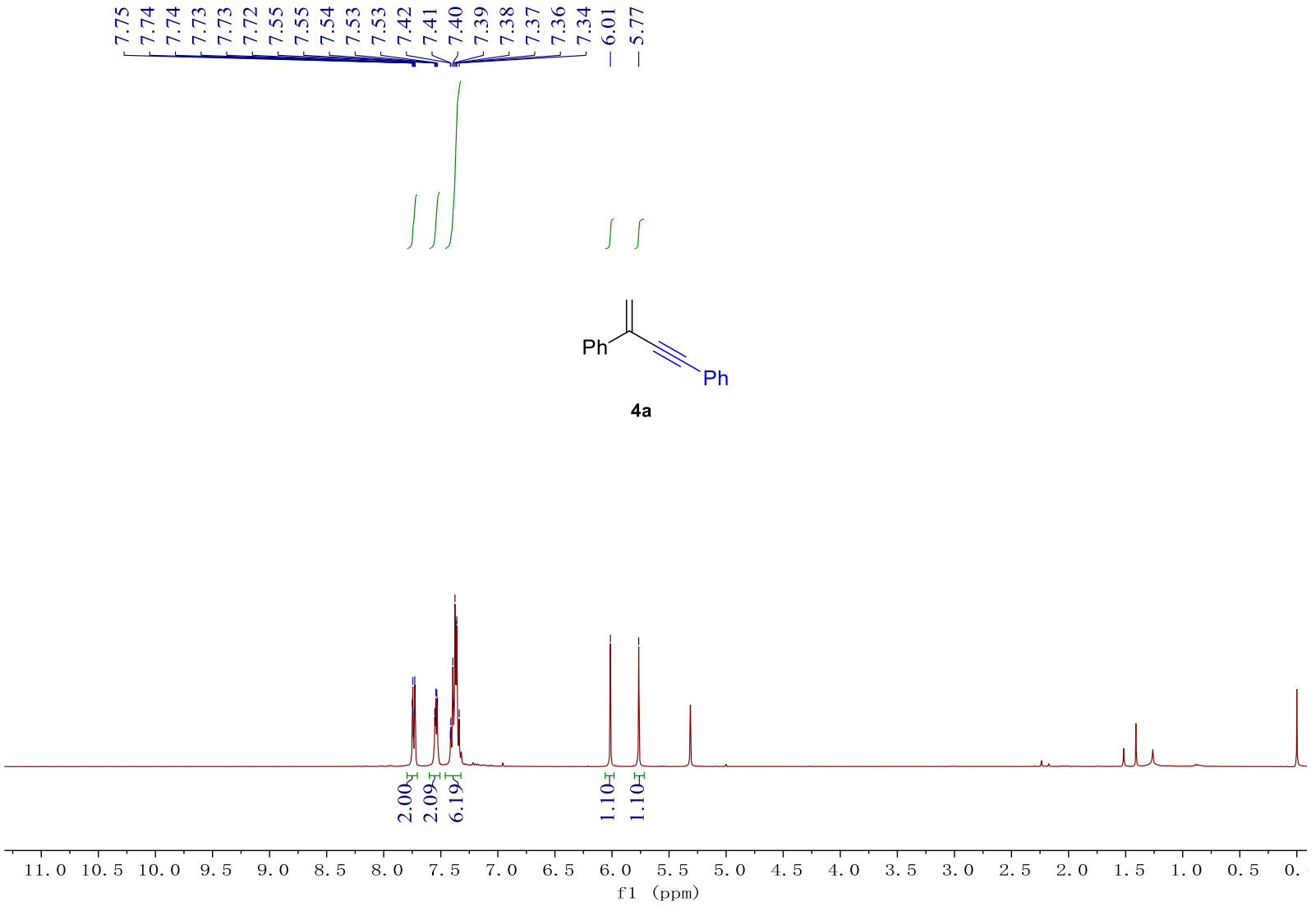
161.29
149.49
147.97
134.76
129.28
128.97
127.52
127.05
124.40
124.29
118.64
117.48
113.94
113.16
112.91
110.13
104.47
97.99
97.72
96.60
66.99
56.01
54.57
52.75
33.22
29.52
29.03
26.00
17.30
Ph
H
Me
OMe
O
N
F
N
O
3af
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


-109.66
Ph
H
Me
OMe
O
N
F
N
O
3af
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
f1 (ppm)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7.72
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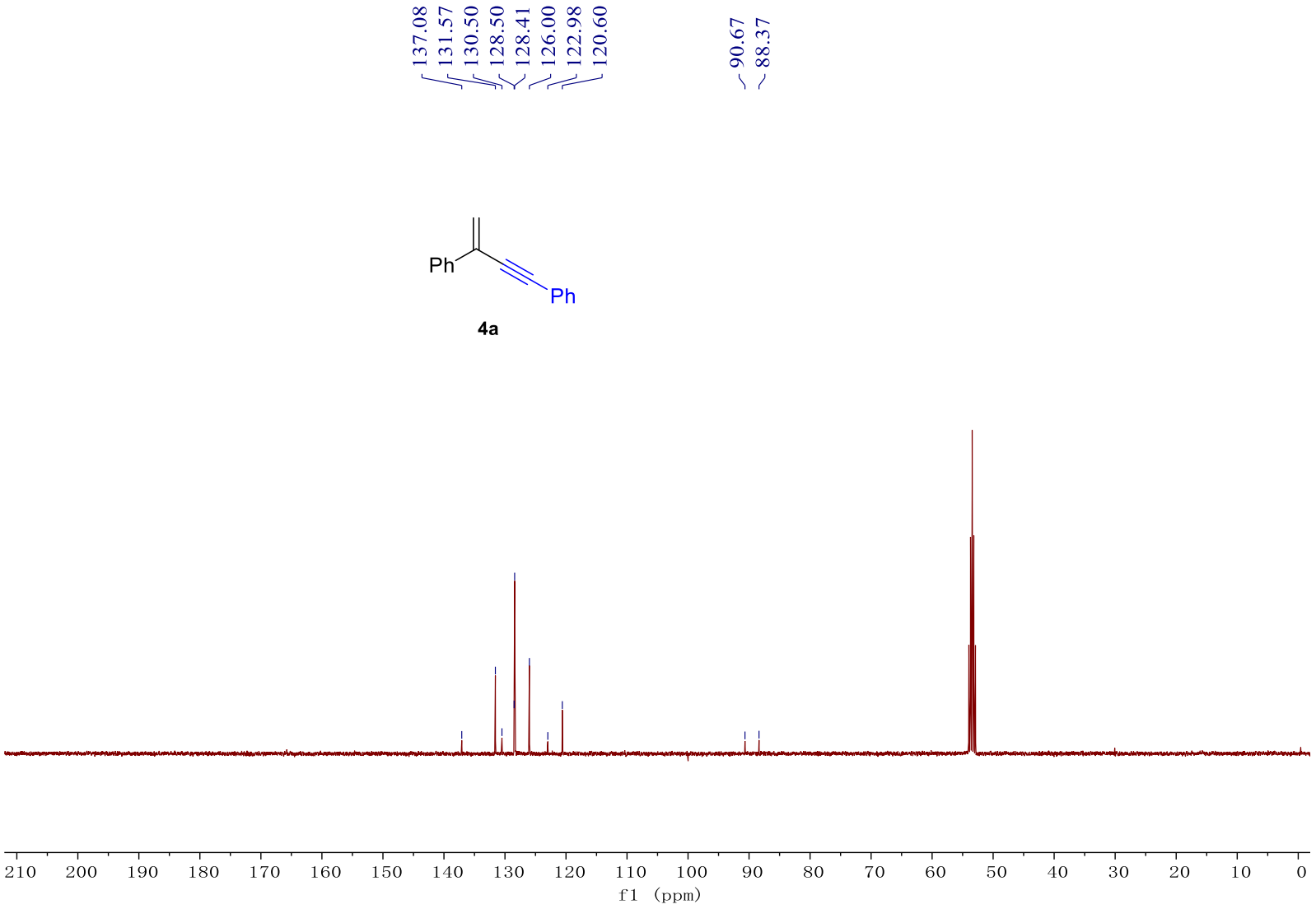
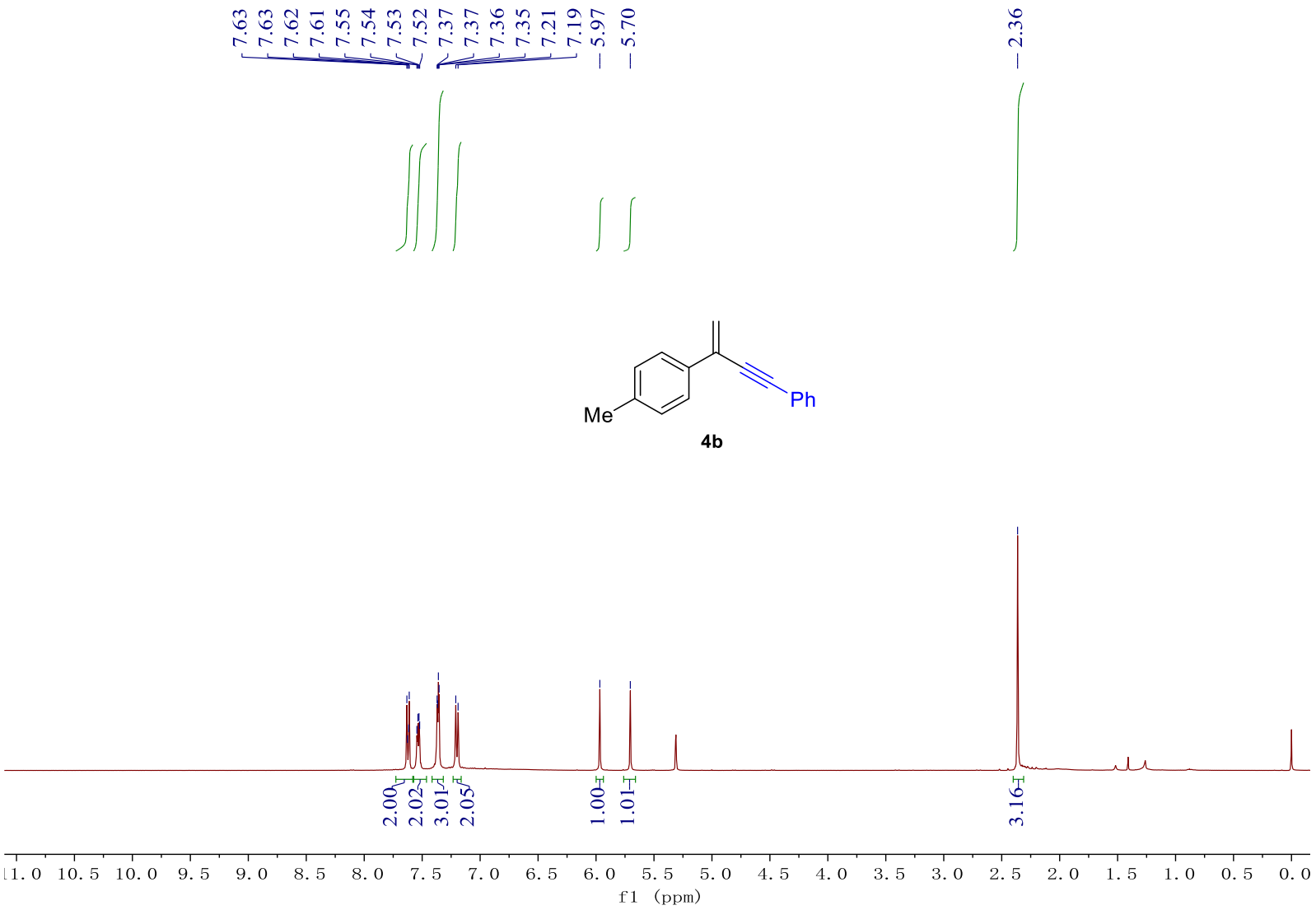
7.55
7.54
7.53
7.53
7.42
7.41
7.40
7.39
7.38
7.37
7.36
7.34
6.01
5.77
Ph
Ph
2.00
2.09
6.19
1.10
1.10
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)

4a


137.08
131.57
130.50
128.50
128.41
126.00
122.98
120.60
90.67
88.37
Ph
Ph
4a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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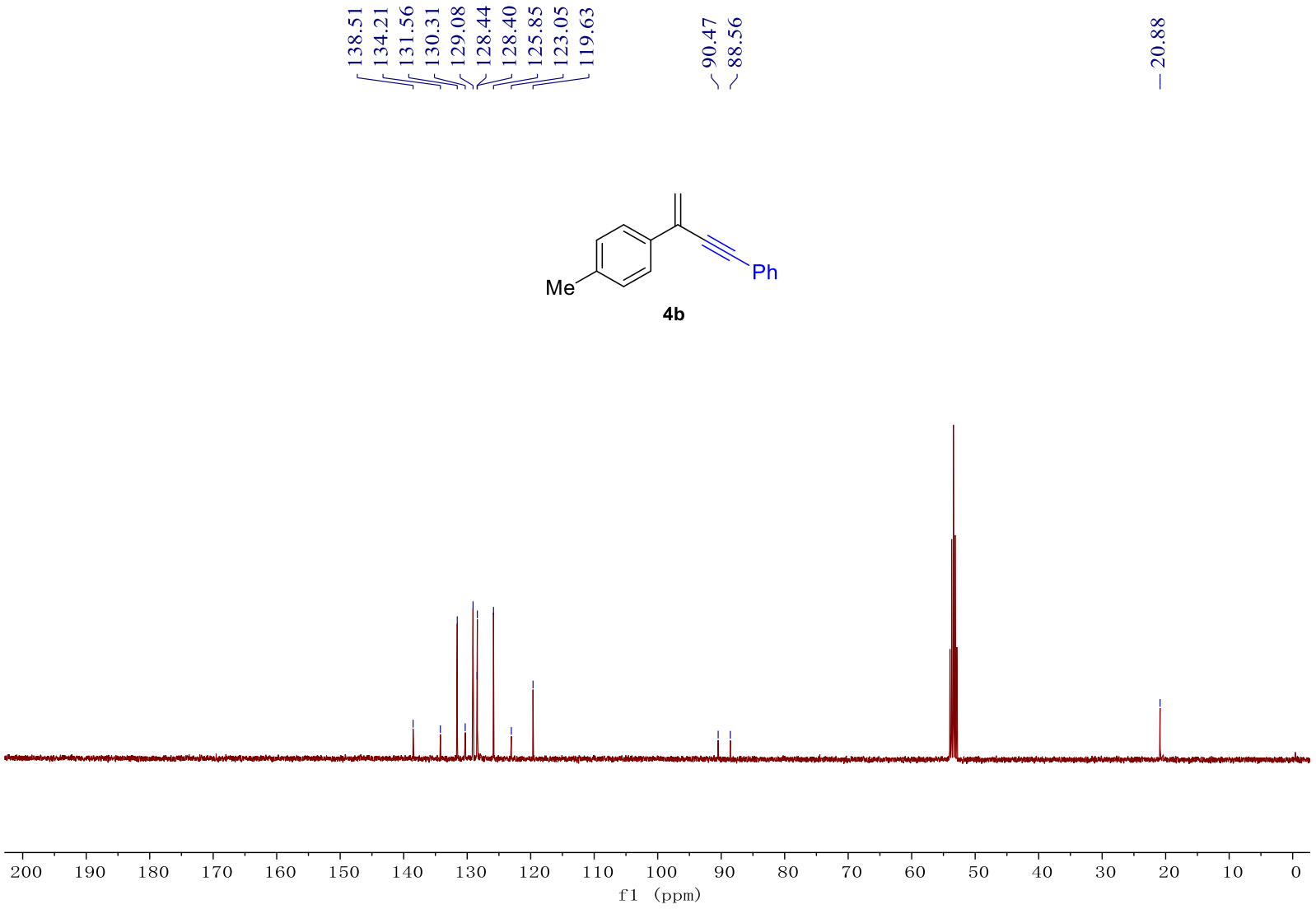
7.54
7.53
7.52
7.37
7.37
7.36
7.35
7.21
7.19
5.97
5.70
2.36
Me
Ph
4b
2.00
2.02
3.01
2.05
1.00
1.01
3.16
f1 (ppm)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134.21
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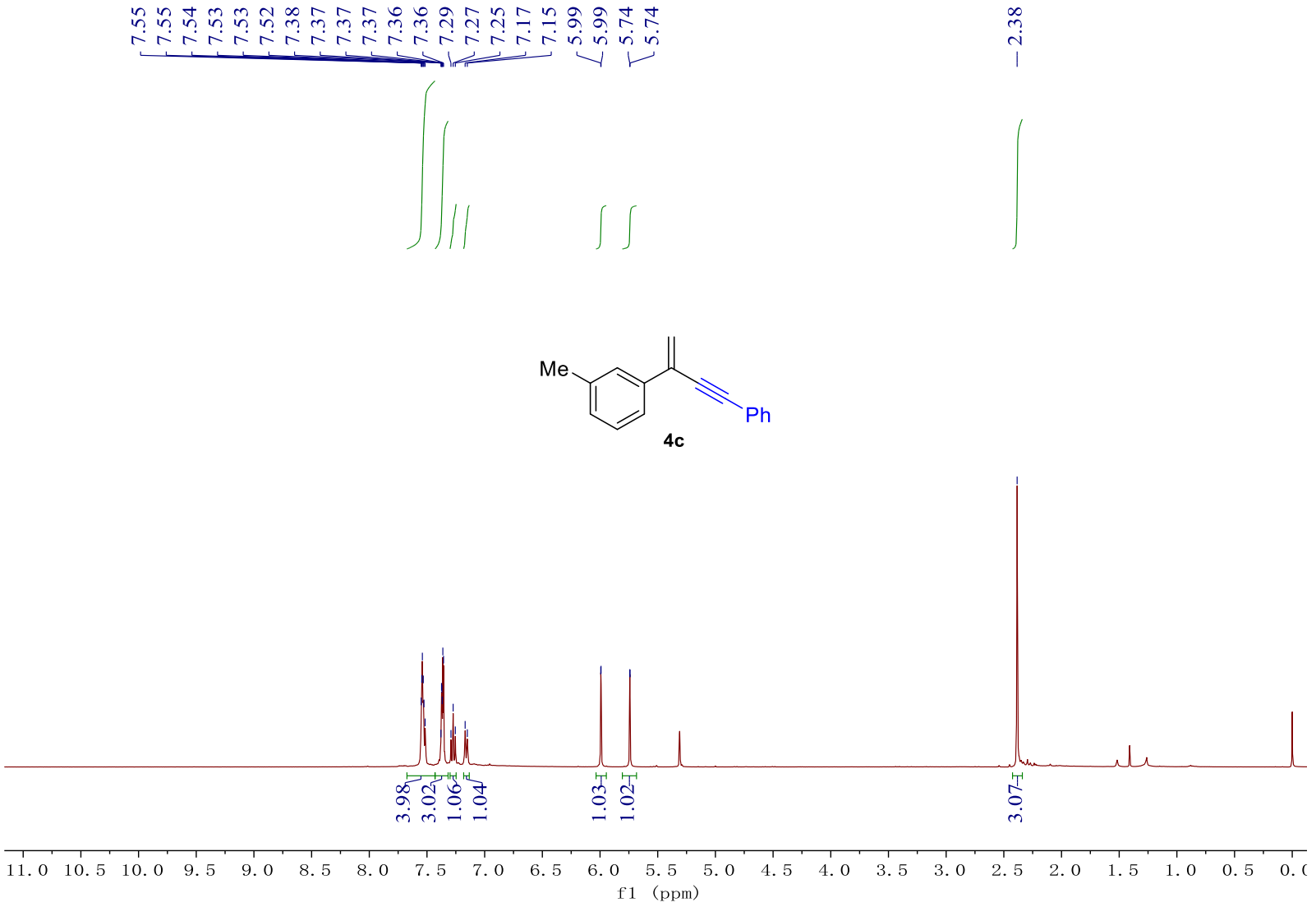
130.31
129.08
128.44
128.40
125.85
123.05
119.63
90.47
88.56
20.88
Me
Ph
4b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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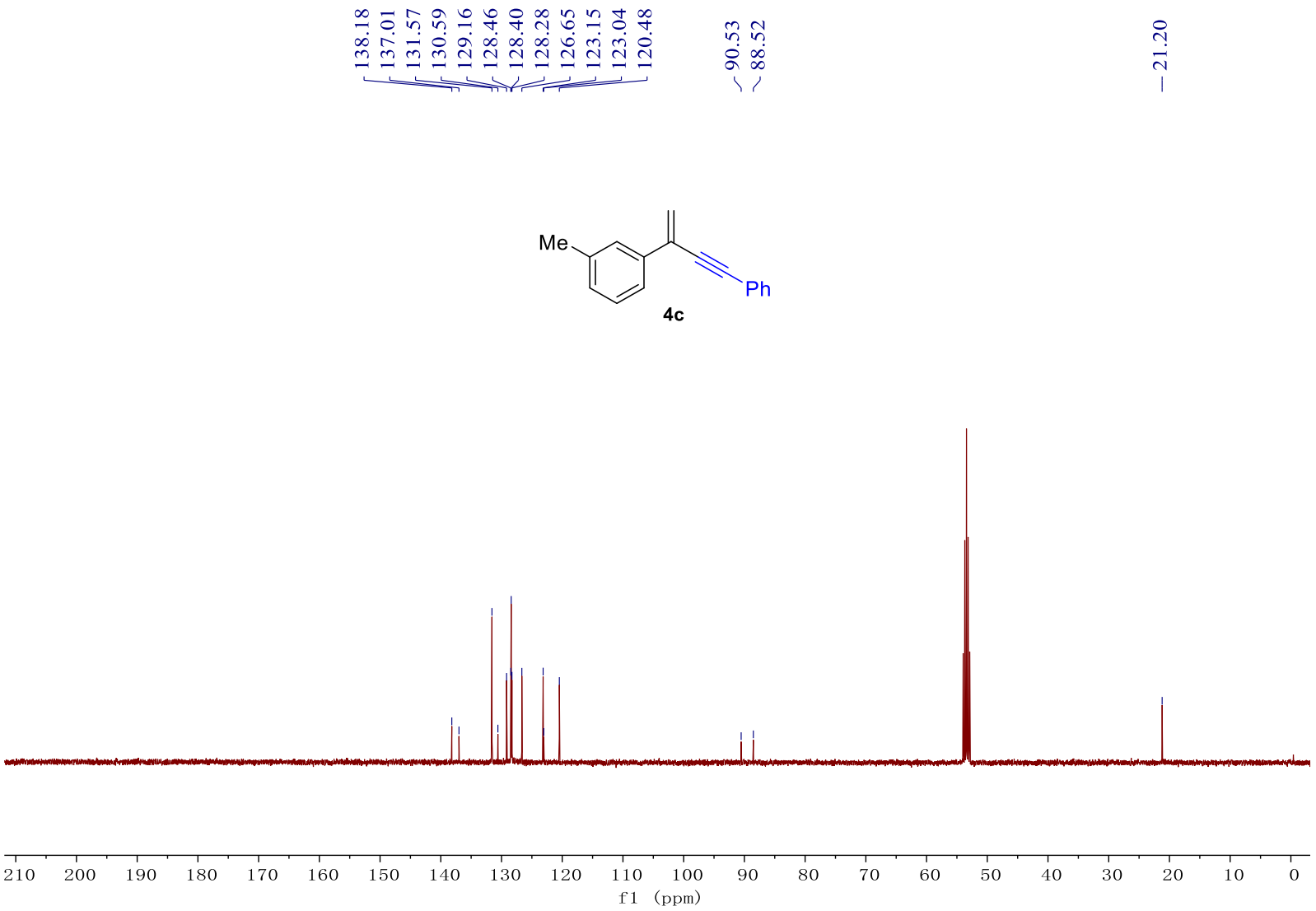
7.29
7.27
7.25
7.17
7.15
5.99
5.99
5.74
5.74
2.38
Me
Ph
4c
3.98
3.02
1.06
1.04
1.03
1.02
3.07
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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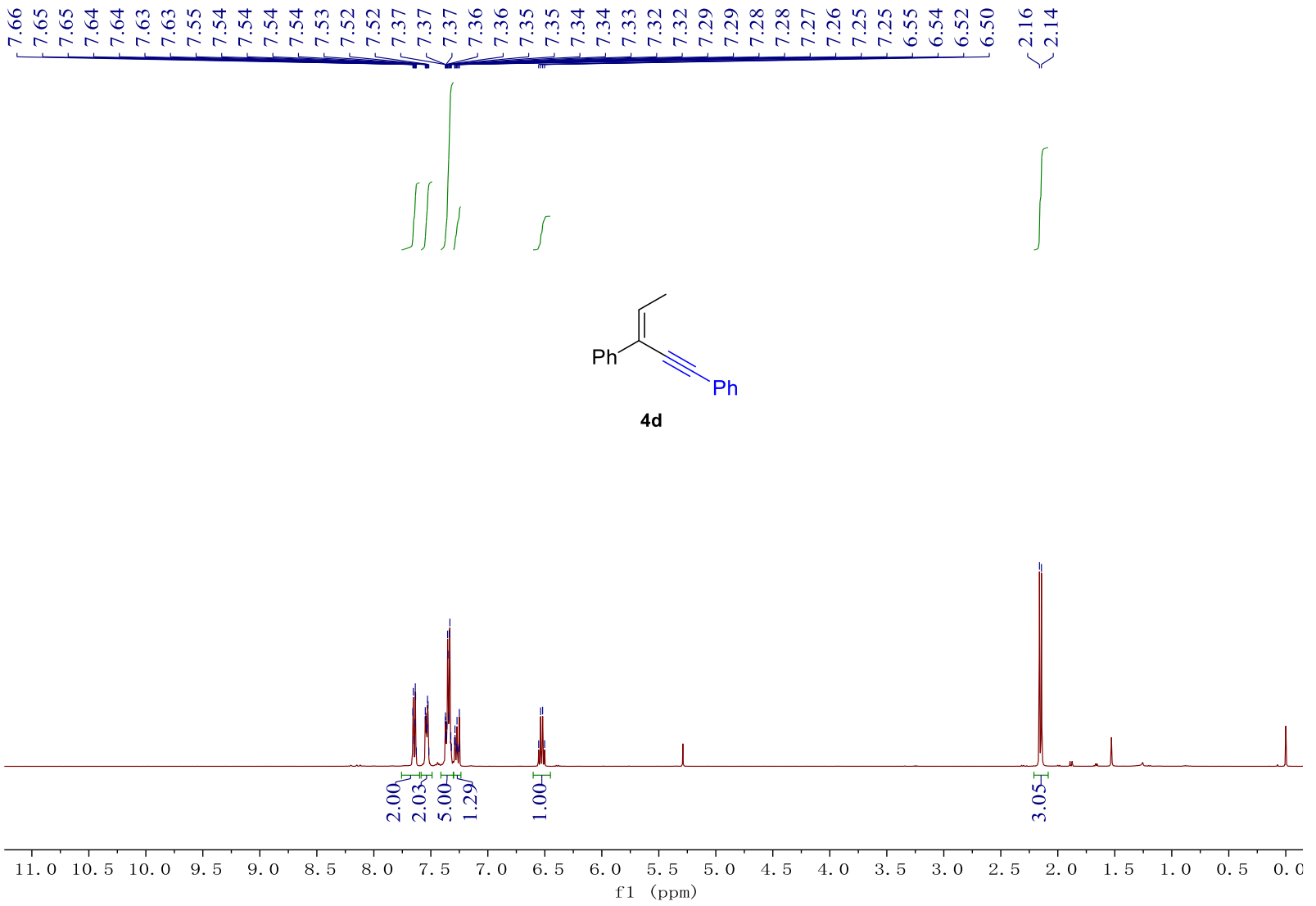
128.46
128.40
128.28
126.65
123.15
123.04
120.48
90.53
88.52
21.20
Me
Ph
4c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.52
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7.35
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7.34
7.33
7.32
7.32
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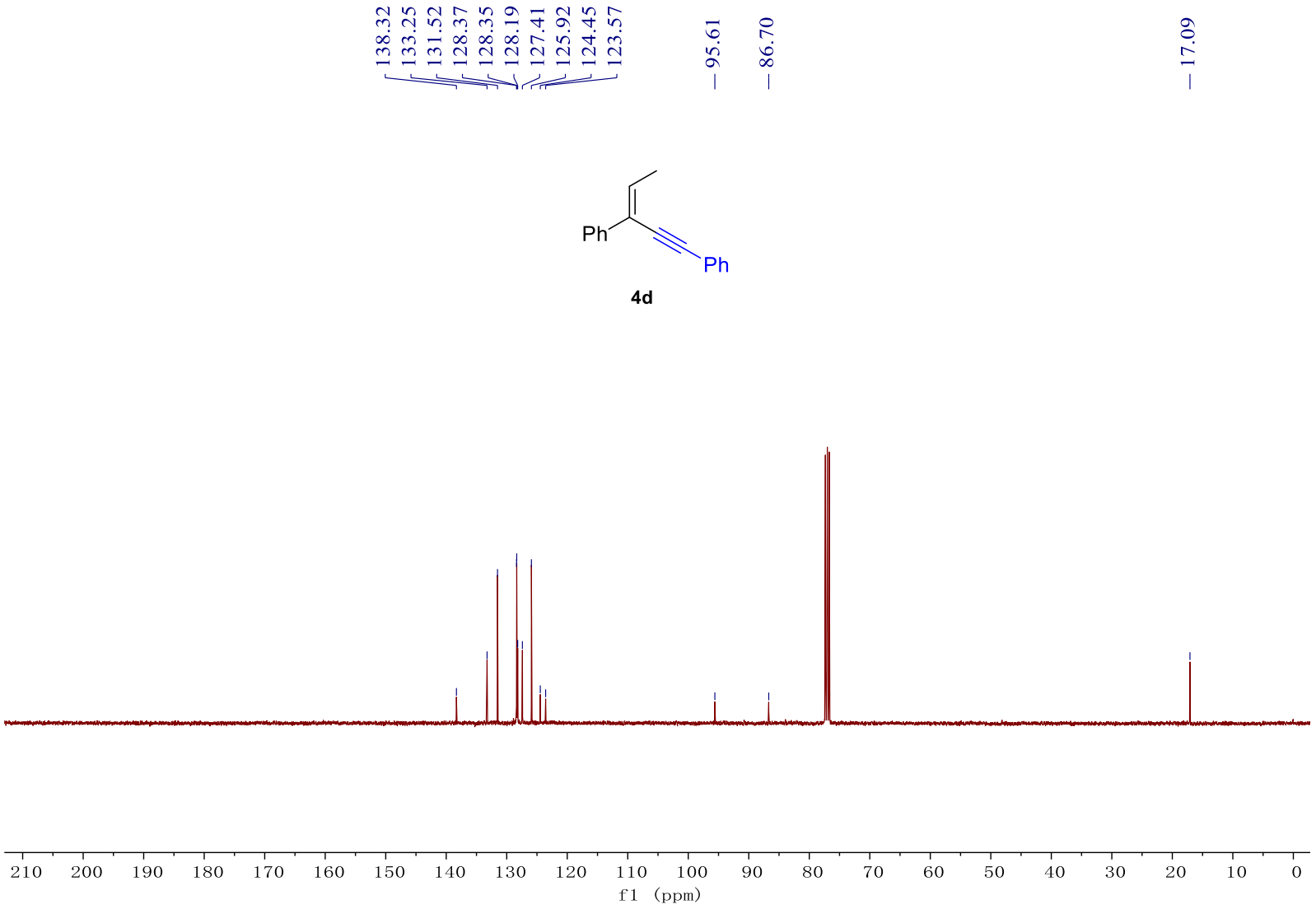
7.29
7.28
7.28
7.27
7.26
7.25
7.25
6.55
6.54
6.52
6.50
2.16
2.14
Ph
Ph
2.00
2.03
5.00
1.29
1.00
3.05
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

4d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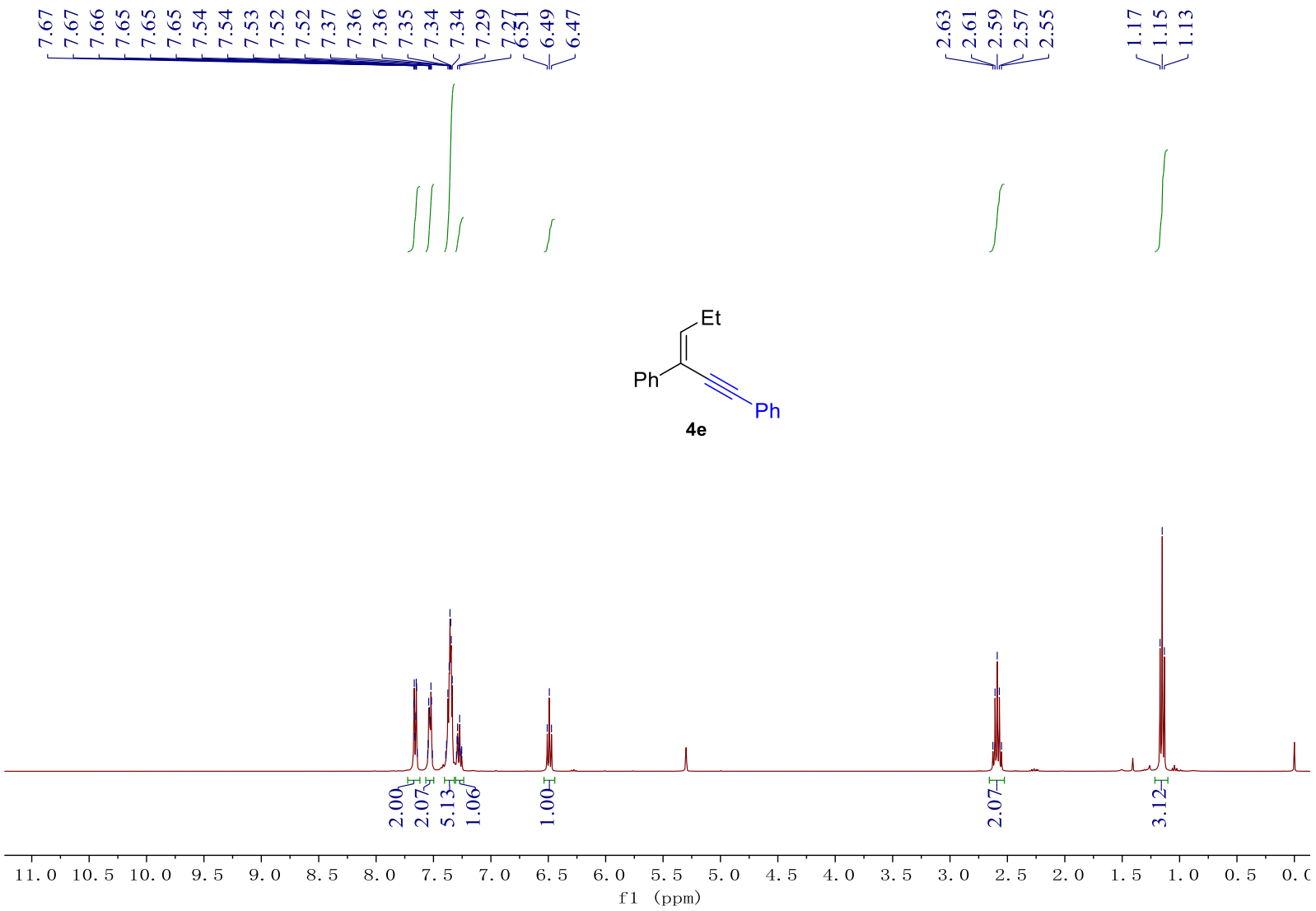
131.52
128.37
128.35
128.19
127.41
125.92
124.45
123.57
95.61
86.70
17.09
Ph
Ph
4d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.67
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7.65
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7.54
7.53
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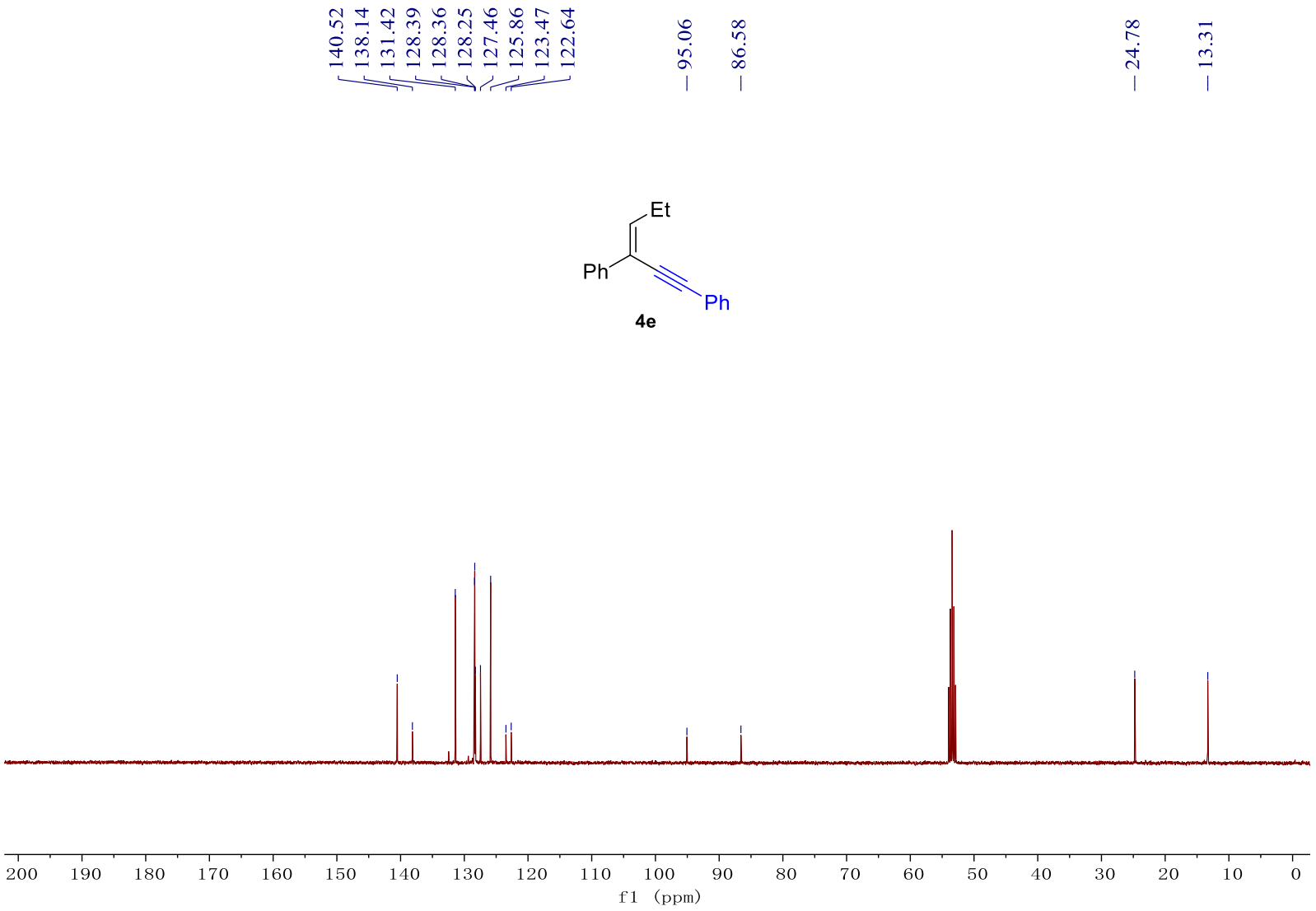
7.52
7.37
7.36
7.36
7.35
7.34
7.34
7.29
7.27
6.51
6.49
6.47
2.63
2.61
2.59
2.57
2.55
1.17
1.15
1.13
Et
Ph
Ph
4e
2.00
2.07
5.13
1.06
1.00
2.07
3.12
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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127.46
125.86
123.47
122.64
95.06
86.58
24.78
13.31
Et
Ph
Ph
4e
200
190
180
170
160
150
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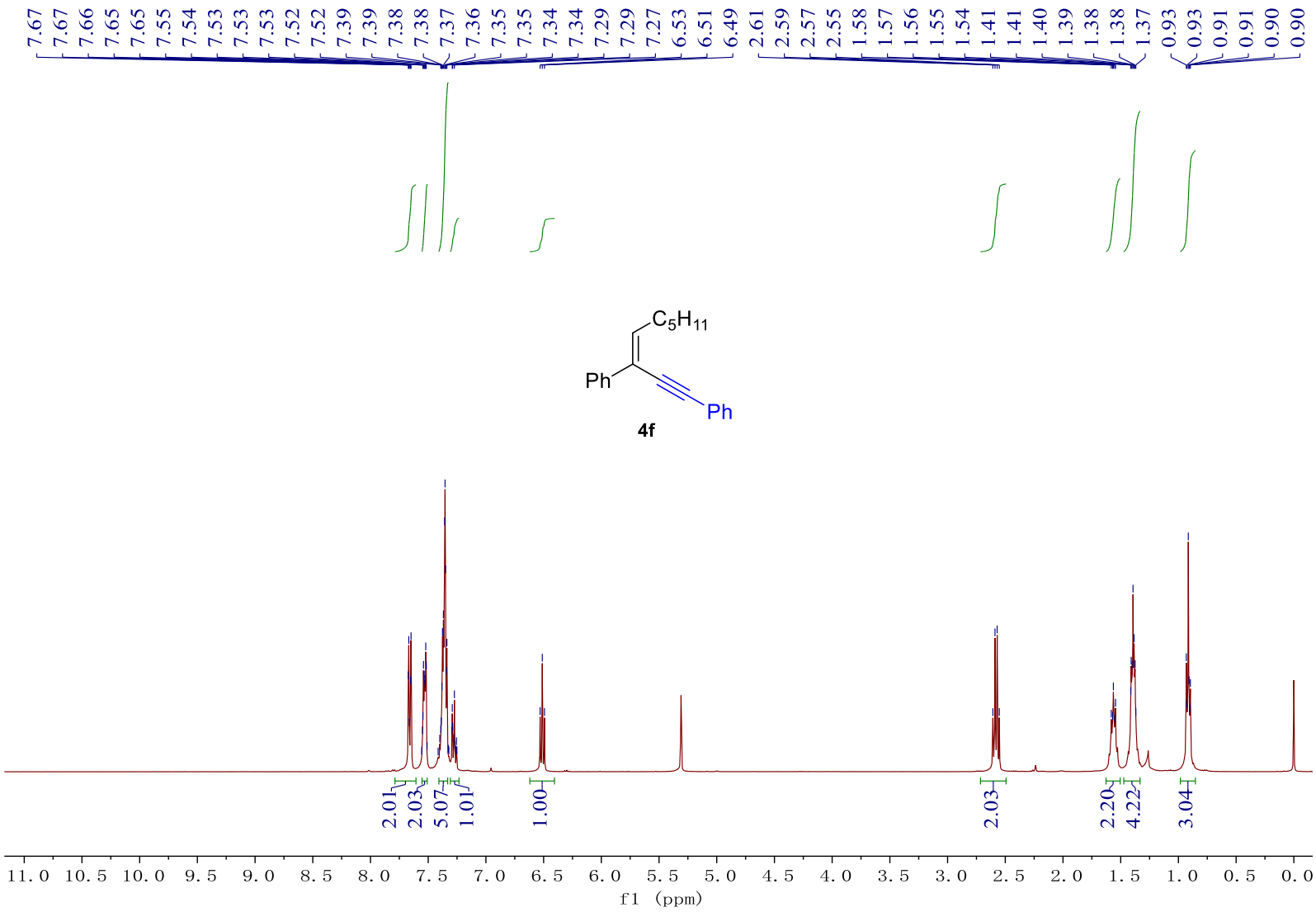
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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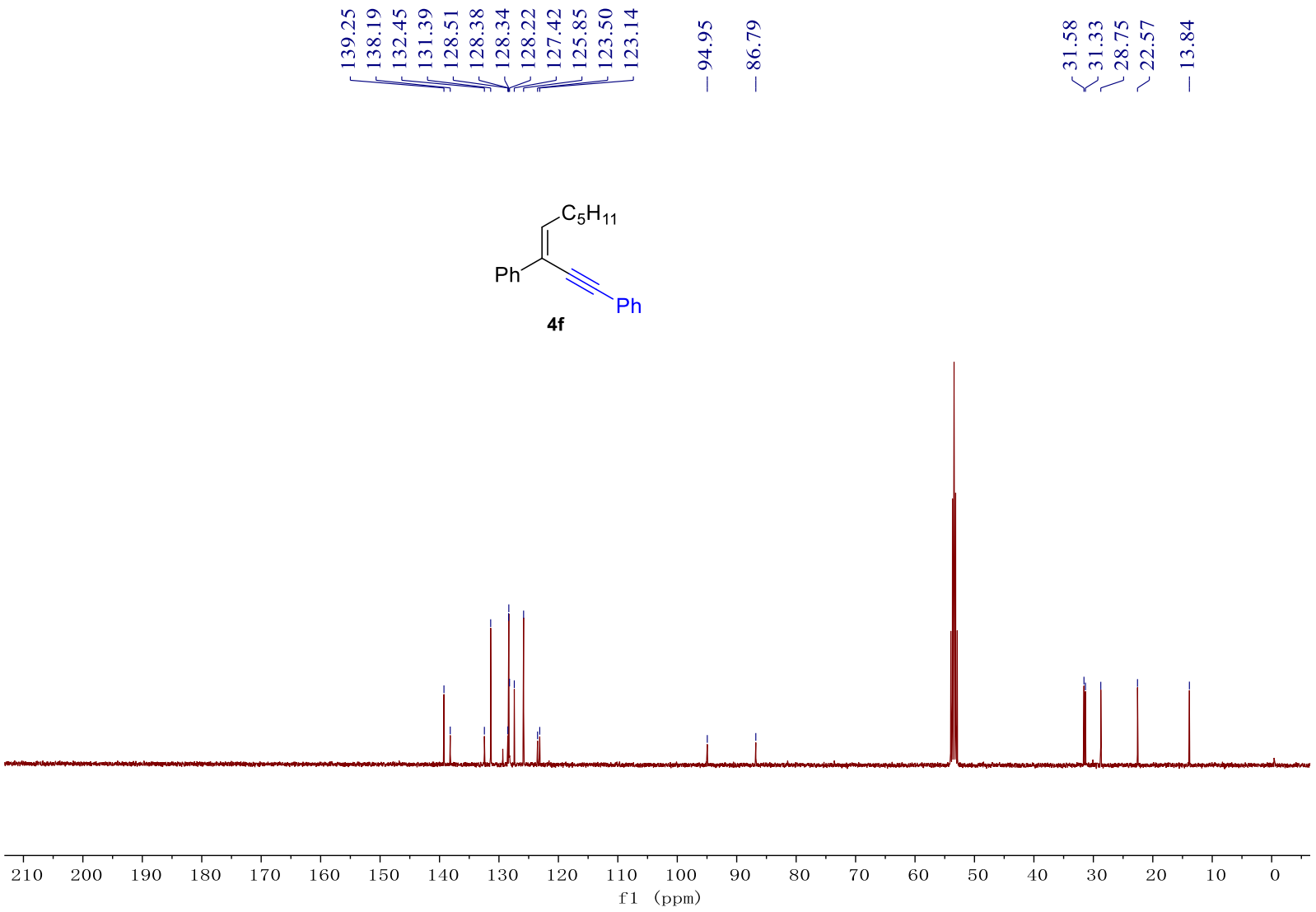
2.61 2.59 2.57 2.55 1.58 1.57 1.56 1.55 1.54 1.41 1.41 1.40 1.39 1.38 1.38 1.37 0.93 0.93 0.91 0.91 0.90 0.90
C5H11
Ph
Ph
4f
2.01 2.03 5.07 1.01 1.00 2.03 2.20 4.22 3.04
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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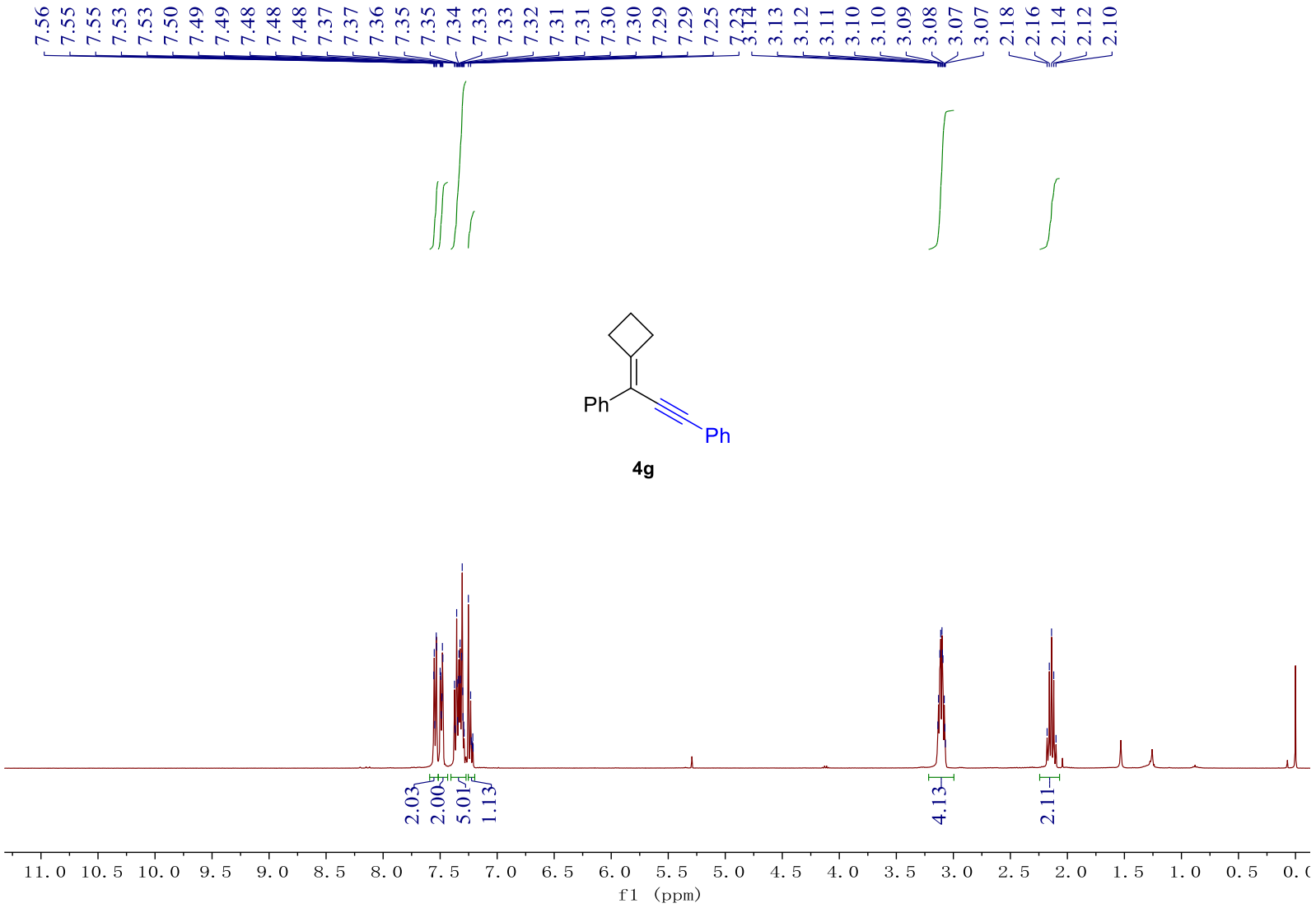
128.38
128.34
128.22
127.42
125.85
123.50
123.14
94.95
86.79
31.58
31.33
28.75
22.57
13.84
C5H11
Ph
Ph
4f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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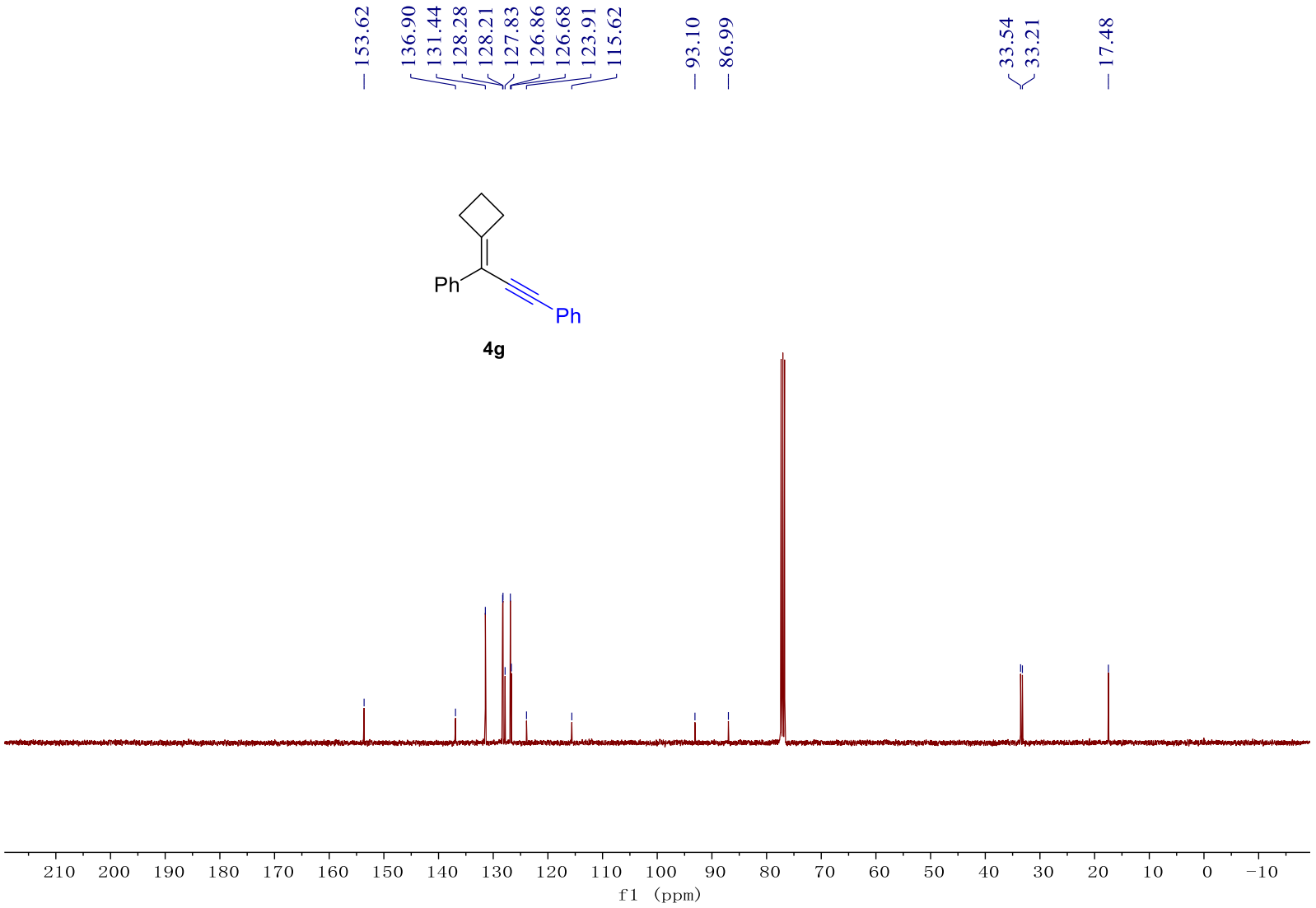
7.37
7.36
7.35
7.35
7.34
7.33
7.33
7.32
7.31
7.31
7.30
7.30
7.29
7.29
7.25
3.13
3.12
3.11
3.10
3.10
3.09
3.08
3.07
3.07
2.18
2.16
2.14
2.12
2.10
Ph
Ph
4g
2.03
2.00
5.01
1.13
4.13
2.11
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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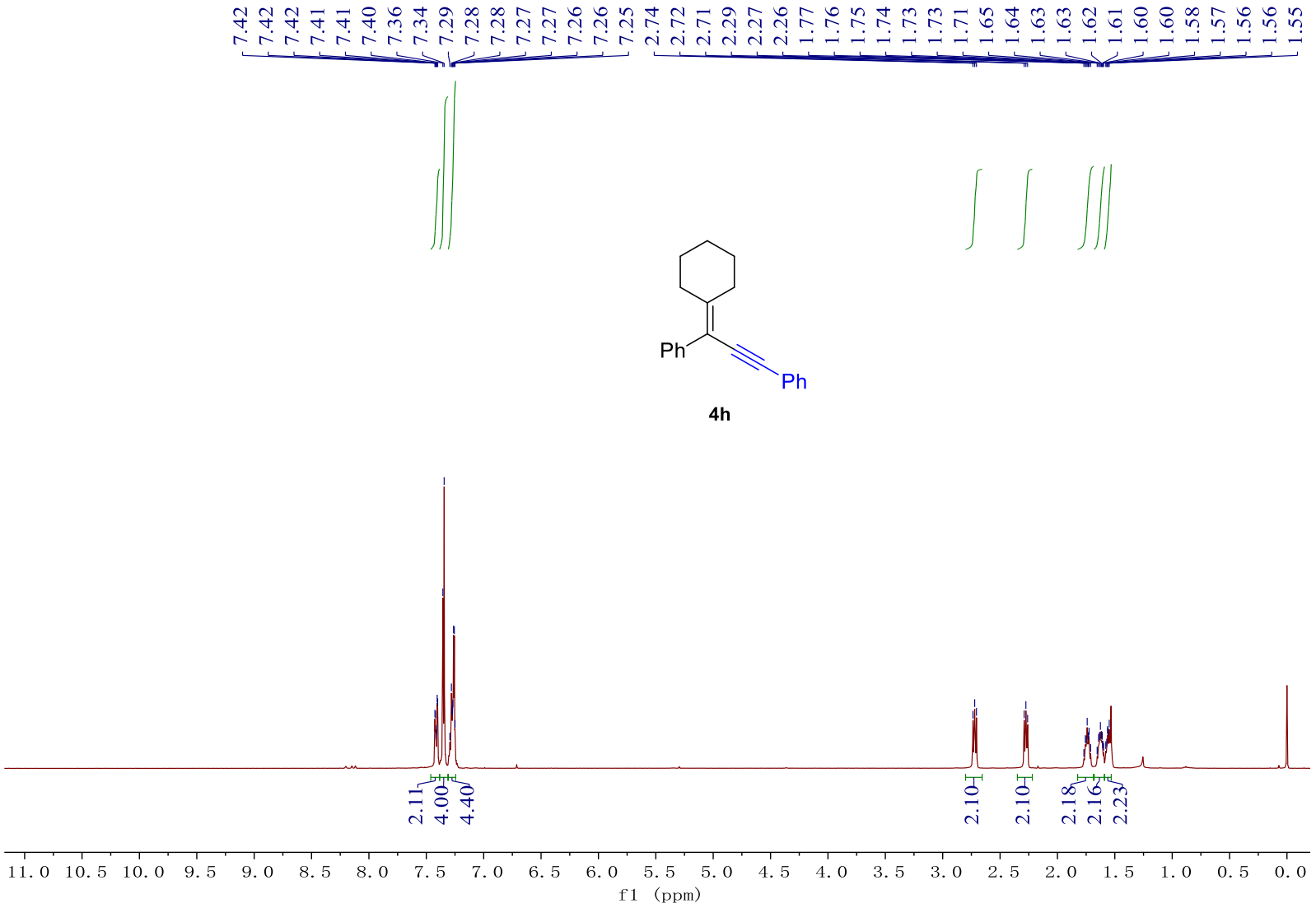
136.90
131.44
128.28
128.21
127.83
126.86
126.68
123.91
115.62
93.10
86.99
33.54
33.21
17.48
Ph
Ph
4g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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7.42
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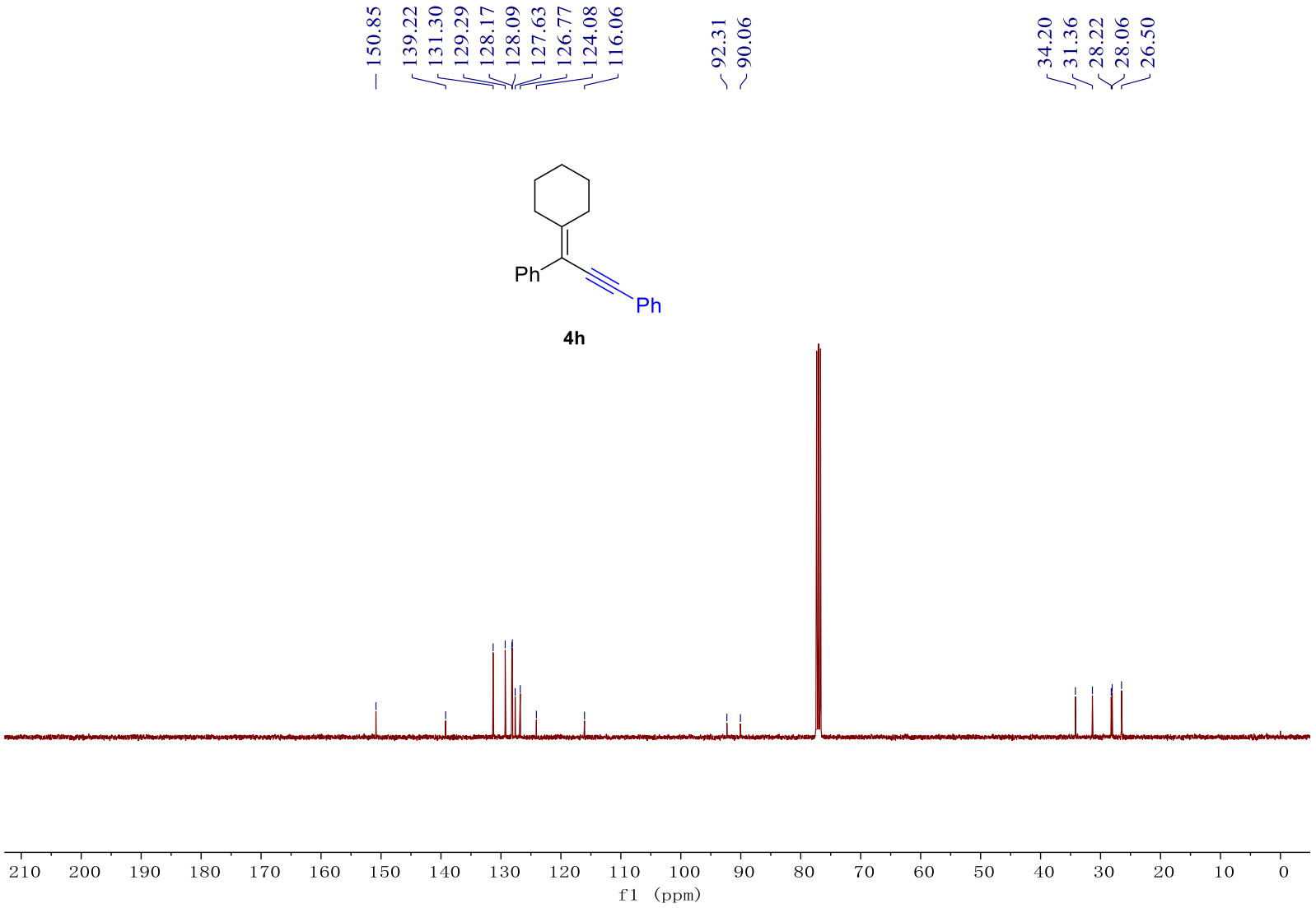
7.41
7.40
7.36
7.34
7.29
7.28
7.28
7.27
7.27
7.26
7.26
7.25
2.74
2.72
2.71
2.29
2.27
2.26
1.77
1.76
1.75
1.74
1.73
1.73
1.71
1.65
1.64
1.63
1.63
1.62
1.61
1.60
1.60
1.58
1.57
1.56
1.56
1.55
Ph
Ph
4h
2.11
4.00
4.40
2.10
2.10
2.18
2.16
2.23
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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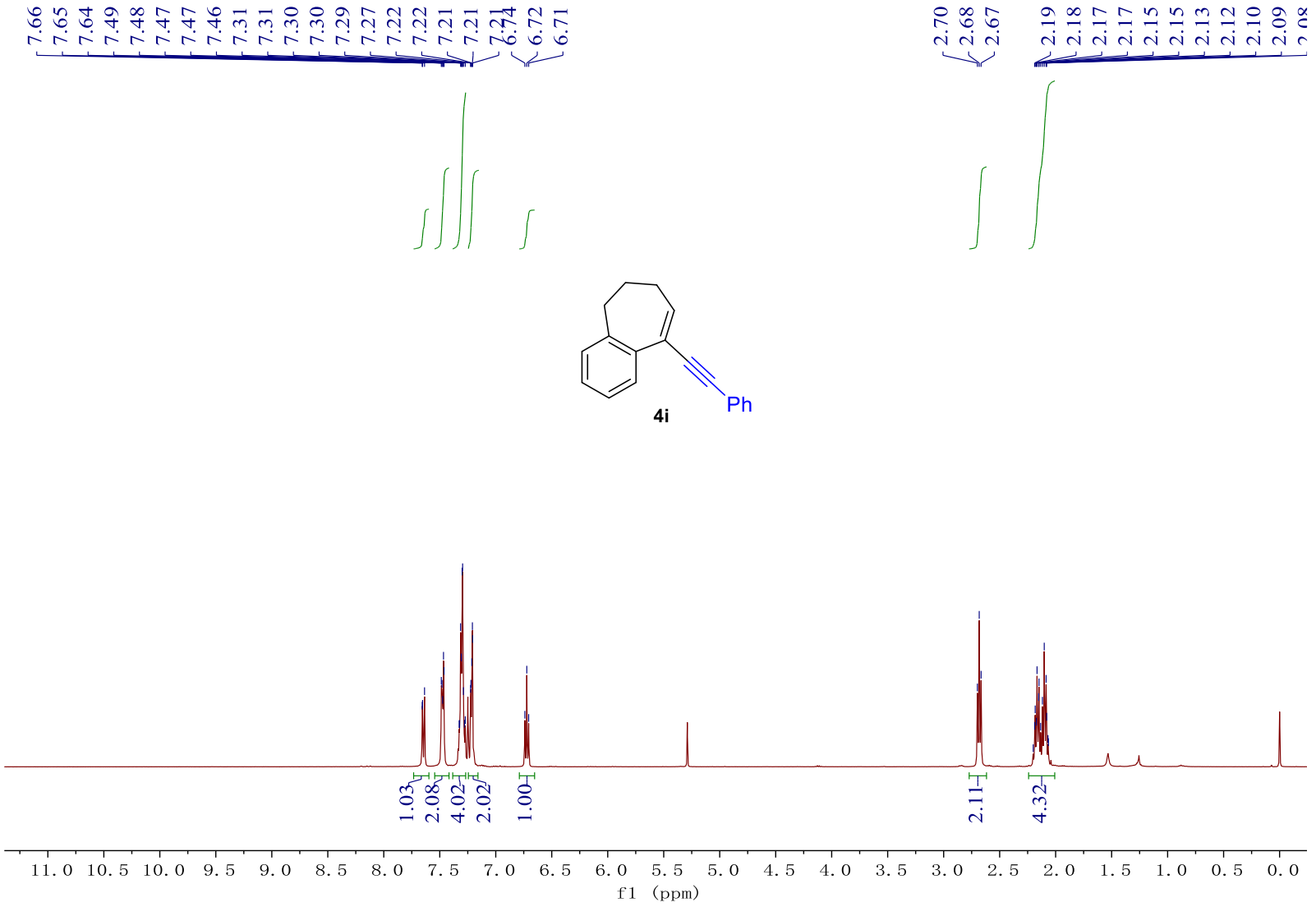
139.22
131.30
129.29
128.17
128.09
127.63
126.77
124.08
116.06
92.31
90.06
34.20
31.36
28.22
28.06
26.50
Ph
Ph
4h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.47
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7.46
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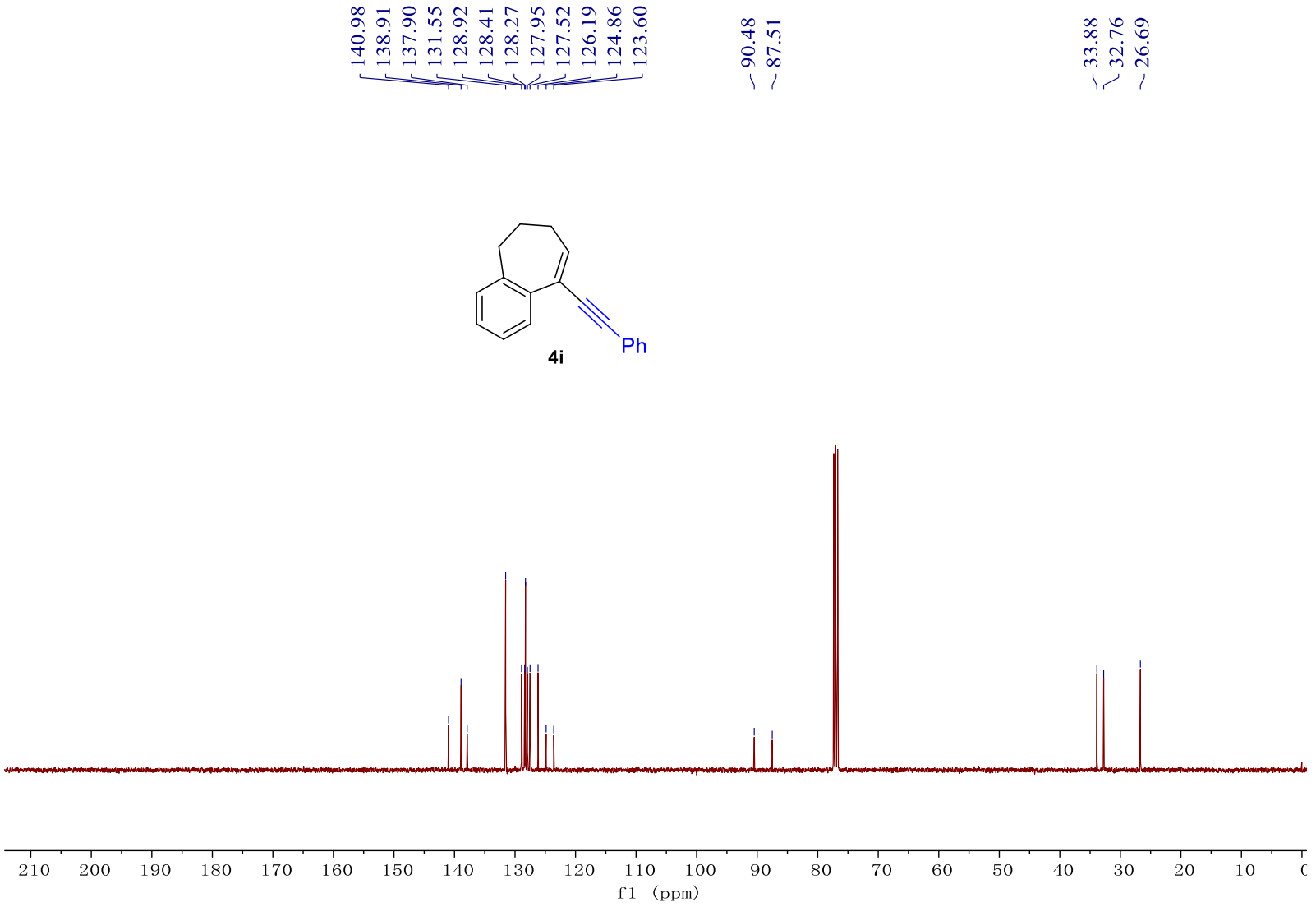
7.31
7.30
7.30
7.29
7.27
7.22
7.22
7.21
7.21
7.21
6.74
6.72
6.71
2.70
2.68
2.67
2.19
2.18
2.17
2.17
2.15
2.15
2.13
2.12
2.10
2.09
Ph
4i
1.03
2.08
4.02
2.02
1.00
2.11
4.32
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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131.55
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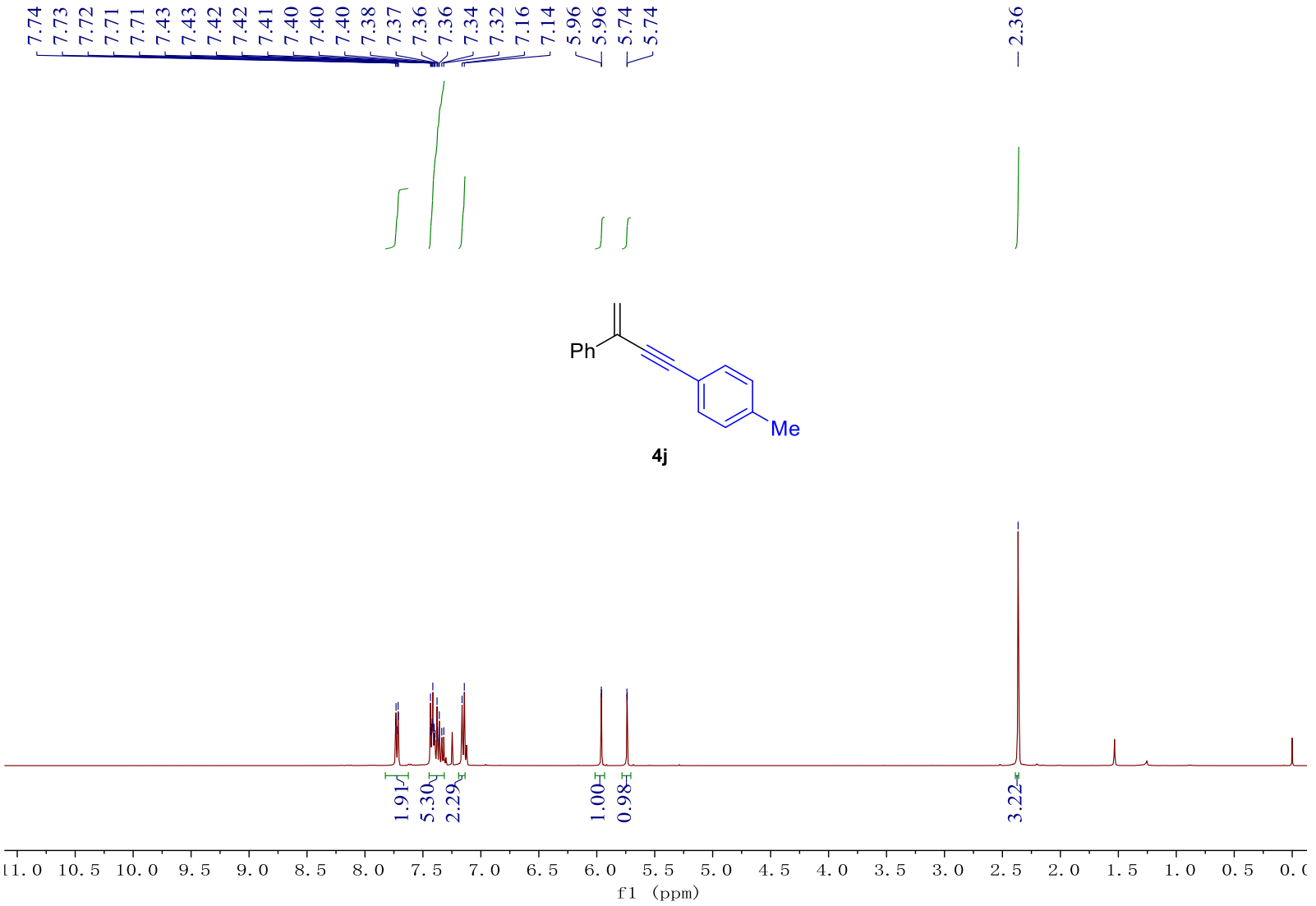
127.95
127.52
126.19
124.86
123.60
90.48
87.51
33.88
32.76
26.69
Ph
4i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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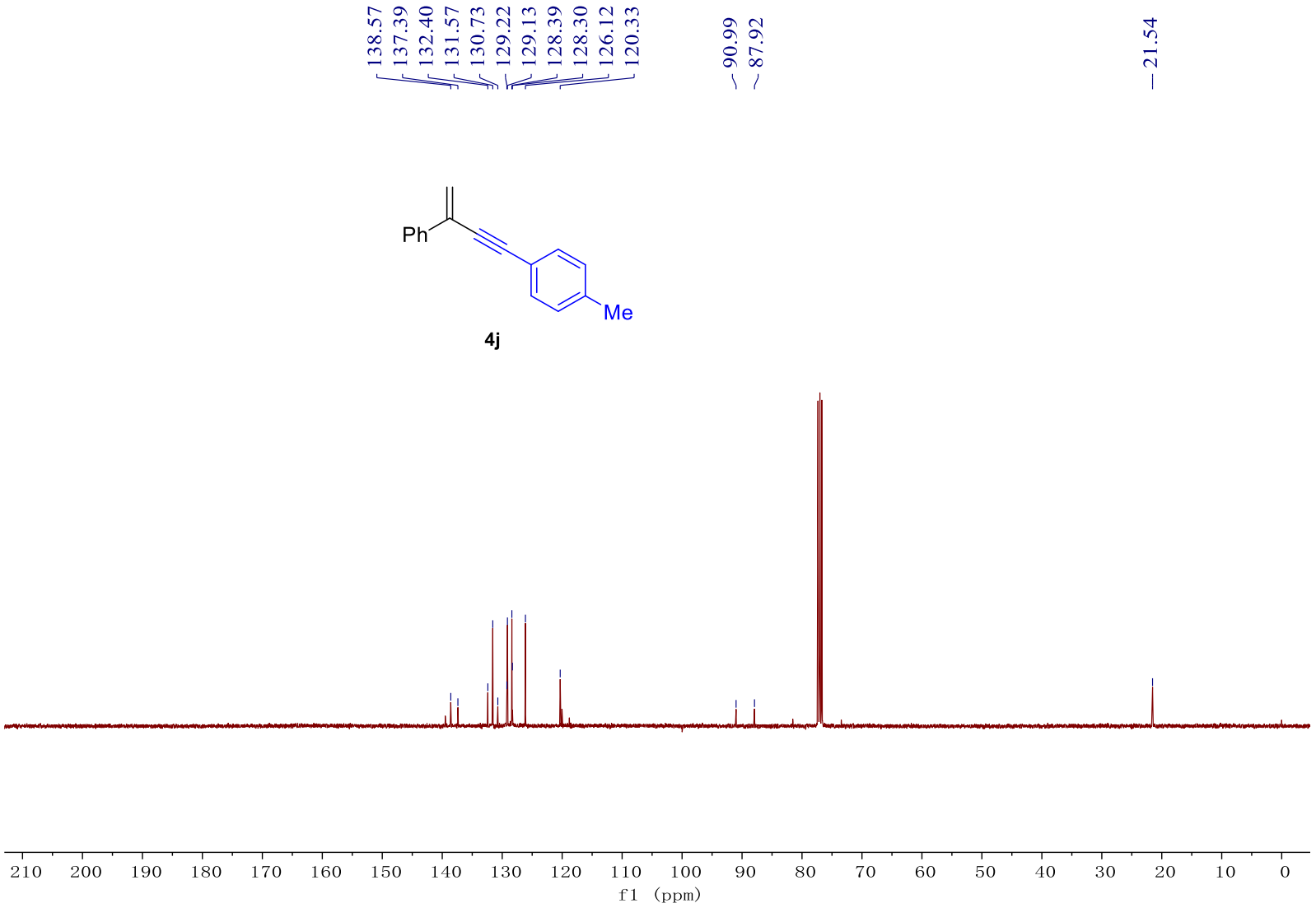
7.72
7.71
7.71
7.43
7.43
7.42
7.42
7.41
7.40
7.40
7.40
7.38
7.37
7.36
7.36
7.34
7.32
7.16
7.14
5.96
5.96
5.74
5.74
2.36
Ph
Me
4j
1.91
5.30
2.29
1.00
0.98
3.22
f1 (ppm)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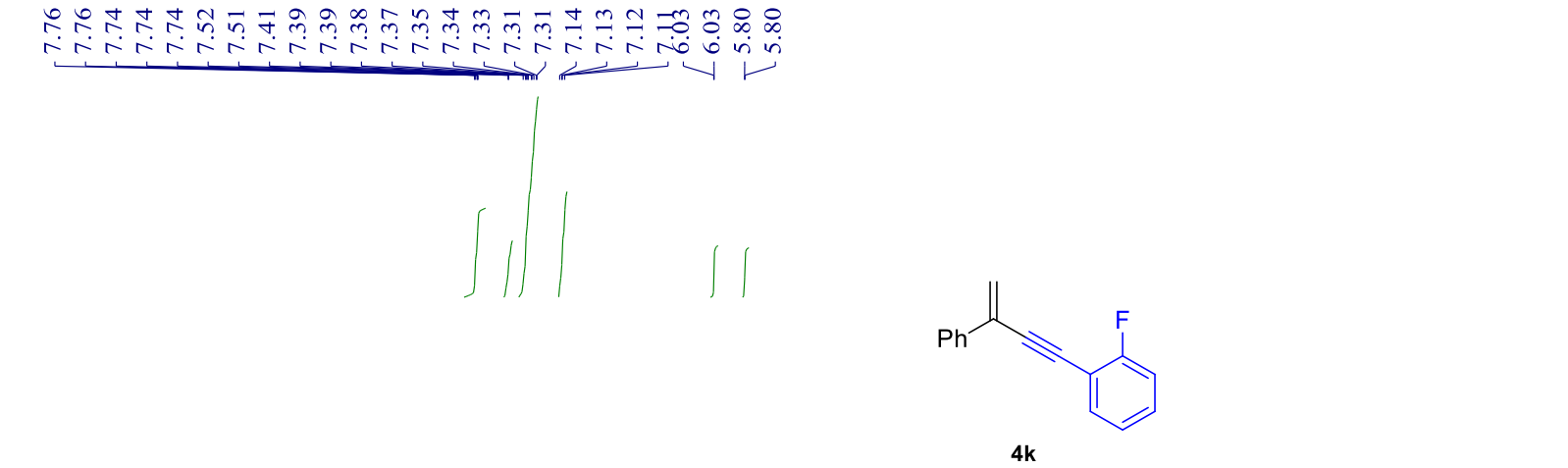
132.40
131.57
130.73
129.22
129.13
128.39
128.30
126.12
120.33
90.99
87.92
21.54
Ph
Me
4j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.39
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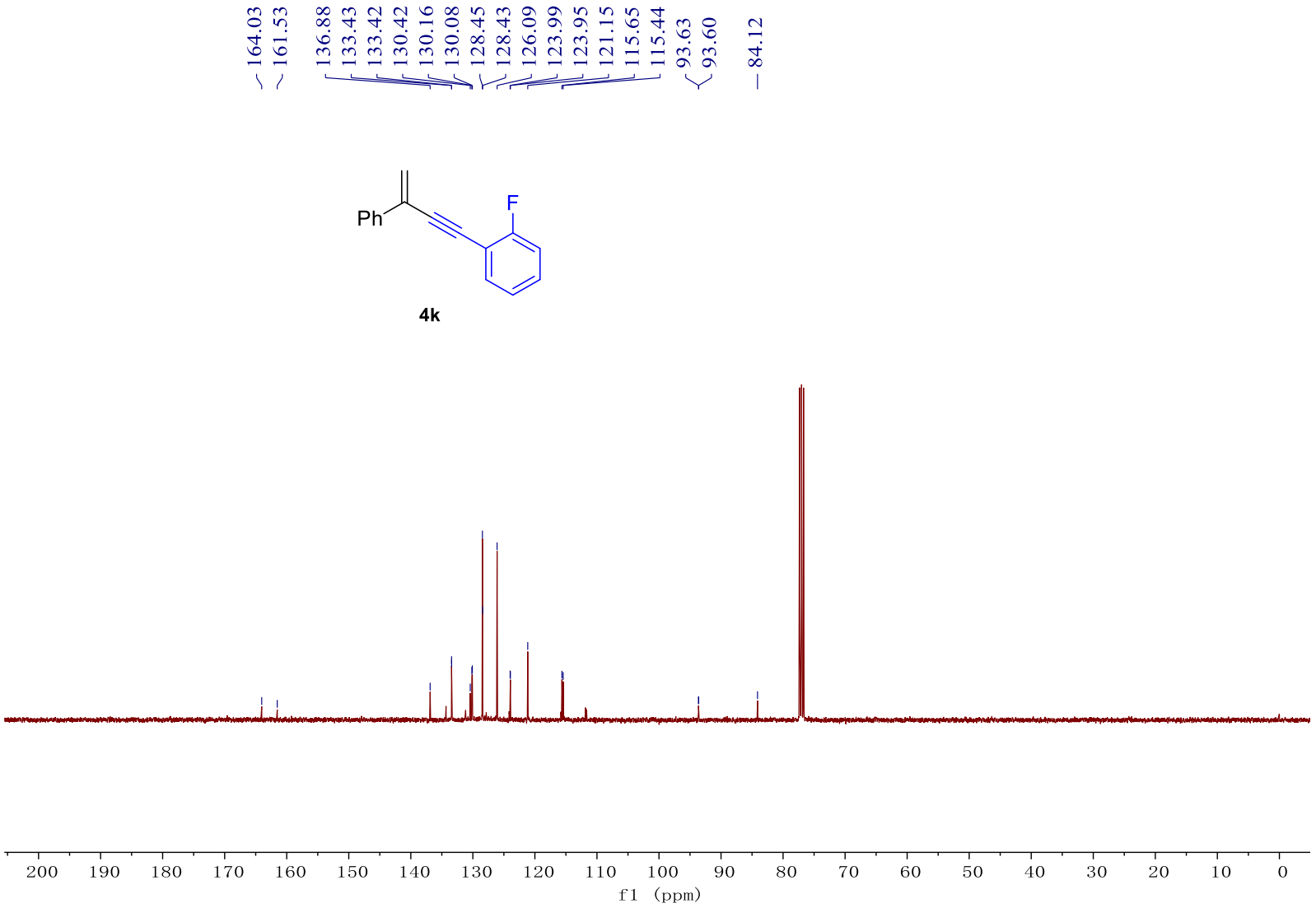
7.37
7.35
7.34
7.33
7.31
7.31
7.14
7.13
7.12
7.11
6.03
6.03
5.80
5.80
Ph
F
4k
1.80
1.14
4.05
2.13
1.04
1.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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133.42
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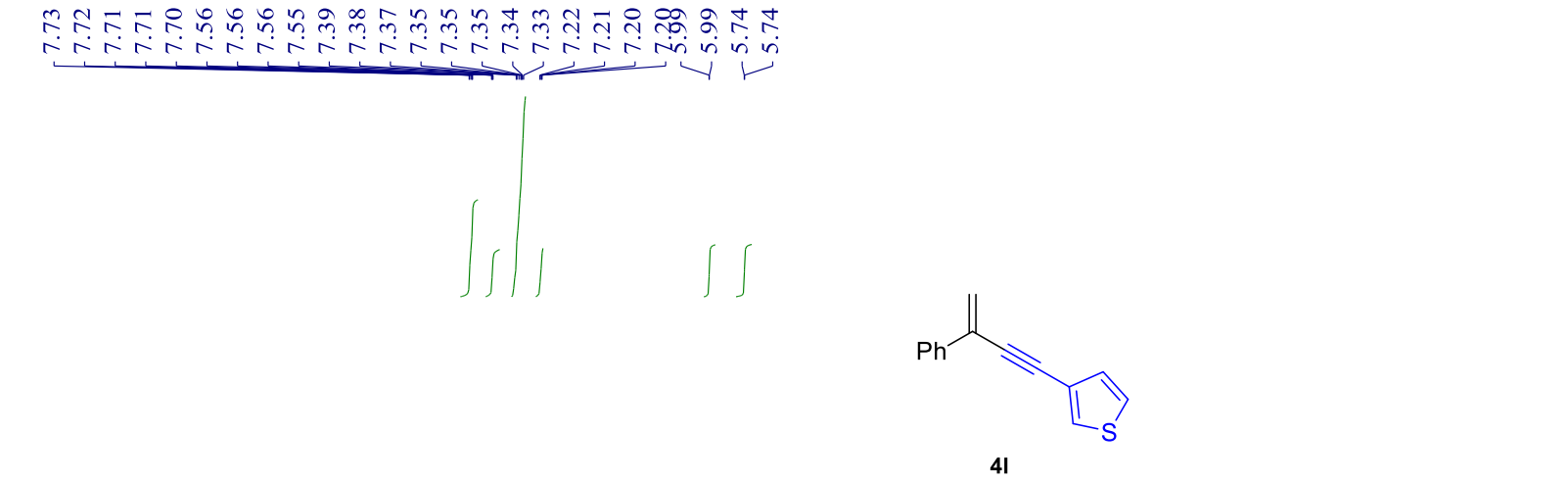
130.16
130.08
128.45
128.43
126.09
123.99
123.95
121.15
115.65
115.44
93.63
93.60
84.12
Ph
F
4k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.39
7.38
7.37
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7.35
7.35
7.34
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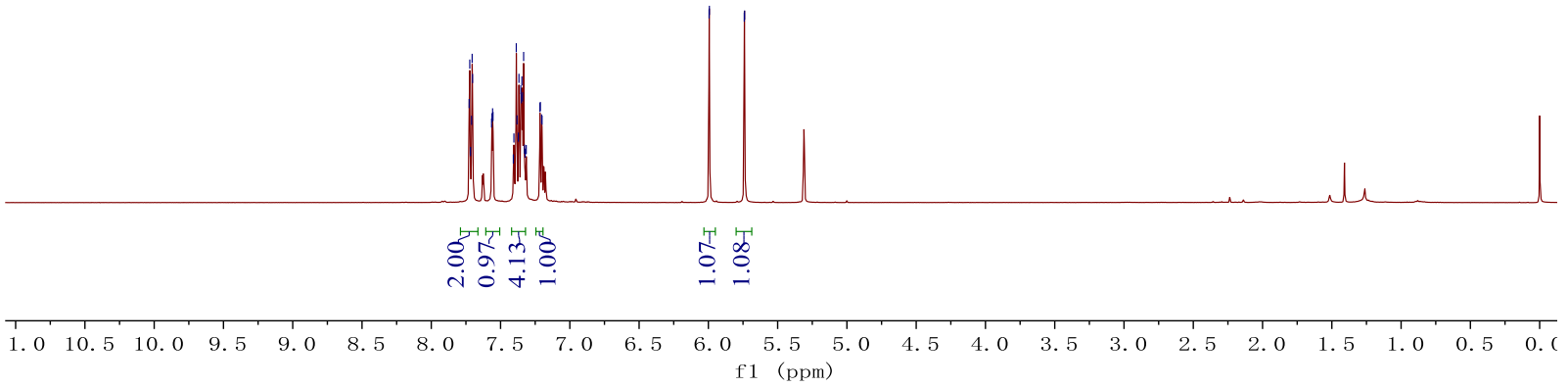
7.22
7.21
7.20
7.20
5.99
5.99
5.74
5.74
Ph
S

4l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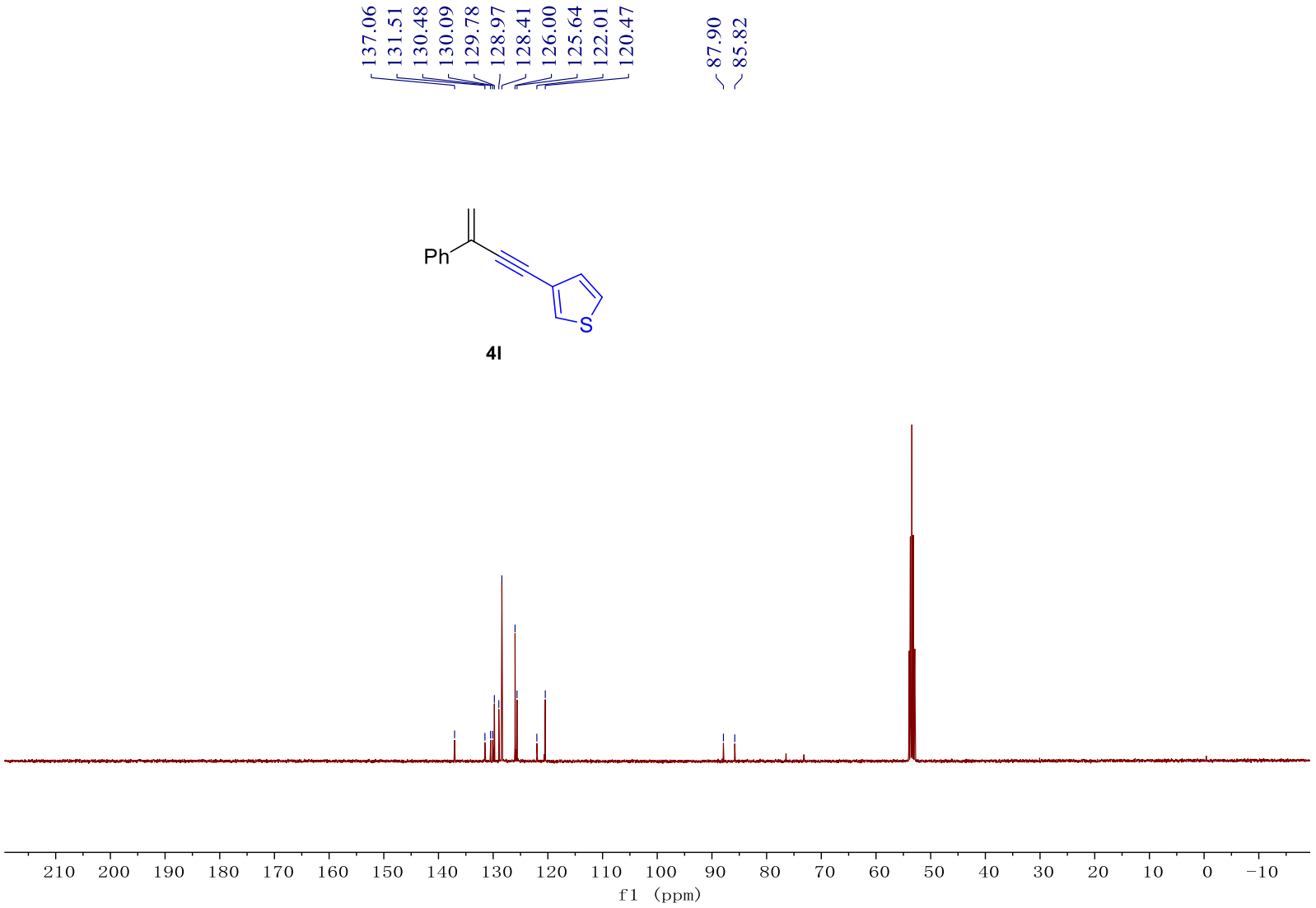
0.97
4.13
1.00
1.07
1.08
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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130.48
130.09
129.78
128.97
128.41
126.00
125.64
122.01
120.47
87.90
85.82
Ph
S
4l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)