

Supplementary Information

Unexpected and Powerful Effect of Chlorobenzene in Direct Palladium-Catalyzed Cascade Sonogashira-hydroarylation Reaction

Bo Yu,^a Huaming Sun,^a Binxun Yu,^a Guofang Zhang,^a Li-Wen Xu,^{a,b} Weiqiang Zhang,^{a,*} Ziwei Gao^{a,*}

^a Key Laboratory of Applied Surface and Colloid Chemistry, Ministry of Education, School of Chemistry & Chemical Engineering, Shaanxi Normal University, Xi'an 710062, P. R. China. *Corresponding Author Email

Address: zwgao@snnu.edu.cn, zwq@snnu.edu.cn

Table of Contents:

1. Experimental Procedure and Characterization
2. Procedure for Extensive π -Conjugated Systems of 1,3-diodobenzene
3. Kinetics Measurement of Pd/PhCl Catalyzed Direct Preparation of Triarylethenes
4. Pd/PhCl Catalyzed Reaction of 1-Hexyne and Iodobenzene
5. Deuterium Labeling Experiment
6. Copies of ¹H NMR and ¹³C NMR of compounds

1. Experimental Procedure and Characterization

General Methods. Unless otherwise stated, all manipulations were performed in a sealed Schlenk tubes under nitrogen atmosphere. Chemicals were commercially available and used without purification. ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker 400 spectrometer and chemical shifts are reported in ppm using $(\text{CH}_3)_4\text{Si}$ (for ^1H , $\delta = 0.00$; for ^{13}C , $\delta = 77.16$) as the internal standard. The following abbreviations are used to denote the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants (J) were reported in hertz unit (Hz). Kinetics measurement were obtained by GC-MS.

General Procedure for Pd/PhCl Catalyzed Direct Preparation of Triarylethenes. Under the protection of N_2 , 5 mL EtOH was cannula transferred into Schlenk tubes containing $\text{Pd}_2(\text{dba})_3$ (0.0114 g, 0.0125 mmol) and K_2CO_3 (0.138 g, 1 mmol). Alkyne (0.5 mmol), aryl iodine (1.5 mmol), chlorobenzene (5 μL , 0.05mmol) were added by syringe. The reaction mixture was stirred at 70 $^\circ\text{C}$ for 12 h. After the reaction cooled down to room temperature, the solvent was removed under vacuum and crude products were filtered off and purified by column chromatography on silica gel using dichloromethane/petroleum ether as eluent. All products were identified by comparing their spectral data with those of authentic samples.

Triphenylethylene (4a): Isolated yield 80%, white thick fluid, $R_f = 0.51$ (petroleum ether). ^1H NMR (400 MHz, CDCl_3) δ 7.25-7.14 (m, 8H), 7.10 (dd, 2H), 7.04-6.97 (m, 3H), 6.93 (d, 2H), 6.86 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.5, 142.7, 140.5, 137.5, 130.5, 129.6, 128.7, 128.3, 128.1, 127.6, 126.8.

4,4',4''-(ethene-1,1,2-triyl)tris(methoxybenzene) (4b): Isolated yield 72%, yellow solid, $R_f = 0.45$ (petroleum ether/dichloromethane = 2:1). ^1H NMR (400 MHz, CDCl_3) δ 7.18-7.15 (m, 2H), 7.07-7.02 (m, 2H), 6.90 (t, 2H), 6.81-6.74 (m, 4H), 6.70 (s, 1H), 6.62-6.57 (m, 2H), 3.75 (s, 3H), 3.73 (s, 3H), 3.67 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.1, 158.9, 158.2, 140.0, 136.8, 133.1, 131.7, 130.7, 128.7, 125.9, 114.2, 113.6, 55.4, 55.3, 55.2.

4,4',4''-(ethene-1,1,2-triyl)tris(methylbenzene) (4c): Isolated yield 50%, white thick fluid, $R_f = 0.51$ (petroleum ether). ^1H NMR (400 MHz, CDCl_3) δ 7.20 (d, 2H), 7.10 (dd, 6H), 6.92 (s, 4H), 6.86 (s, 1H), 2.36 (s, 3H), 2.33 (s, 3H), 2.24 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 141.7, 141.1, 137.8, 137.2, 137.0, 136.3, 134.9, 130.3, 129.4, 128.8, 127.6, 127.2, 21.4, 22.3, 21.2.

3,3',3''-(ethene-1,1,2-triyl)tris(methylbenzene) (4d): Isolated yield 51%, white solid, $R_f = 0.4$ (petroleum ether). ^1H NMR (400 MHz, CDCl_3) δ 7.29 (dd, 3H), 7.20 (dd, 3H), 7.14-7.05 (m, 3H), 6.99 (t, 3H), 6.88 (d, 1H), 2.41 (d, 6H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.7, 142.7, 140.6, 138.2, 137.7, 137.4, 130.9, 130.6, 128.5, 128.3, 128.2, 128.1, 127.8, 127.5, 126.5, 124.9, 21.6, 21.5, 21.4.

4,4',4''-(ethene-1,1,2-triyl)tris(bromobenzene) (4e): Isolated yield 40%, white solid, Rf = 0.4 (petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 7.44 (dd, 4H), 7.27 (d, 2H), 7.13 (d, 2H), 7.02 (d, 2H), 6.89 (s, 1H), 6.86 (d, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 141.6, 141.2, 138.4, 135.7, 132.2, 131.5, 131.1, 129.3, 127.9, 122.2, 121.2.

(Z)-(1-(4-ethylphenyl)ethene-1,2-diyl)dibenzene and (2-(4-ethylphenyl)ethene-1,1-diyl)dibenzene (4f): Isolated yield 70%, white oil, isomer ratio: 50/50, Rf = 0.5 (petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 7.26-7.09 (m, 15H), 7.07-6.94 (m, 8H), 6.86 (s, 4H), 6.83 (s, 1H), 2.58 (q, 2H), 2.46 (q, 2H), 1.16 (d, 3H), 1.08 (t, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 143.8, 143.6, 143.5, 143.0, 142.7, 141.7, 140.7, 137.6, 134.87 (s, 1H), 130.4, 129.6, 128.8, 128.2, 127.8, 127.6, 127.5, 127.4, 126.7, 28.7, 15.4.

(Z)-(1-(4-methoxyphenyl)ethene-1,2-diyl)dibenzene and (2-(4-methoxyphenyl)ethene-1,1-diyl)dibenzene (4g): Isolated yield 65%, white oil, isomer ratio: 49/51, Rf = 0.5 (petroleum ether/dichloromethane = 1:1). ¹H NMR (400 MHz, CDCl₃) δ 7.37-7.28 (m, 10H), 7.26-7.14 (m, 5H), 7.11 (dt, 5H), 7.06 (d, 2H), 6.94 (d, 2H), 6.91 (s, 1H), 6.89 (s, 1H), 6.84 (d, 2H), 6.65 (d, 2H), 3.80 (s, 3H), 3.71 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 159.1, 158.5, 143.9, 143.7, 142.4, 140.7, 137.8, 132.6, 131.7, 130.9, 130.5, 130.2, 129.6, 128.8, 128.2, 128.1, 127.9, 127.8, 127.6, 127.5, 127.4, 127.3, 126.7, 114.1, 113.5, 55.2.

(Z)-(1-(p-tolyl)ethene-1,2-diyl)dibenzene and (2-(p-tolyl)ethene-1,1-diyl)dibenzene (4h): Isolated yield 52%, white oil, isomer ratio: 50/50, Rf = 0.5 (petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 7.28 (d, 15H), 7.23 (d, 2H), 7.15-7.01 (m, 9H), 6.92 (d, 6H), 2.35 (s, 3H), 2.23 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 143.7, 142.7, 141.8, 140.7, 137.7, 137.4, 137.1, 136.6, 134.6, 130.4, 129.7, 129.6, 129.4, 128.8, 128.7, 128.3, 128.2, 128.0, 127.8, 127.6, 127.5, 127.4, 126.7, 21.3.

(E)-3,3'-(1-(4-methoxyphenyl)ethene-1,2-diyl)bis(methylbenzene) and 3,3'-(2-(4-methoxyphenyl)ethene-1,1-diyl)bis(m-ethylbenzene) (4i): Isolated yield 62%, white oil, isomer ratio: 50/50, Rf = 0.4 (petroleum ether/dichloromethane = 2:1). ¹H NMR (400 MHz, CDCl₃) δ 7.25-6.97 (m, 17H), 6.94 (d, 2H), 6.90 (d, 2H), 6.88-6.79 (m, 5H), 6.66 (d, 2H), 3.81 (s, 3H), 3.72 (s, 3H), 2.31 (d, 3H), 2.19 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 159.0, 158.4, 143.9, 142.3, 140.8, 138.3, 137.7, 137.5, 132.9, 131.7, 130.9, 130.6, 130.3, 128.6, 128.5, 128.3, 128.1, 128.0, 127.9, 127.5, 127.4, 126.5, 125.0, 124.8, 114.0, 113.5, 55.2, 21.5.

(E)-3,3'-(1-phenylethene-1,2-diyl)bis(methylbenzene) and 3,3'-(2-phenylethene-1,1-diyl)bis(methylbenzene) (4j): Isolated yield 52%, white oil, isomer ratio: 44/56, Rf = 0.6 (petroleum ether/dichloromethane = 30:1). ¹H NMR (400 MHz, CDCl₃) δ 7.34-7.28 (m, 3H), 7.01 (t, 19H), 6.91 (s, 3H), 6.84 (s, 1H), 6.78 (d, 1H), 2.31 (d, 8H), 2.17 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 143.6, 142.9, 142.6, 140.7, 140.4, 138.3, 137.8, 137.6, 137.5, 137.4, 130.9, 130.6, 130.4, 129.6, 128.6, 128.5, 128.4, 128.3, 128.2, 128.0, 127.9, 127.6, 127.5, 127.4, 126.7, 126.6, 125.0, 124.9, 21.6, 21.5, 21.4.

(E)-3,3'-(1-(p-tolyl)ethene-1,2-diyl)bis(methylbenzene) and 3,3'-(2-(p-tolyl)ethene-1,1-diyl)bis(methylbenzene) (4k): Isolated yield 50%, white oil, isomer ratio: 50/50, Rf = 0.5 (petroleum ether). ¹H NMR (400MHz, CDCl₃) δ 7.20 (dd, 2H), 7.18 (s, 1H), 7.15 (d, 2H), 7.13-7.05 (m, 9H), 7.03-6.94 (m, 4H), 6.91 (d, 4H), 6.88 (d, 3H), 6.79 (d, 1H), 2.37 (s, 3H), 2.31 (s, 6H), 2.29 (s, 3H), 2.25 (s, 3H), 2.18 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 142.7, 141.5, 140.8, 139.5, 137.1, 136.4, 135.8, 135.4, 133.5, 129.7, 129.5, 129.2, 128.2, 127.6, 127.4, 127.2, 127.1, 126.9, 126.8, 126.7, 126.3, 125.3, 123.8, 20.4, 20.3, 20.2, 20.1.

(Z)-4,4'-(1-(4-methoxyphenyl)ethene-1,2-diyl)bis(methylbenzene) and 4'-(2-(4-methoxyphenyl)ethene-1,1-diyl)bis(methylbenzene) (4l): Isolated yield 48%, white oil, isomer ratio: 50/50, Rf = 0.4 (petroleum ether/dichloromethane = 2:1). ¹H NMR (400 MHz, CDCl₃) δ 7.16-7.10 (m, 4H), 7.07-6.99 (m, 10H), 6.88 (d, 2H), 6.86 (s, 2H), 6.84 (s, 1H), 6.76 (dd, 4H), 6.59 (d, 2H), 3.74 (s, 2H), 3.71 (s, 1H), 3.65 (s, 3H), 2.30 (s, 4H), 2.26 (s, 5H), 2.18 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 159.2, 158.9, 158.3, 141.3, 140.6, 137.8, 137.2, 137.0, 136.9, 136.6, 136.3, 135.0, 133.0, 131.7, 130.8, 130.4, 129.5, 129.4, 129.0, 128.8, 128.7, 127.5, 127.1, 126.7, 126.4, 114.1, 113.5, 55.4, 55.3, 55.2, 21.4, 21.3, 21.2.

(E)-hex-1-ene-1,2-diyl dibenzene and hex-1-ene-1,1-diyl dibenzene (4m): Isolated yield 50%, white oil, isomer ratio: 75/25. *Trans*-4m, isolated yield 38%, Rf = 0.4 (petroleum ether/dichloromethane = 20:1). ¹H NMR (400 MHz, CDCl₃) δ 7.46 (d, 2H), 7.35 (dd, 6H), 7.31-7.21 (m, 4H), 6.59 (s, 1H), 2.73-2.68 (m, 2H), 1.40 (d, 2H), 1.32 (d, 2H), 0.85 (d, 3H). *Gem*-4m, isolated yield 12%, Rf = 0.5 (petroleum ether/dichloromethane = 20:1). ¹H NMR (400 MHz, CDCl₃) δ 7.36 (t, 2H), 7.30 (d, 1H), 7.26-7.15 (m, 7H), 6.08 (t, 1H), 2.11 (q, 2H), 1.41 (dd, 2H), 1.31 (dd, 2H), 0.85 (d, 3H).

2. Procedure for Extensive π -Conjugated Systems of 1,3-diiodobenzene

7 and 8 Under N₂ atmosphere, Pd₂(dba)₃ (0.000228 g) and K₂CO₃ (0.414 g), 1,3-diiodobenzene (1 mmol), 1-hexyne (4 mmol), chlorobenzene 10 mol%, and 5 mL EtOH were mixed and stirred at room temperature for 2 h. The crude products were separated by column chromatography on silica gel using petroleum ether/dichloromethane, and identified as *mono*- (**7**) and *bis*- (**8**) acetylene products.

1,3-di(hex-1-yn-1-yl)benzene (7): Isolated yield 58%, white oil, Rf = 0.3 (petroleum ether/dichloromethane = 20:1). ¹H NMR (400 MHz, CDCl₃) δ 7.43 (s, 1H), 7.27 (d, J = 7.7 Hz, 2H), 7.17 (t, J = 7.5 Hz, 1H), 2.39 (t, J = 6.8 Hz, 4H), 1.57 (dd, J = 14.5, 6.9 Hz, 4H), 1.47 (dd, J = 14.5, 7.3 Hz, 4H), 0.94 (t, J = 7.1 Hz, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 134.7, 130.6, 128.2, 124.3, 90.9, 80.1, 30.9, 22.1, 19.2, 13.7.

1-(hex-1-yn-1-yl)-3-iodobenzene (8): Isolated yield 22%, white oil, Rf = 0.5 (petroleum ether/dichloromethane = 20:1). ¹H NMR (400 MHz, CDCl₃) δ 7.75 (s, 1H), 7.59 (d, J = 7.9 Hz, 1H), 7.34 (d, J = 7.7 Hz, 1H), 7.00 (t, J = 7.8 Hz, 1H), 2.40 (t, J = 7.0 Hz, 2H), 1.57 (dd, J = 14.6, 7.1 Hz, 2H), 1.47 (dd, J = 14.8, 7.3 Hz, 2H), 0.95 (t, J

= 7.2 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 140.3, 136.6, 130.8, 129.8, 126.3, 93.7, 92.1, 79.1, 30.8, 22.1, 19.2, 13.7.

3-(hex-1-yn-1-yl)-1,1'-biphenyl (11): Under N₂ atmosphere, the mixture of Pd₂(dba)₃ (0.00685 g), K₂CO₃ (0.1242 g), PhB(OH)₂ (0.36 mmol), **8**. (0.3 mmol) and chlorobenzene 2 mol% was stirred at 50 °C for 2 h. The crude products were purified by flash column chromatography using petroleum ether as the eluent. Isolated yield 95%, white oil, R_f = 0.4 (petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 7.63 (s, 1H), 7.56 (d, *J* = 8.0 Hz, 2H), 7.47 (d, *J* = 7.4 Hz, 1H), 7.36 (ddd, *J* = 22.1, 14.8, 7.5 Hz, 5H), 2.42 (t, *J* = 6.9 Hz, 2H), 1.59 (dd, *J* = 14.7, 7.1 Hz, 2H), 1.49 (dd, *J* = 14.6, 7.2 Hz, 2H), 0.95 (t, *J* = 6.9 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 141.3, 140.6, 130.4, 128.8, 127.5, 127.2, 126.4, 124.7, 90.7, 80.6, 30.9, 22.1, 19.2, 13.7.

9 and 10 Under N₂ atmosphere, the mixture of Pd₂(dba)₃ (0.0092 g) and K₂CO₃ (0.083 g), iodobenzene (0.8 mmol) **7**. (0.2 mmol), chlorobenzene 10 mol%, 5 mL EtOH, at 70 °C, stirring 12 h. The crude products were obtained as *trans*- and *gem*- isomers by column chromatography on silica gel using petroleum ether as eluent.

1,3-bis((E)-2-phenylhex-1-en-1-yl)benzene (9): Isolated yield 62%, white oil, R_f = 0.3 (petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 7.48 (d, *J* = 7.2 Hz, 4H), 7.38 (t, *J* = 7.5 Hz, 5H), 7.33-7.27 (m, 3H), 7.23 (d, *J* = 7.6 Hz, 2H), 6.72 (s, 2H), 2.82-2.67 (m, 4H), 1.46-1.39 (m, 4H), 1.36-1.30 (m, 4H), 0.85 (t, *J* = 7.2 Hz, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 143.6, 143.3, 138.4, 129.5, 128.4, 128.2, 127.2, 127.0, 126.7, 31.0, 30.1, 22.8, 14.0.

12 and 13 Under N₂ atmosphere, the mixture of Pd₂(dba)₃ (0.00672 g), K₂CO₃ (0.0828 g), iodobenzene (0.6 mmol), **11**. (0.3 mmol), chlorobenzene 10 mol%, 5 mL EtOH, at 70 °C, stirring 12 h. The crude products were obtained as *trans*- and *gem*- isomers by column chromatography on silica gel using petroleum ether as eluent.

(E)-3-(2-phenylhex-1-en-1-yl)-1,1'-biphenyl (12): Isolated yield 57%, white oil, R_f = 0.3 (petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 7.61 (d, *J* = 7.8 Hz, 2H), 7.56 (s, 1H), 7.39 (ddd, *J* = 29.4, 20.3, 8.1 Hz, 11H), 6.75 (s, 1H), 2.82-2.69 (m, 2H), 1.44 (dd, *J* = 14.7, 7.6 Hz, 2H), 1.38-1.32 (m, 2H), 0.84 (d, *J* = 6.5 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 143.8, 143.2, 141.3, 138.9, 128.8, 128.5, 128.1, 127.8, 127.3, 126.7, 125.4, 31.1, 30.2, 22.9, 14.0.

(Z)-3-(1-phenylhex-1-en-1-yl)-1,1'-biphenyl (13): Isolated yield 13%, white oil, R_f = 0.5 (petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 7.59 (d, *J* = 7.5 Hz, 2H), 7.54 (d, *J* = 7.7 Hz, 1H), 7.43 (d, *J* = 7.0 Hz, 4H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.26 (d, *J* = 4.2 Hz, 5H), 7.16 (d, *J* = 7.4 Hz, 1H), 6.13 (t, *J* = 7.4 Hz, 1H), 2.17 (q, *J* = 7.3 Hz, 2H), 1.44 (dd, *J* = 14.8, 7.4 Hz, 2H), 1.37 – 1.31 (m, 2H), 0.85 (d, *J* = 7.1 Hz, 3H).

3. Kinetics Measurement of Pd/PhCl Catalyzed Direct Preparation of Triarylethenes

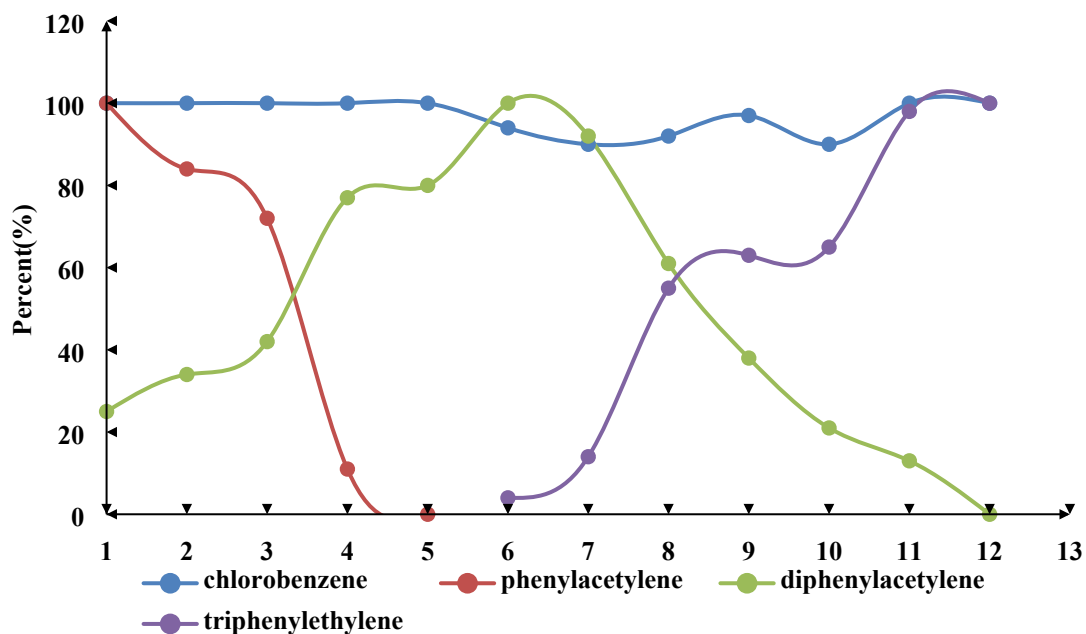


Fig. S1. Conversion v.s time of Pd/PhCl catalyzed tandem reaction of phenylacetylene and iodobenzene

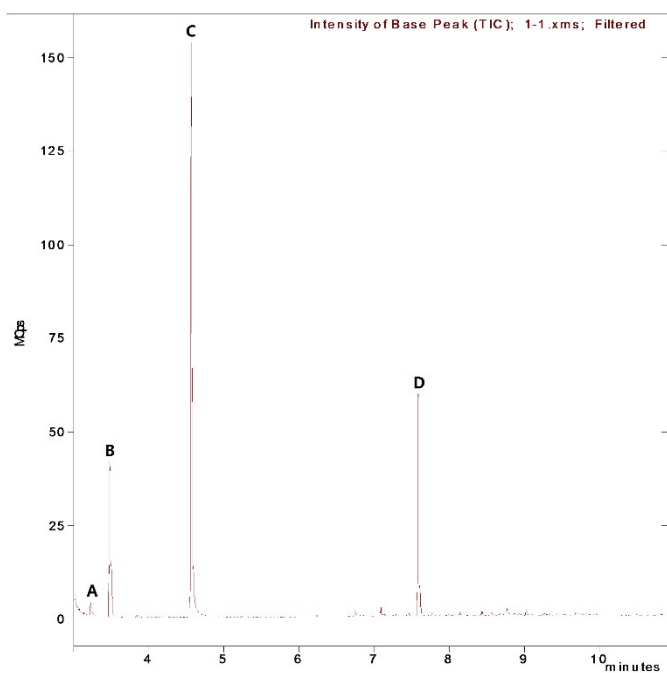


Fig. S2. GC-MS spectrum of the reaction for 10 min

A: chlorobenzene r.t.=3.222 min; B: phenylacetylene r.t.=3.477 min;
C: iodobenzene r.t.=4.561 min; D: diphenylacetylene r.t.=7.585 min

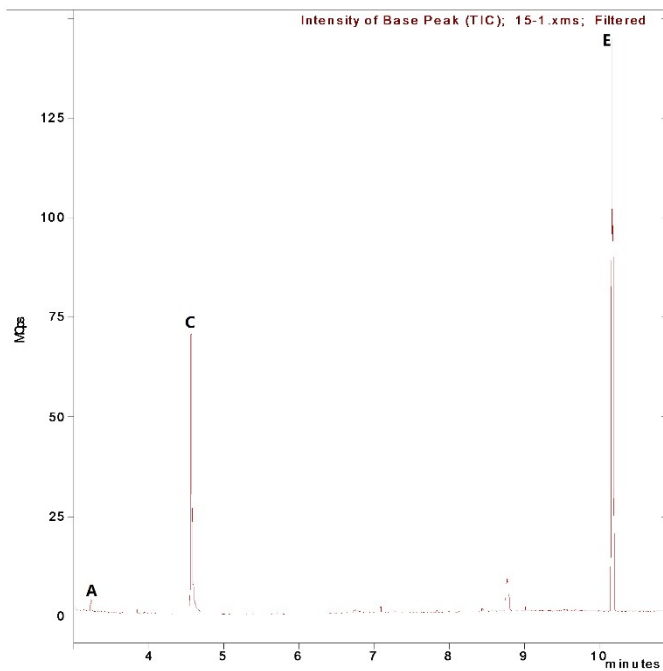


Fig. S3. GC-MS spectrum of the reaction for 120 min
A: chlorobenzene r.t.=3.222 min; C:iodobenzene r.t.=4.561 min;
E: triphenylethylene r.t.=10.167 min

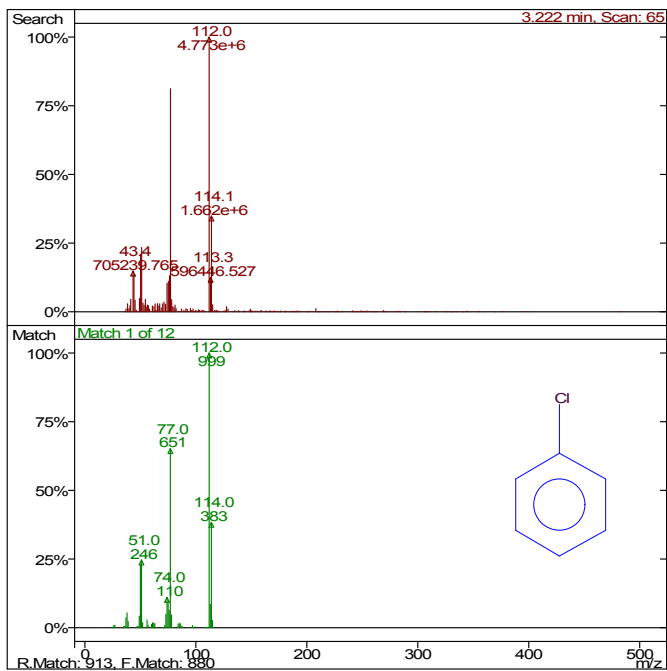


Fig. S4. MS analysis of chlorobenzene (r.t.=3.222 min)

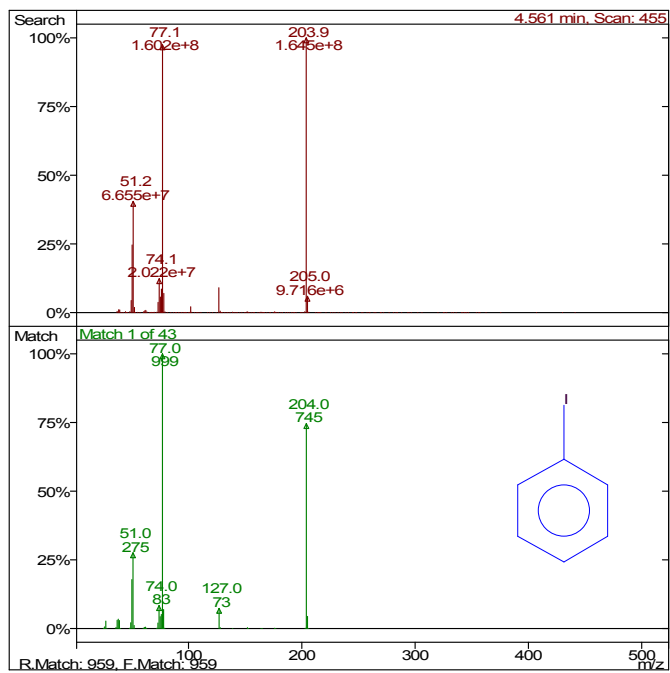


Fig. S5. MS analysis of iodobenzene (r.t.=4.561 min)

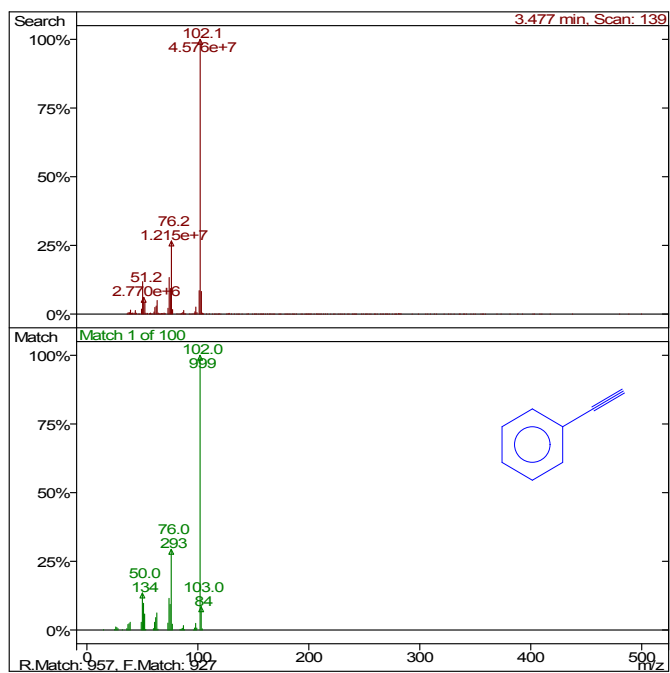


Fig. S6. MS analysis of phenylacetylene (r.t.=3.477 min)

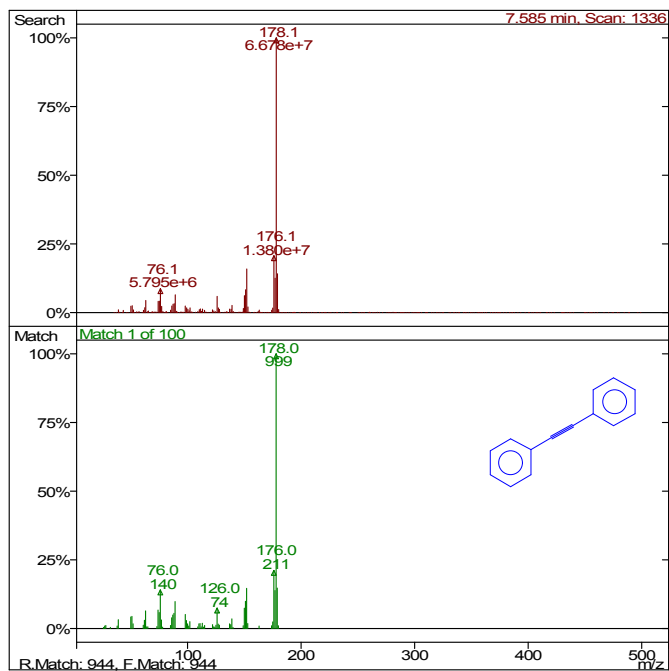


Fig. S7. MS analysis of diphenylacetylene (r.t.=7.585 min)

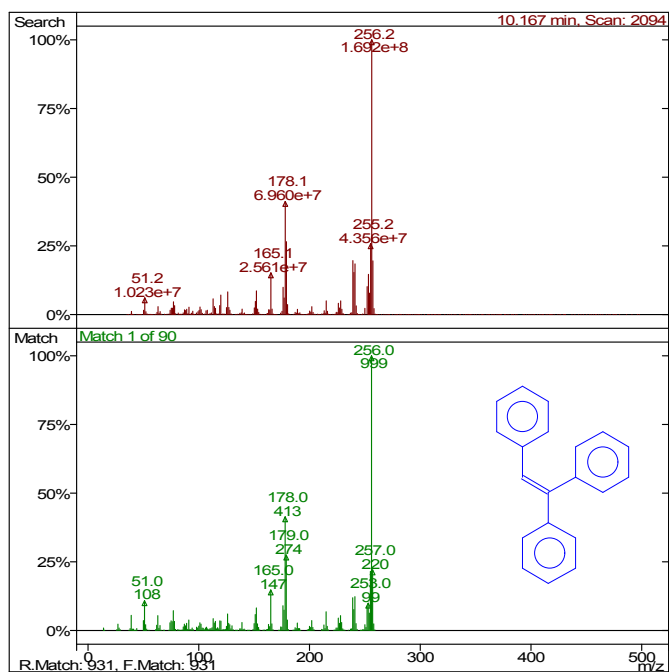


Fig. S8. MS analysis of triphenylethylene (r.t.=10.167 min)

4. Pd/PhCl Catalyzed Reaction of 1-Hexyne and Iodobenzene

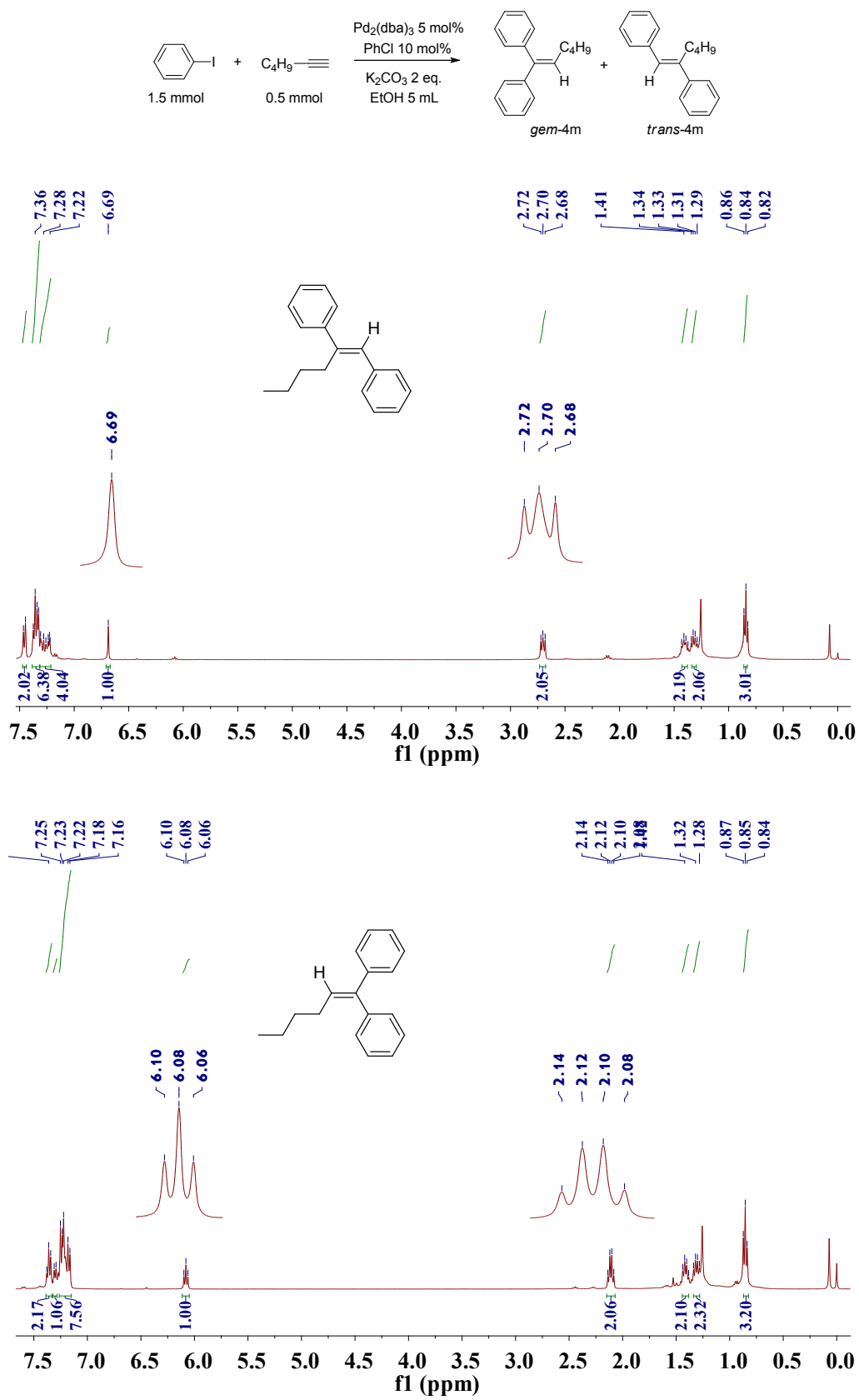
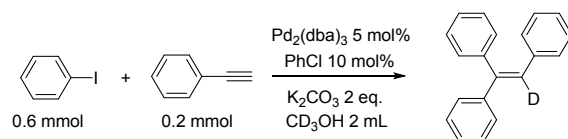


Fig. S9. Identification of *trans*- and *gem*- isomers by the coupling of vinyl- protons: a. Vinyl proton as singlet at 6.69ppm; b. Vinyl proton as triplet at 6.08ppm

5. Deuterium Labeling Experiment



$\text{Pd}_2(\text{dba})_3$ (0.0046 g) and K_2CO_3 (0.0552 g) in sealed Schlenk tubes, vacuum down and refilled with nitrogen for three times. Under atmosphere of N_2 , phenylacetylene (0.2 mmol), iodobenzene (0.6 mmol), chlorobenzene 10 mol%, 2 mL CD_3OH , at 70 °C, stirring 4 h. End of reaction, room temperature, the crude products were purified by column chromatography on silica gel using petroleum ether as the eluent. Isolated yield 78%.

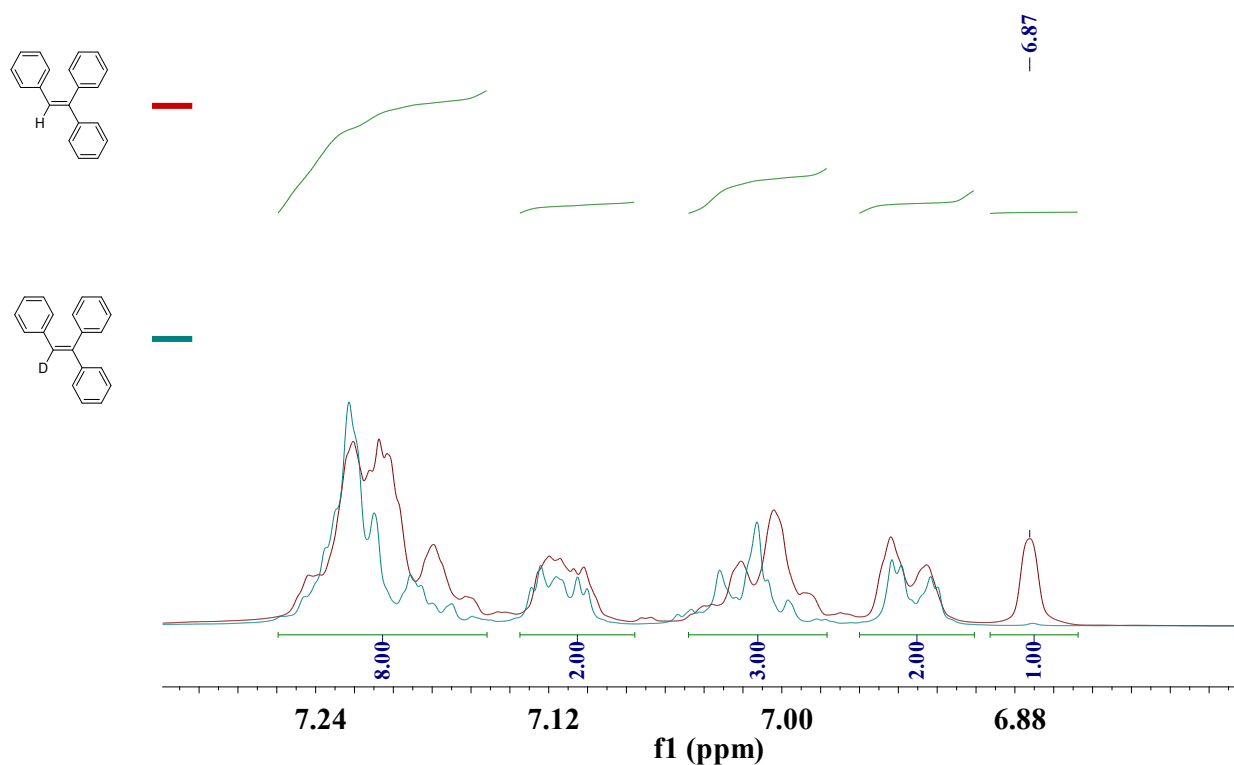
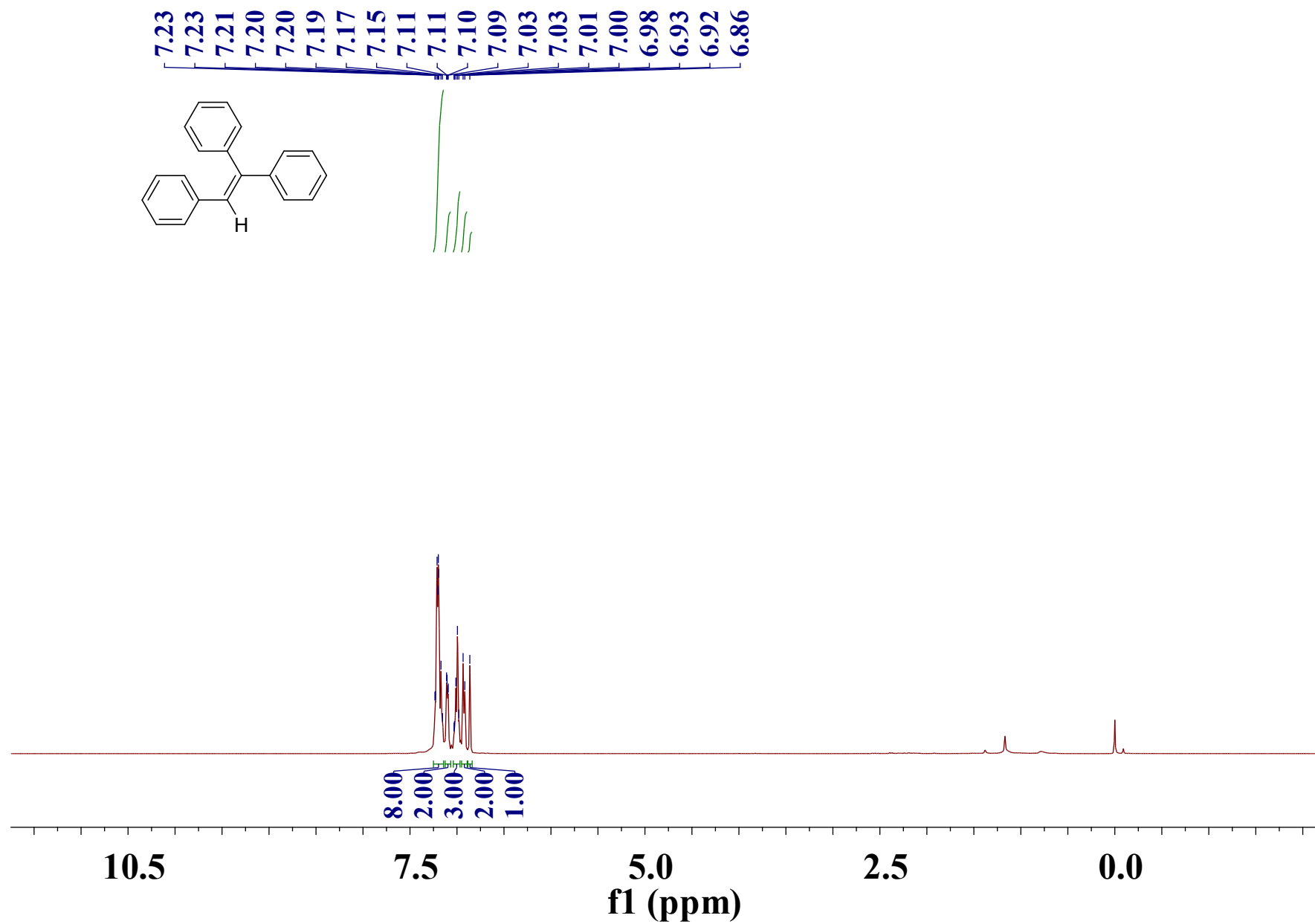


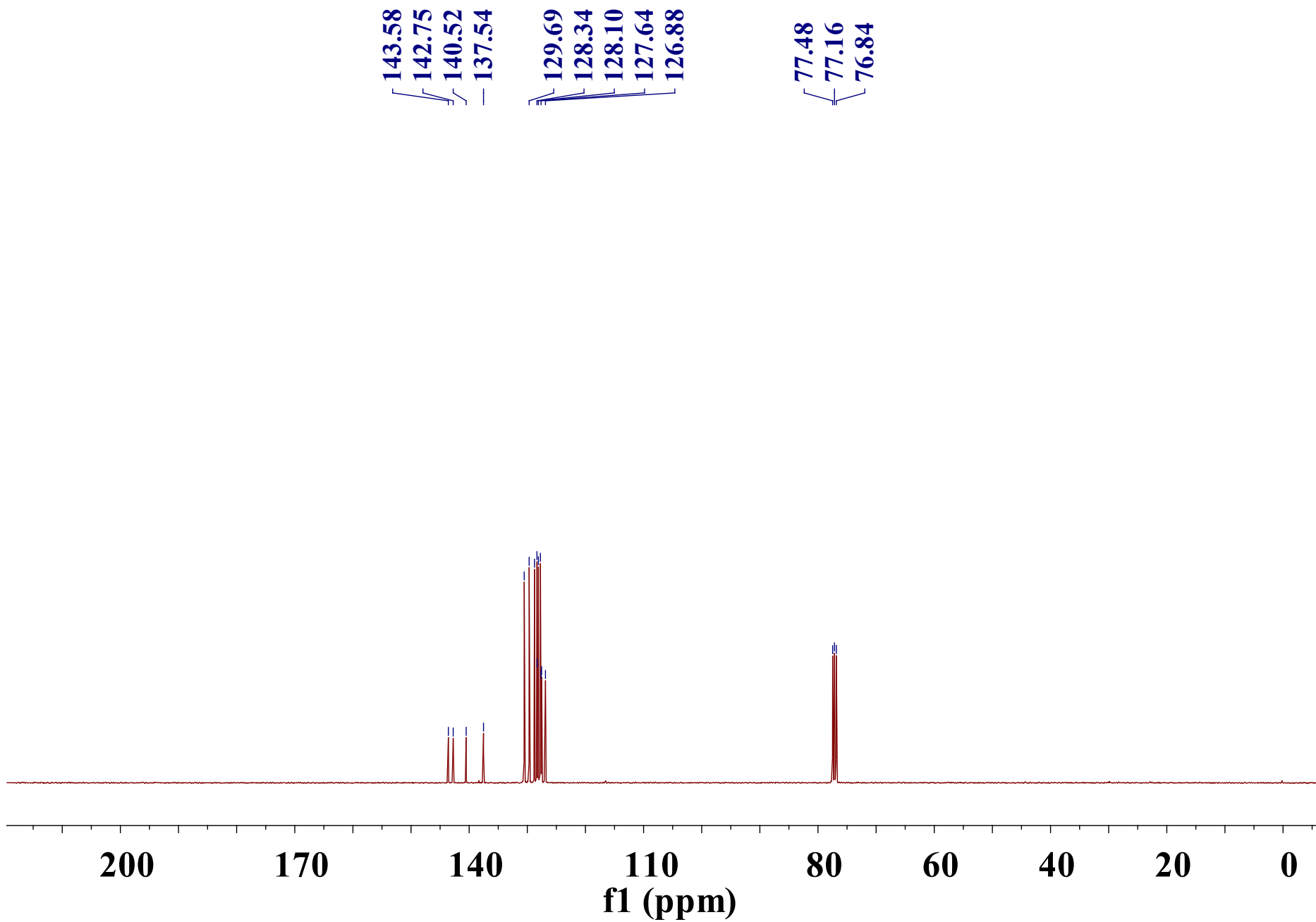
Fig. S10. $^1\text{H-NMR}$ spectra of triarylethenes (red) and deuterium labeled triarylethenes (green)

6. Copies of ^1H NMR and ^{13}C NMR of compounds

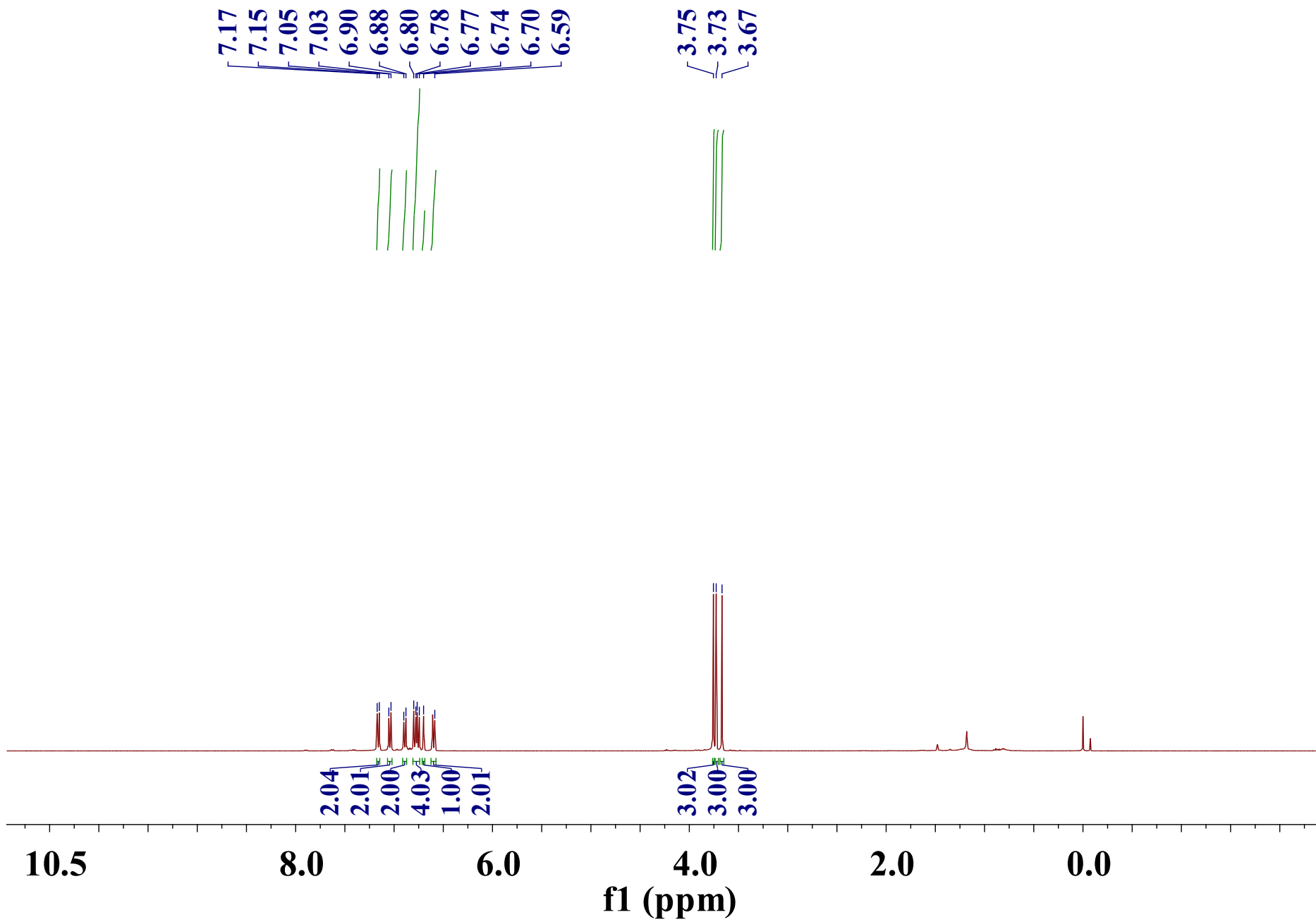
4a ^1H NMR spectrum



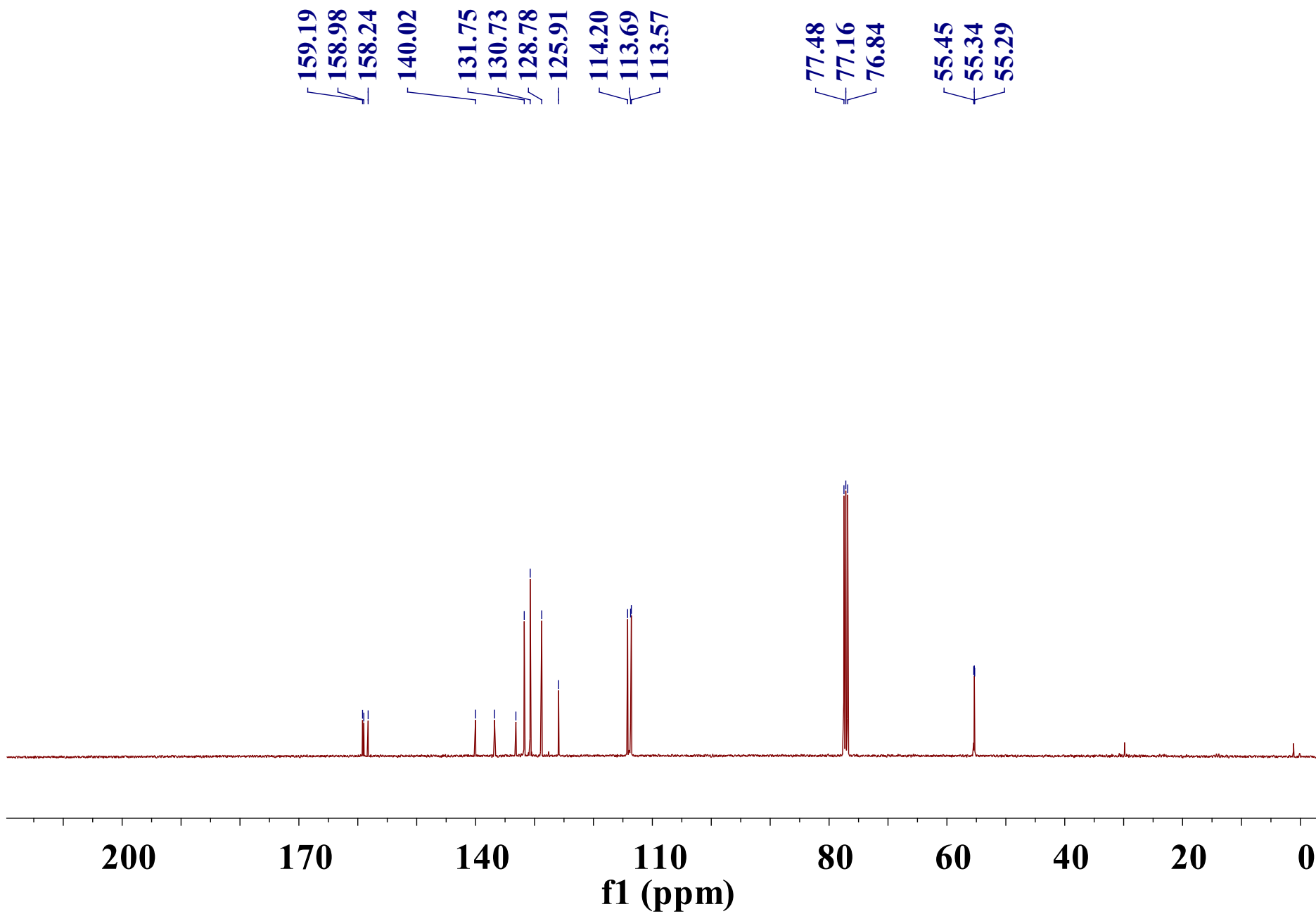
4a ^{13}C NMR spectrum



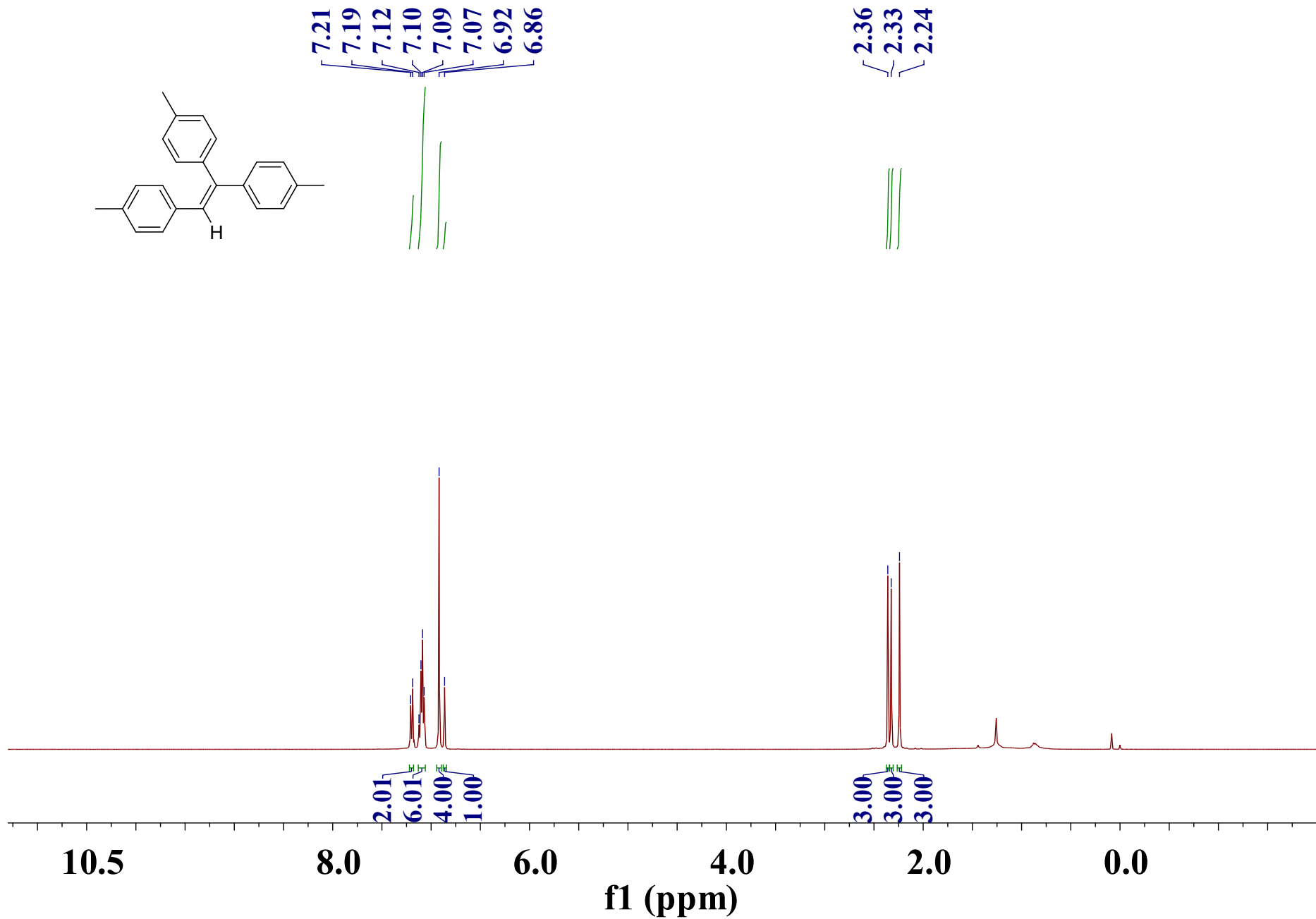
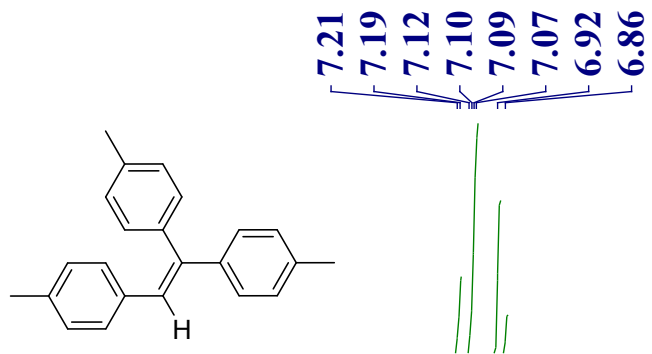
4b ¹³C NMR spectrum



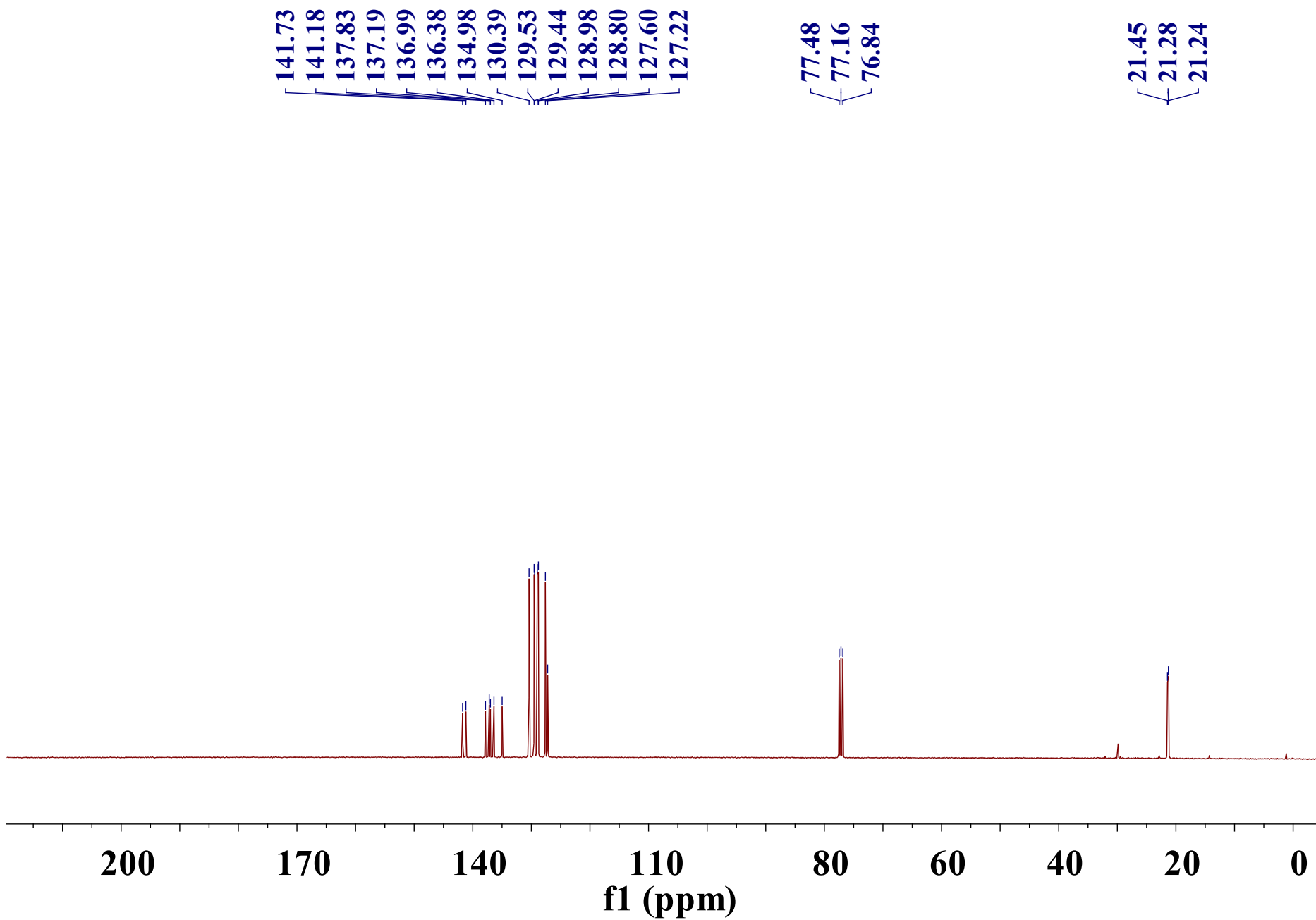
4b ¹³C NMR spectrum



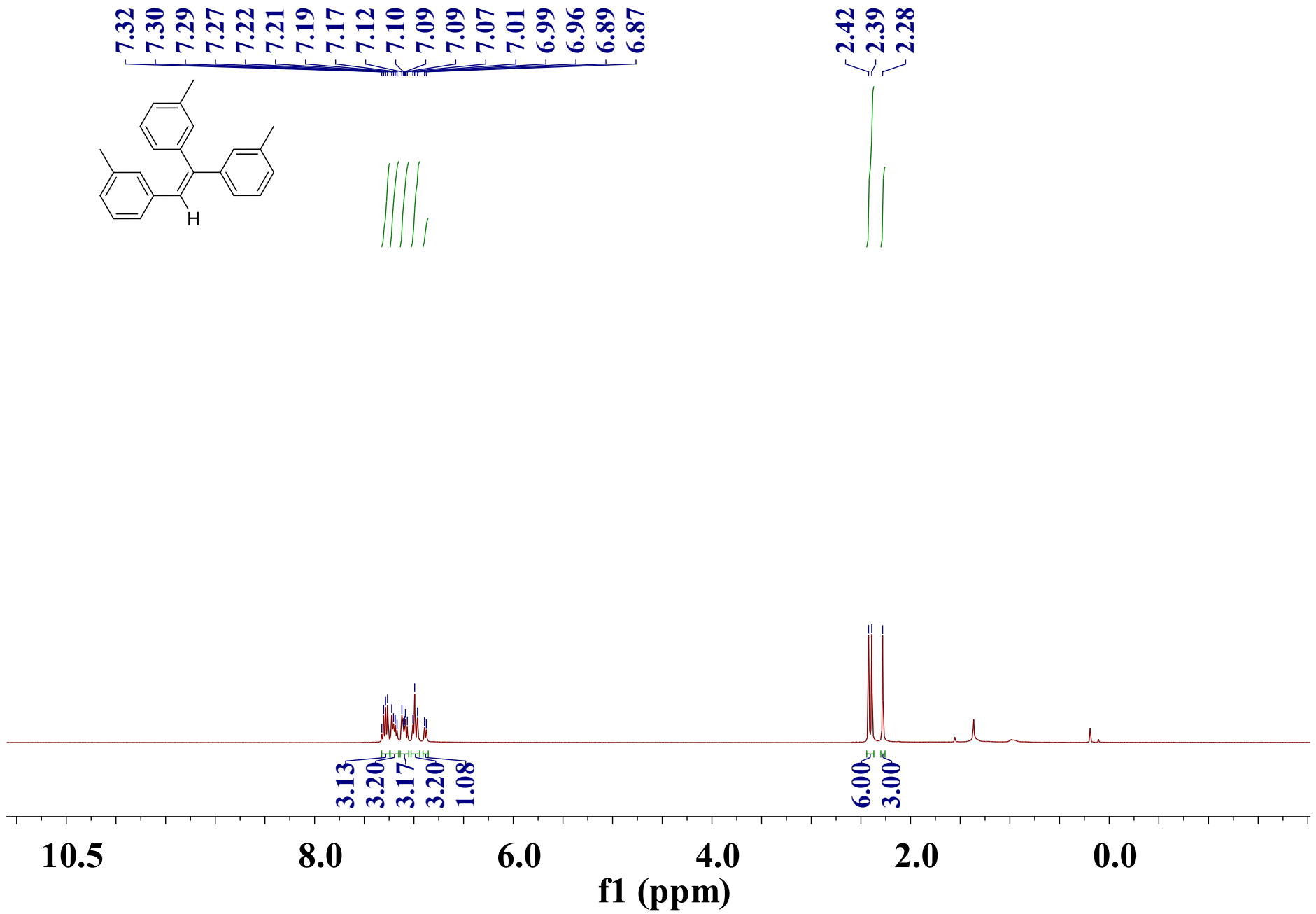
4c ¹³C NMR spectrum



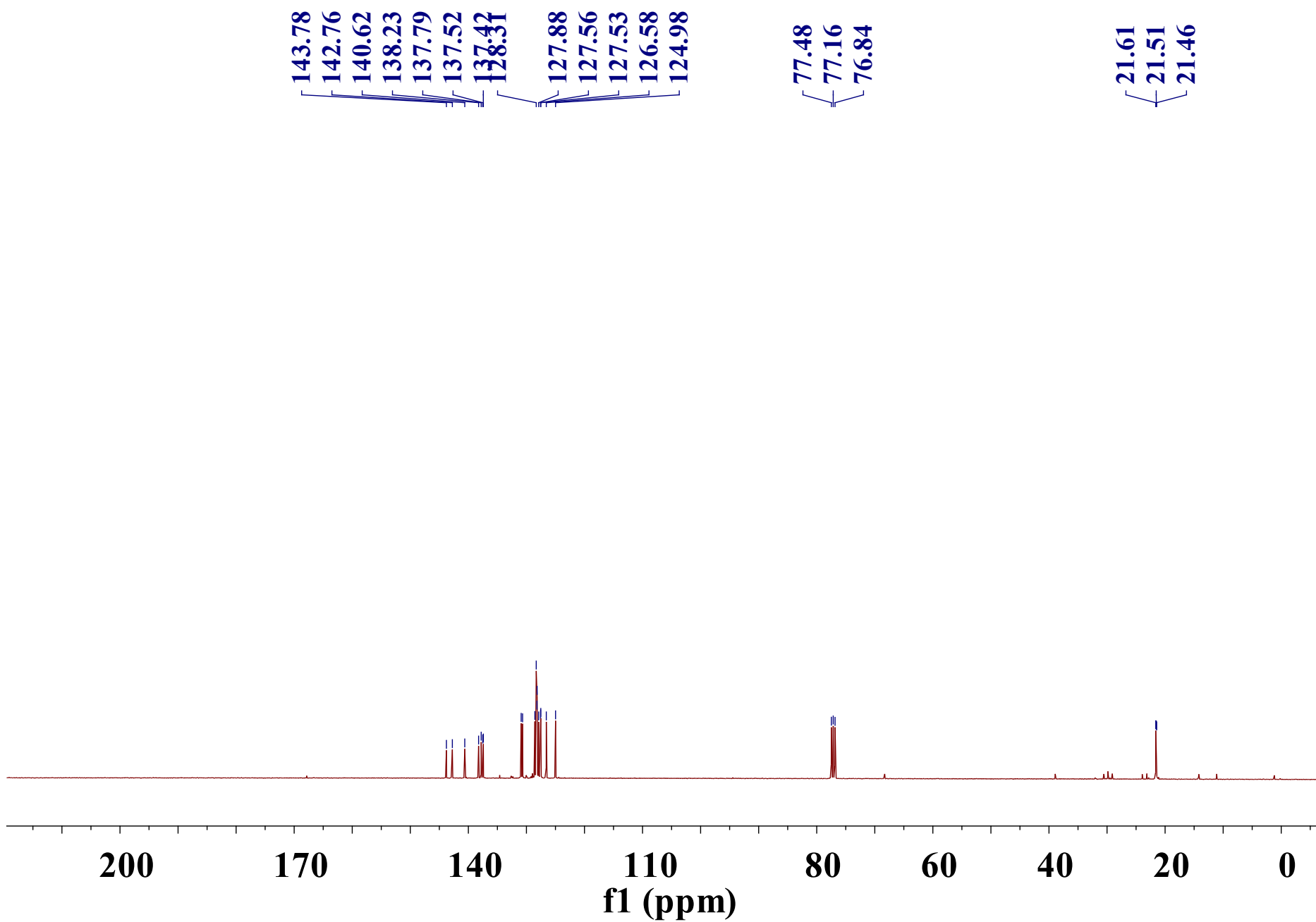
4c ^{13}C NMR spectrum



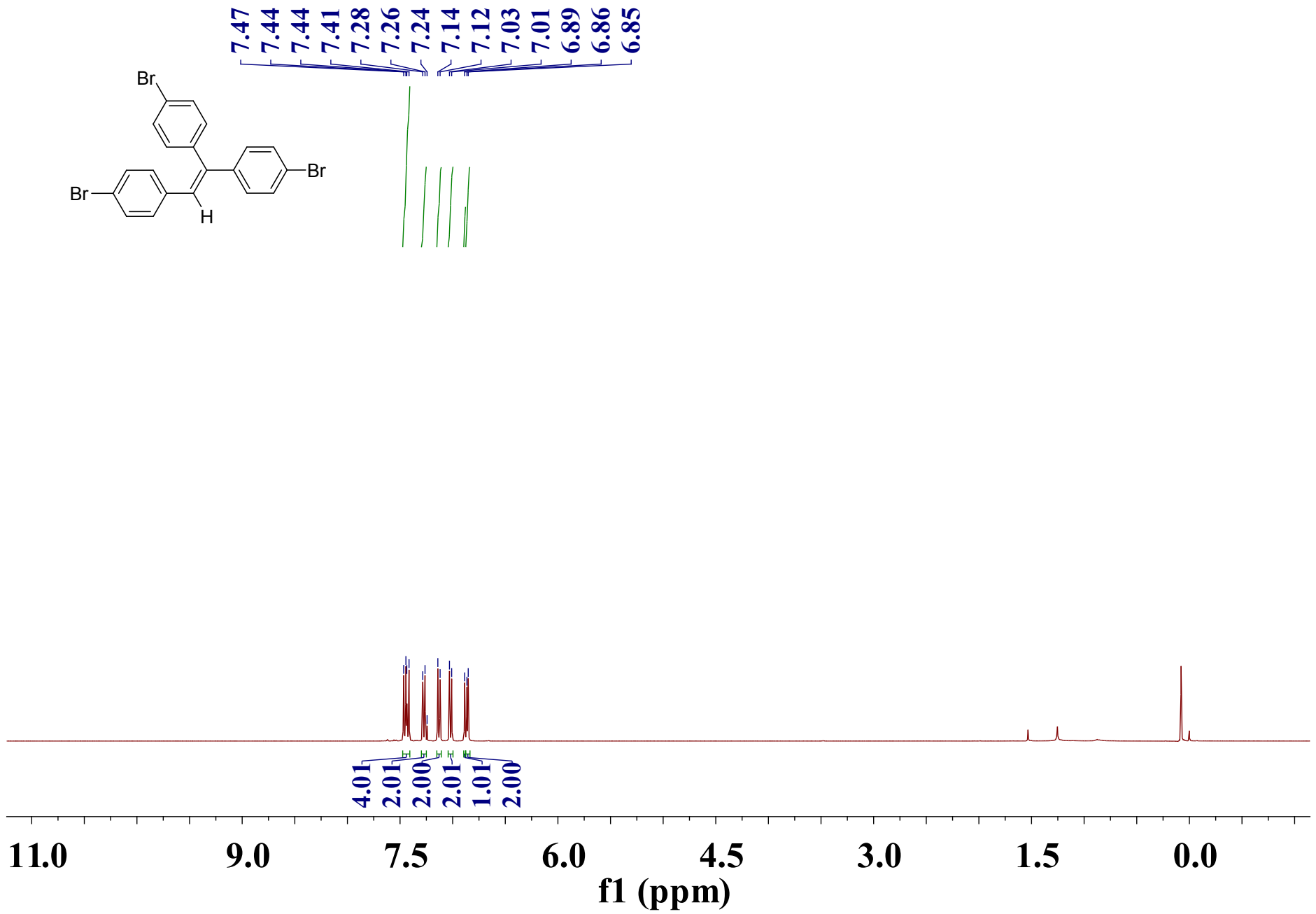
4d ¹³C NMR spectrum



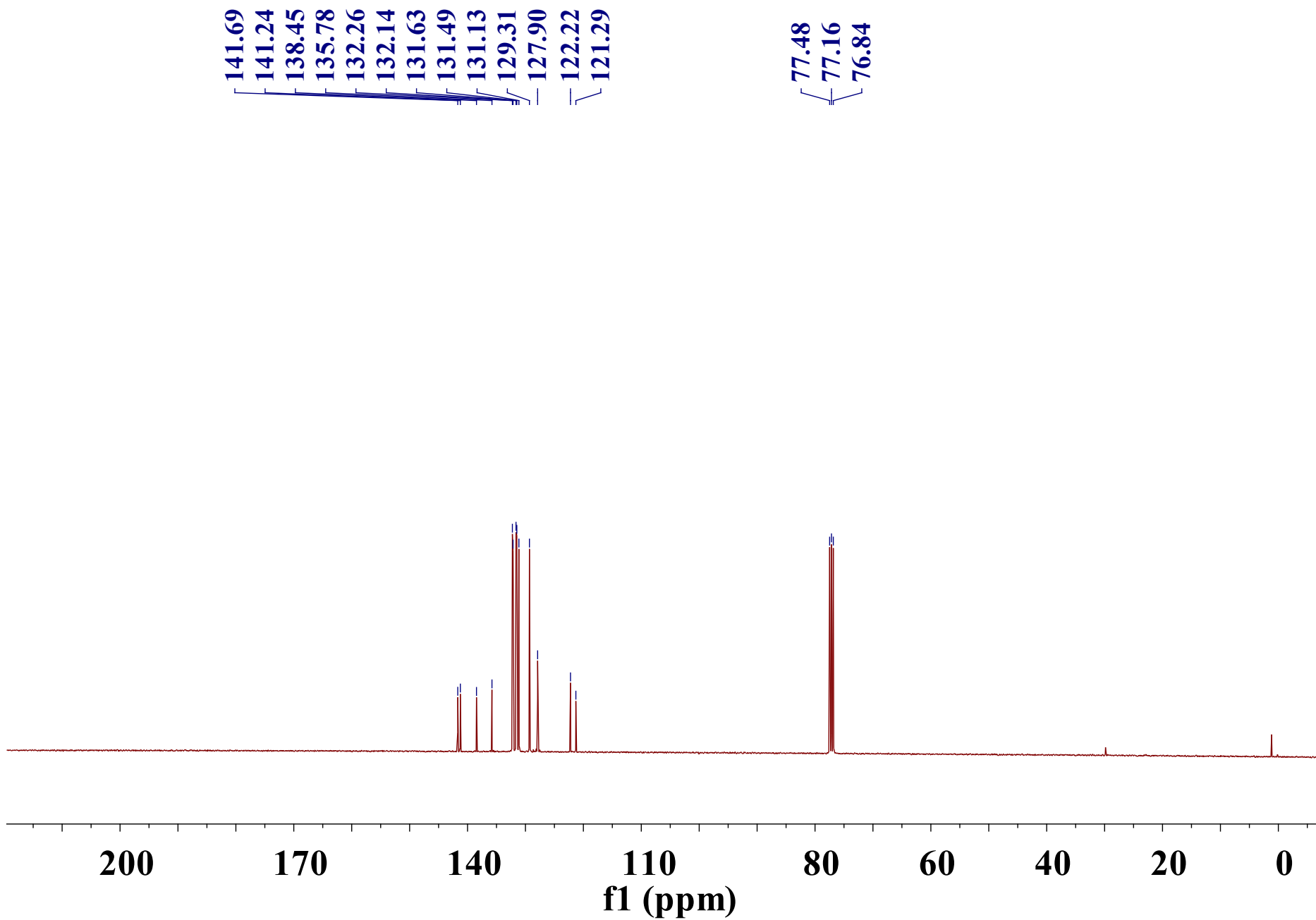
4d ¹³C NMR spectrum



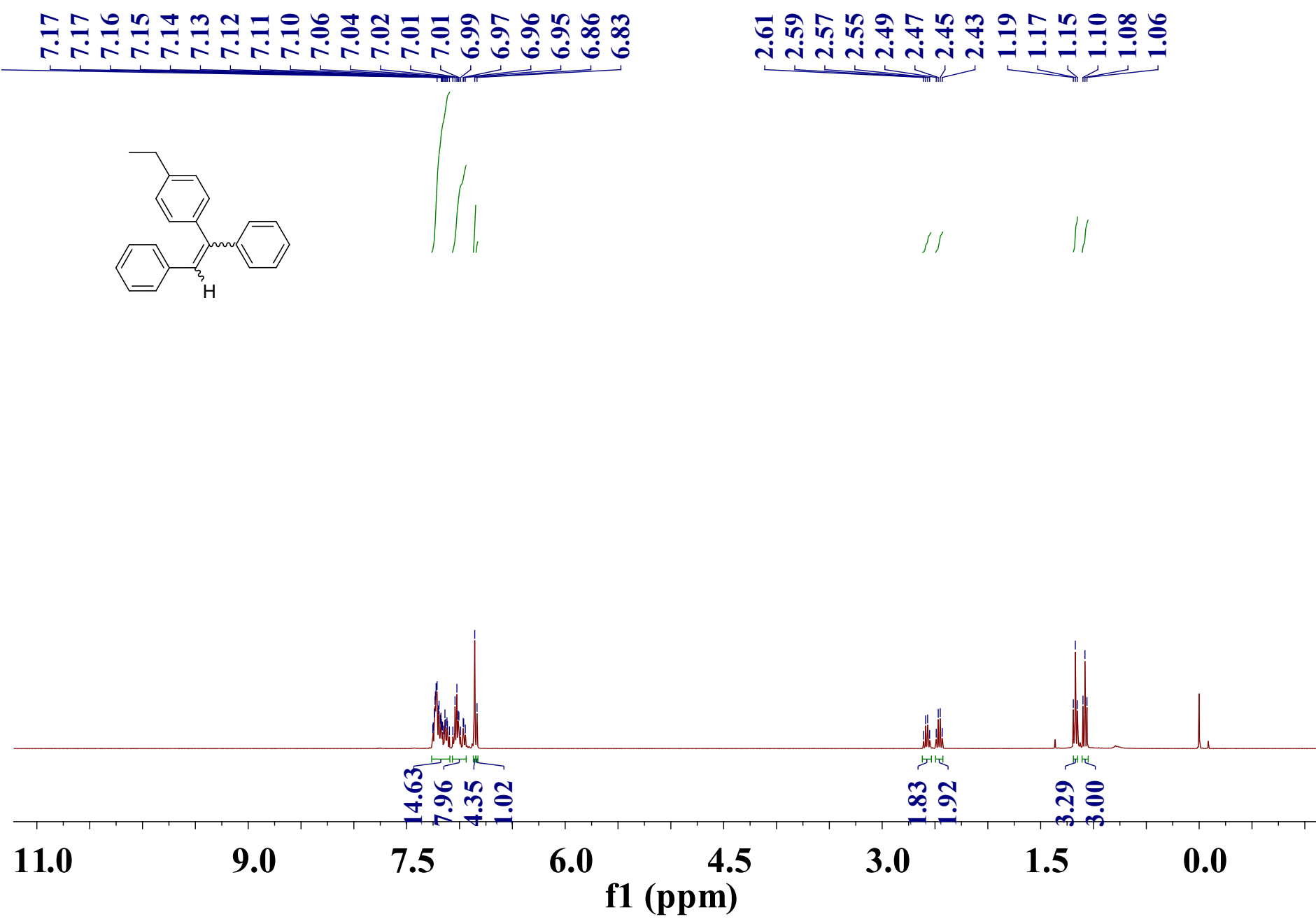
4e ¹³C NMR spectrum



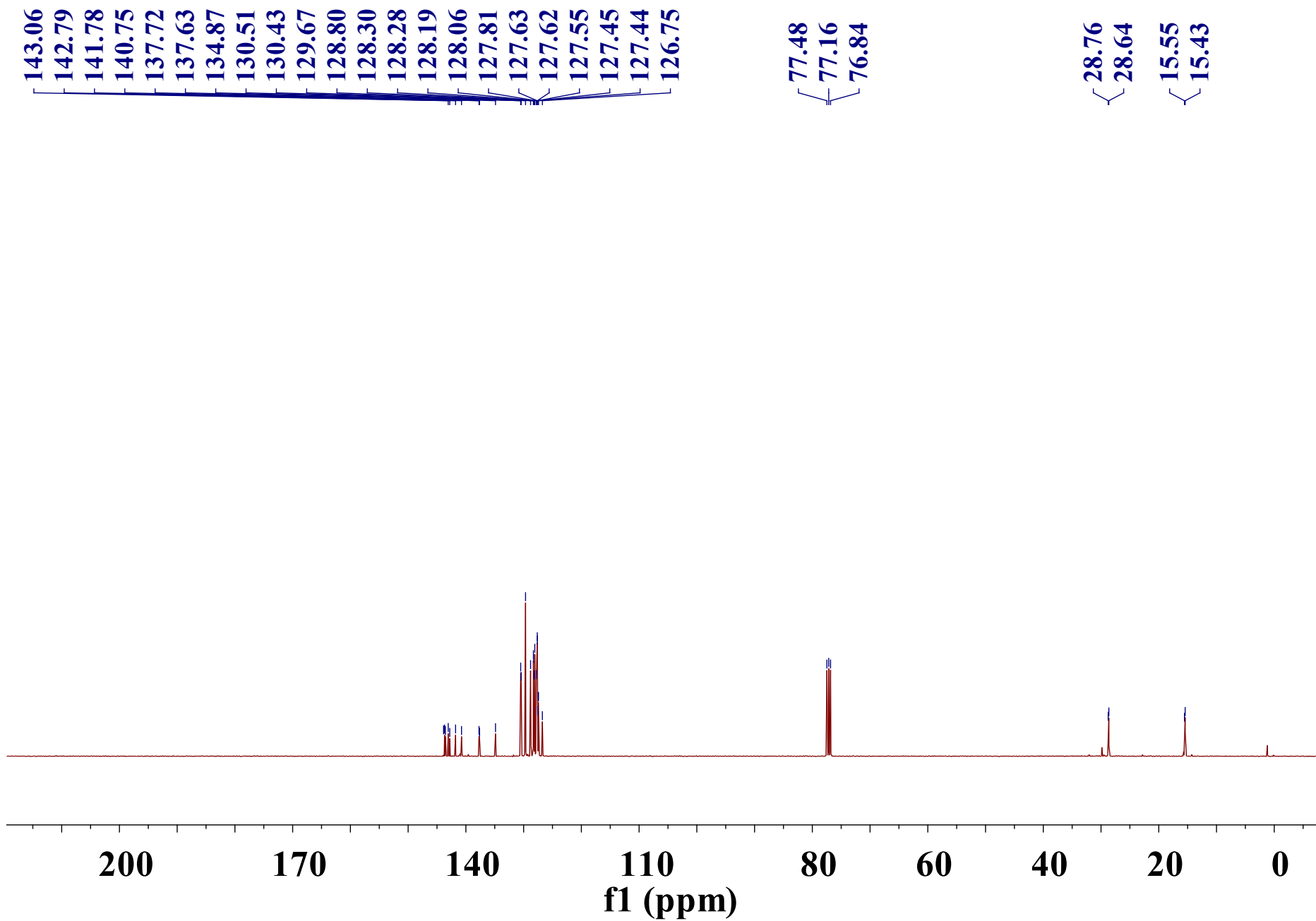
4e ¹³C NMR spectrum



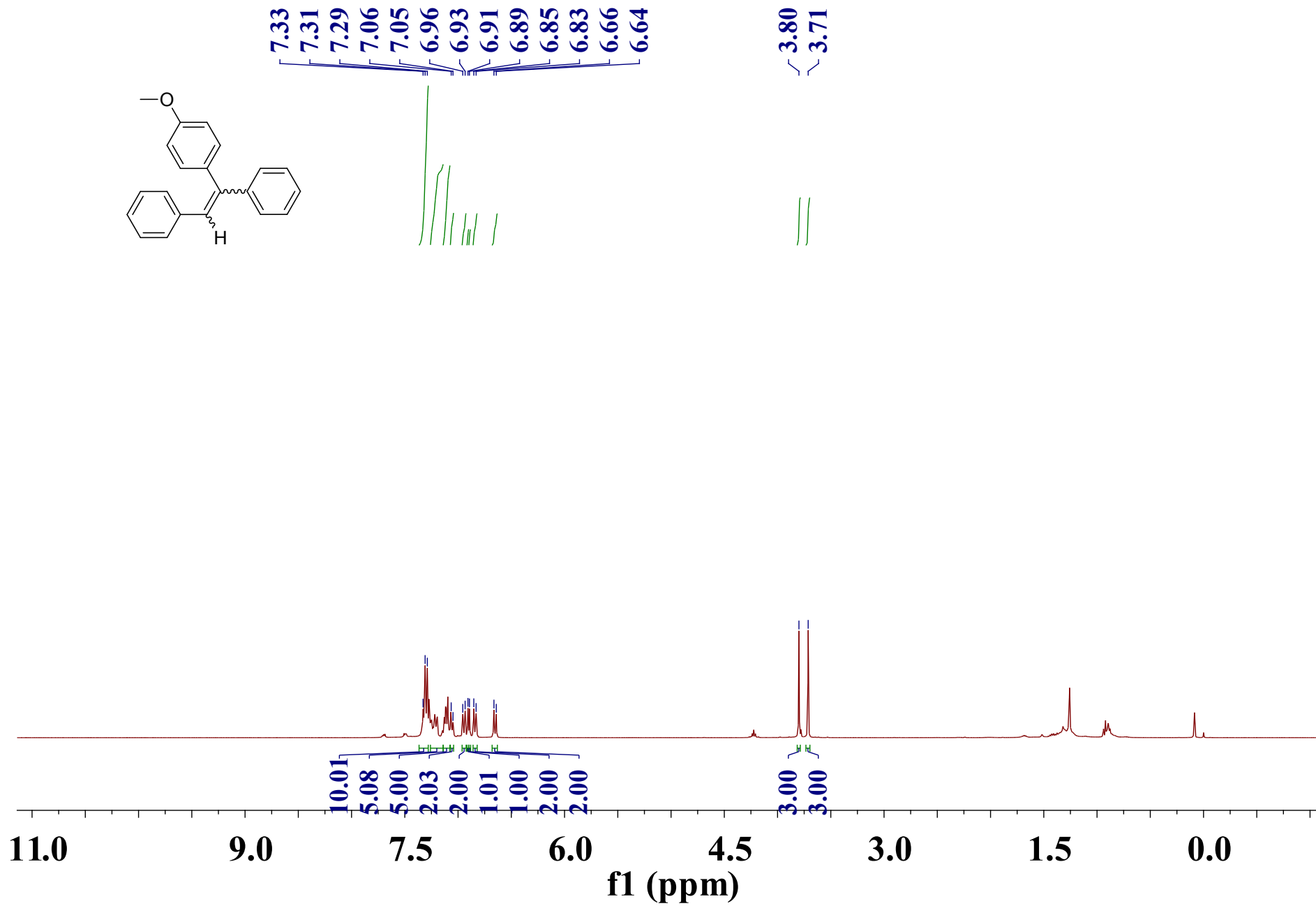
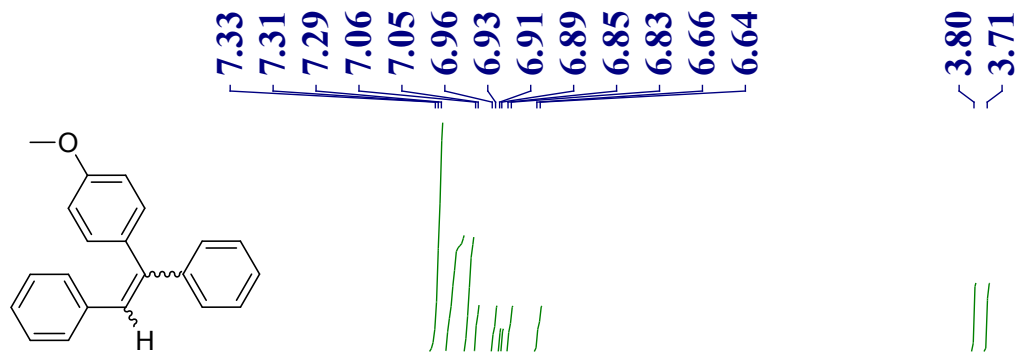
4f ¹³C NMR spectrum



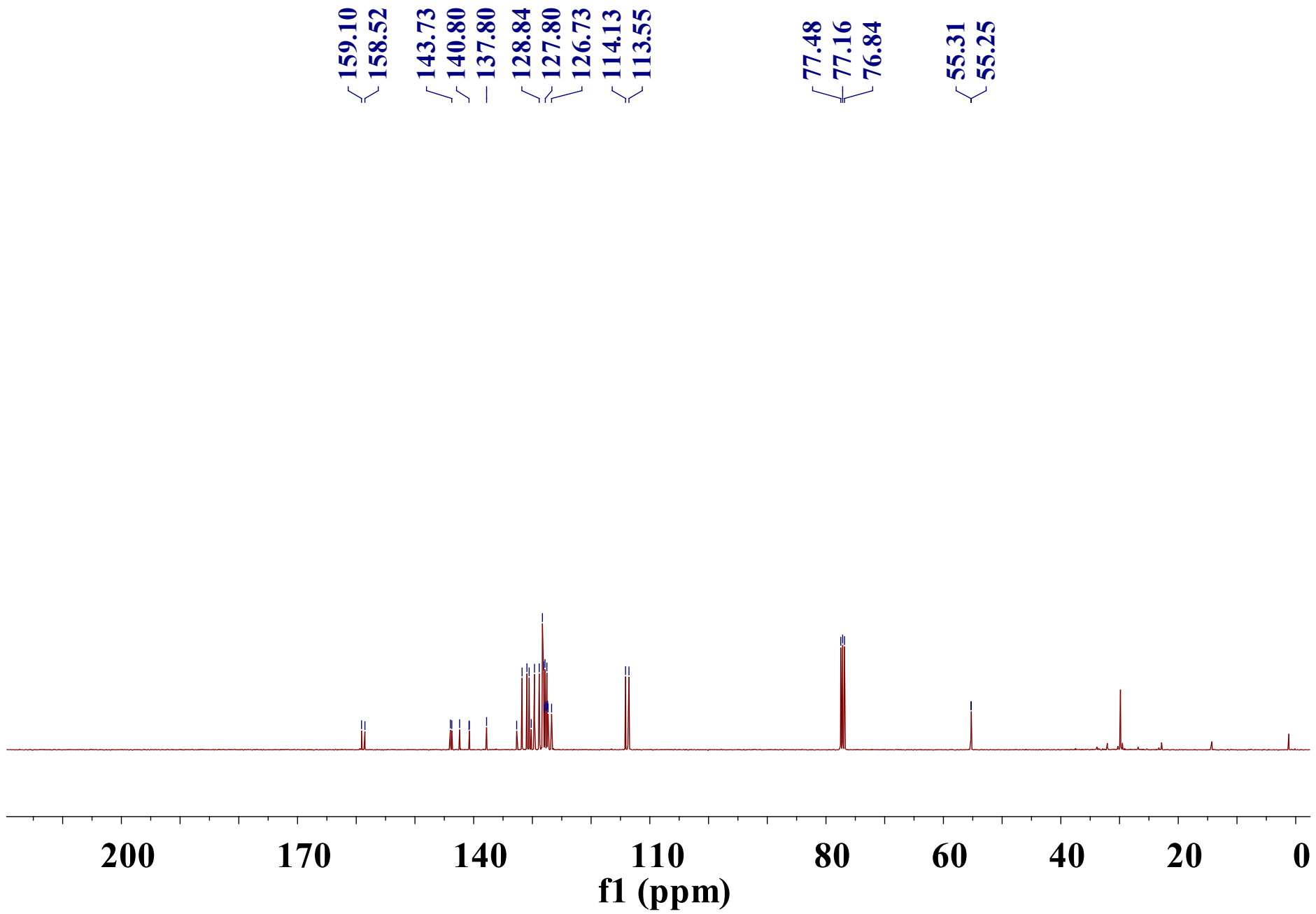
4f ¹³C NMR spectrum



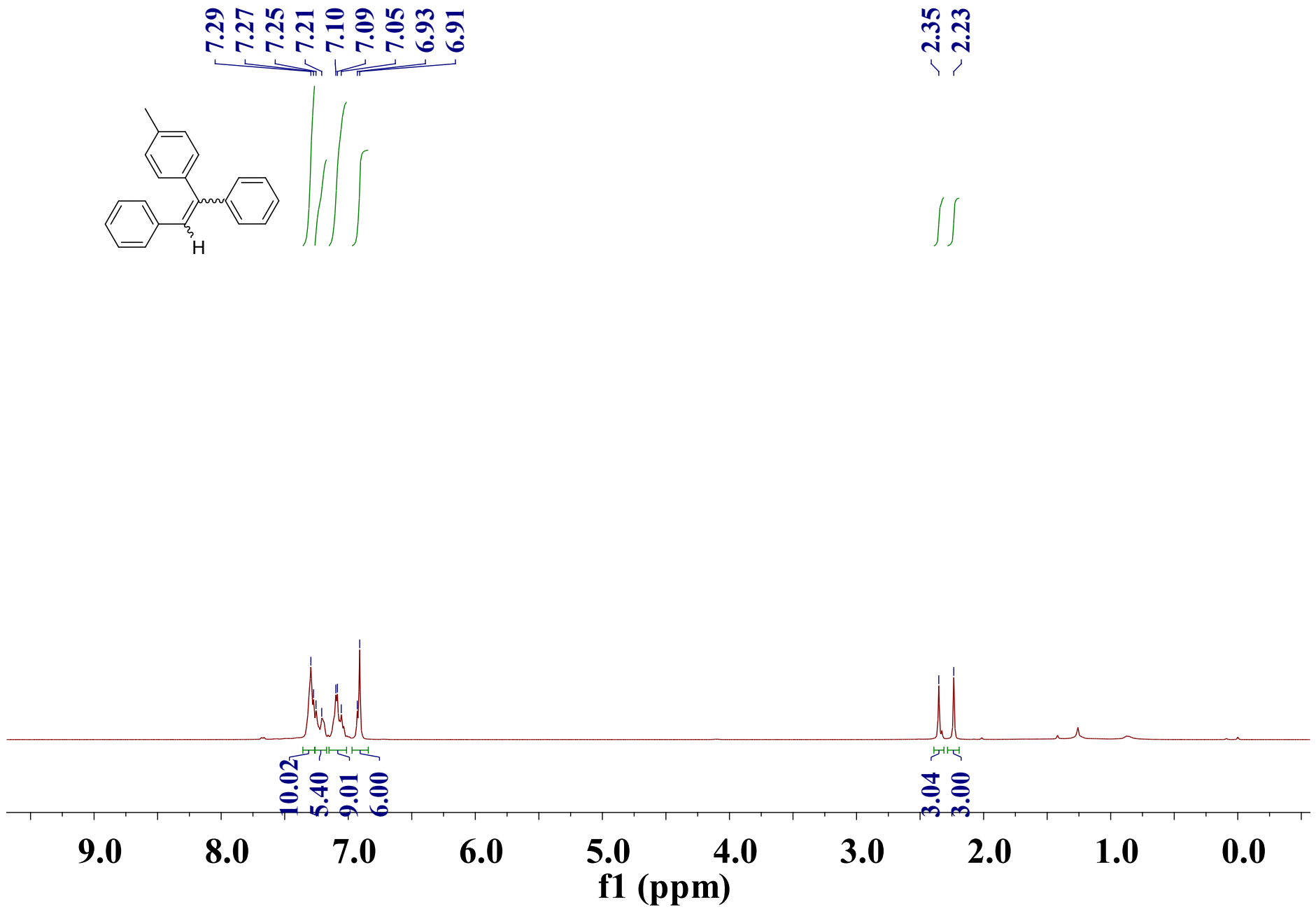
4g ¹³C NMR spectrum



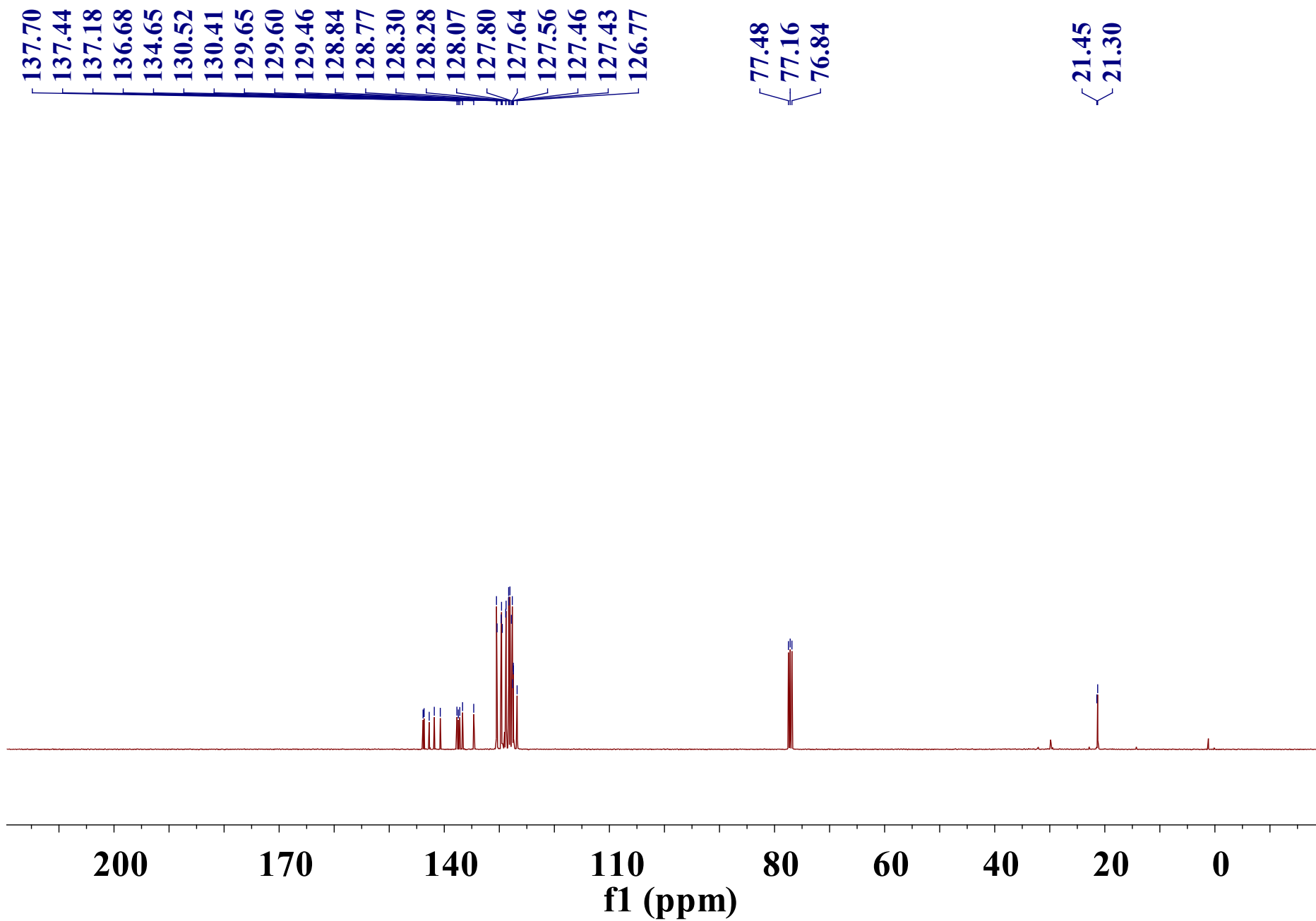
4g ^{13}C NMR spectrum



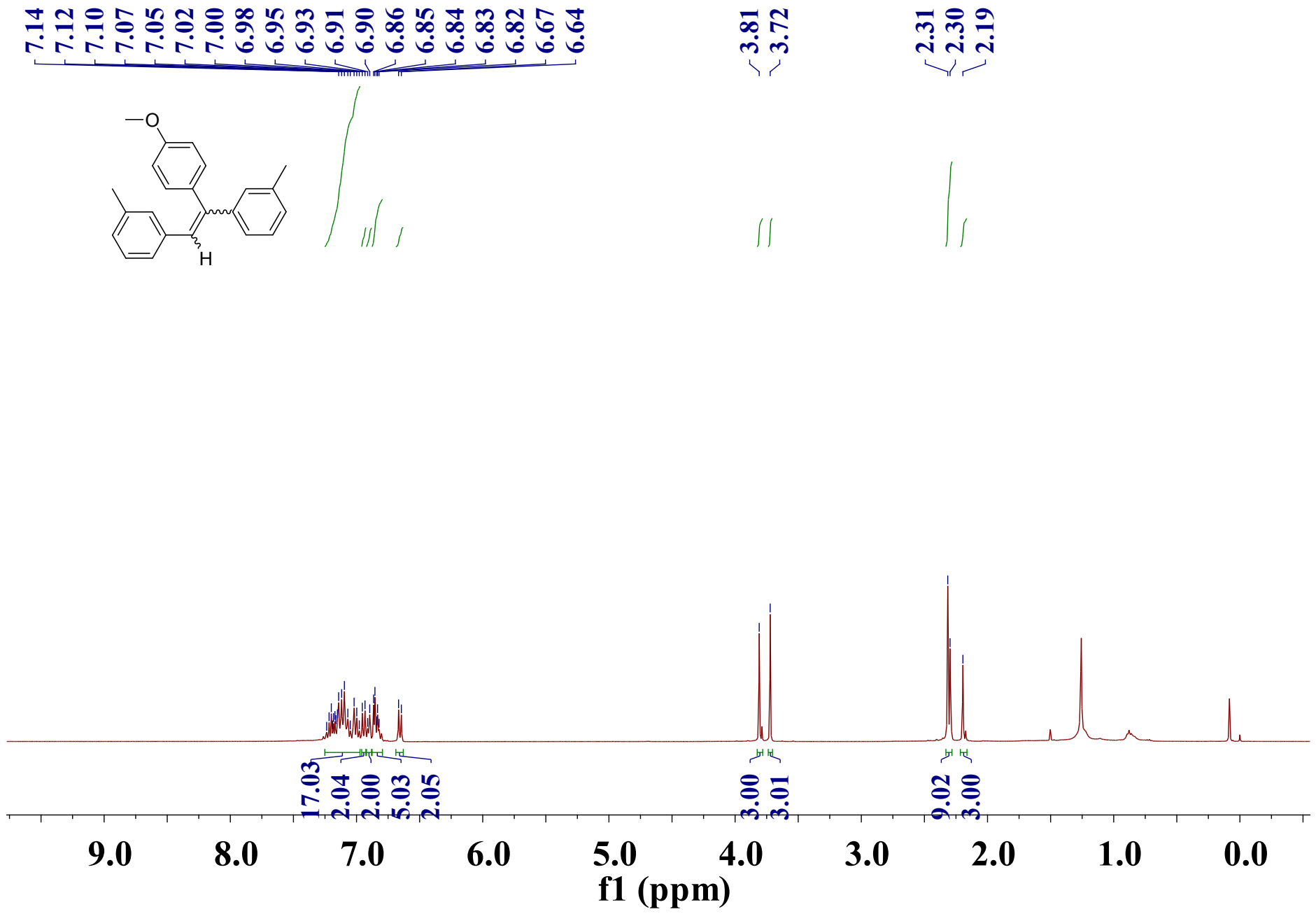
4h ¹H NMR spectrum



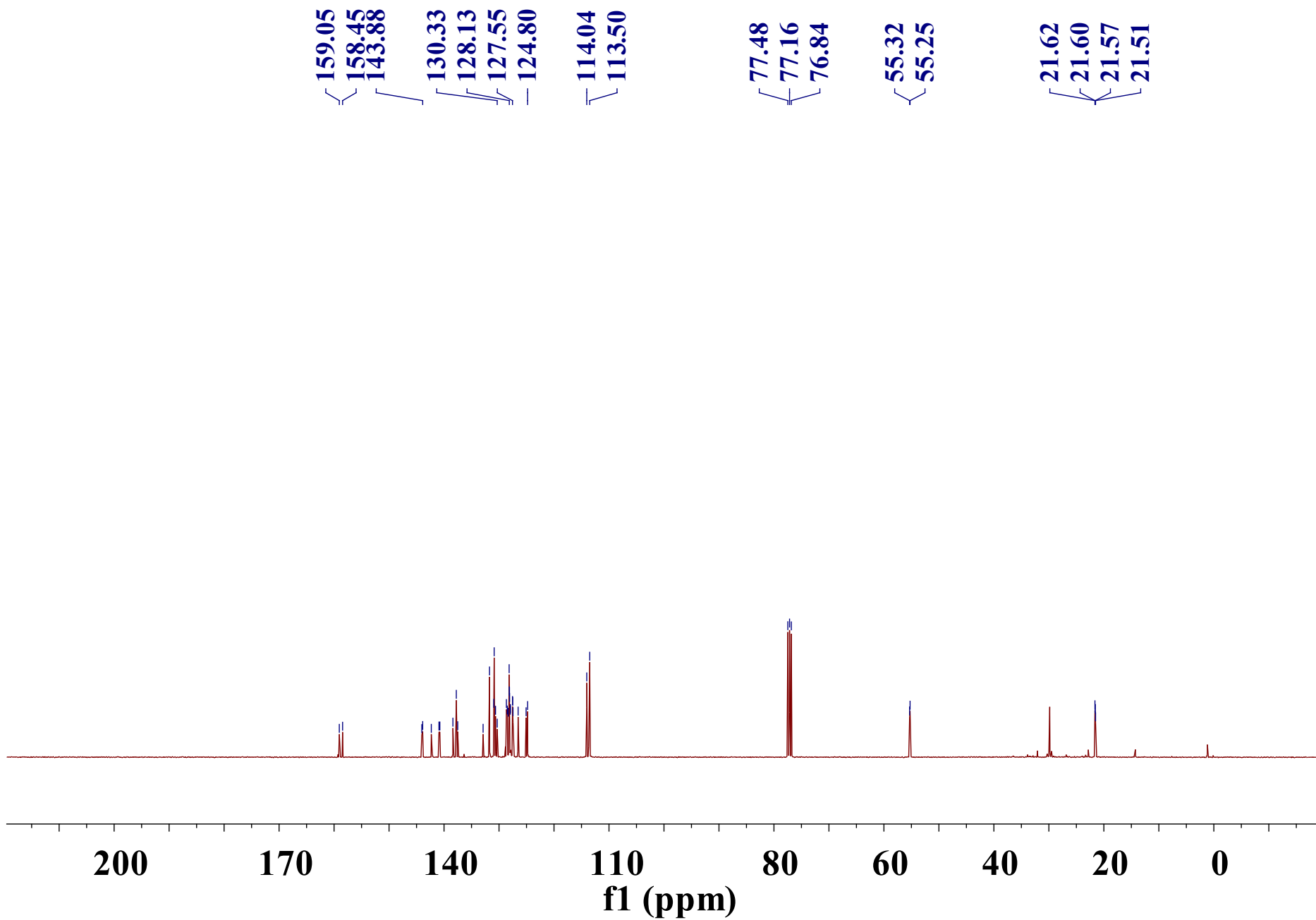
4h ¹³C NMR spectrum



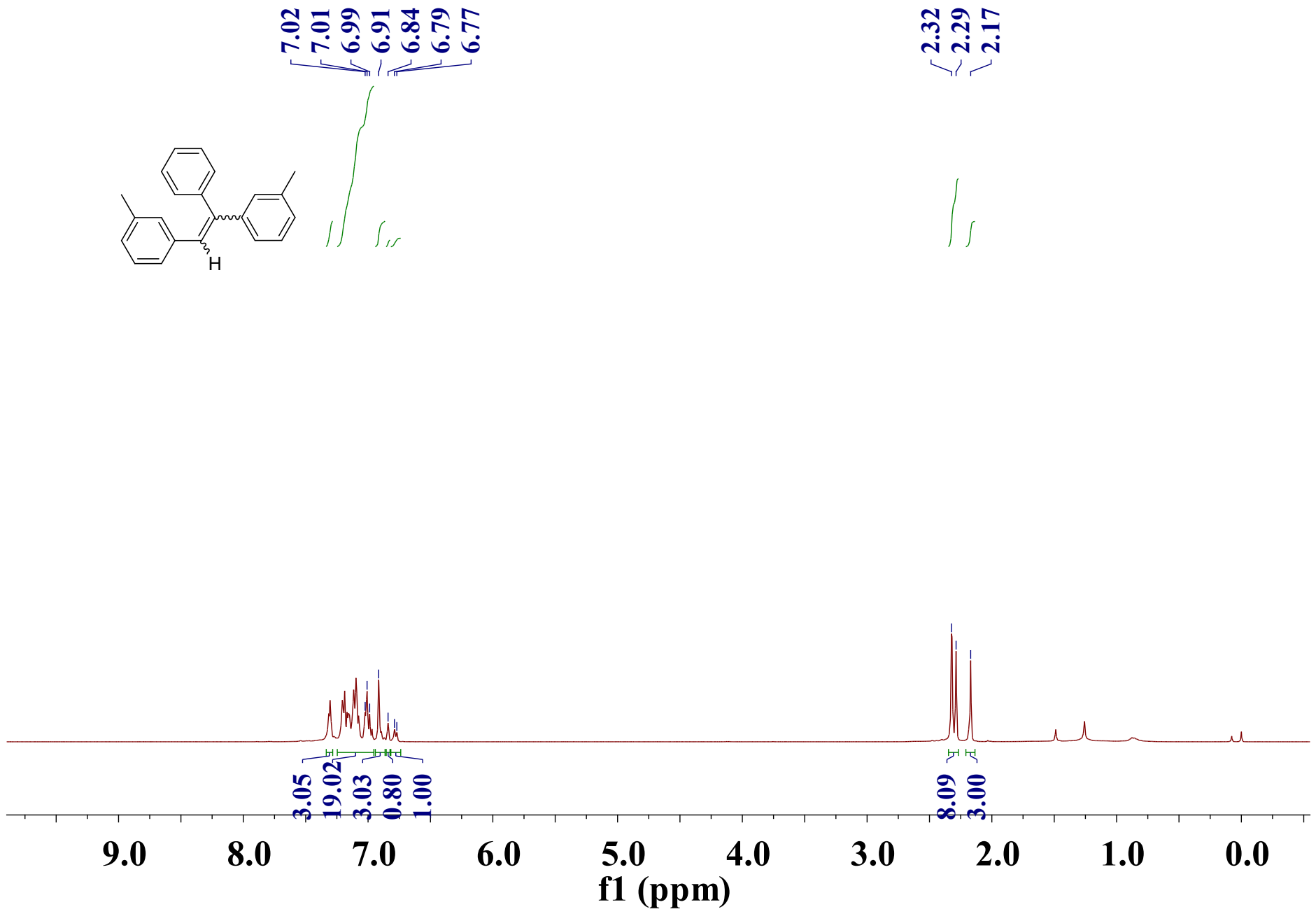
4i ¹H NMR spectrum



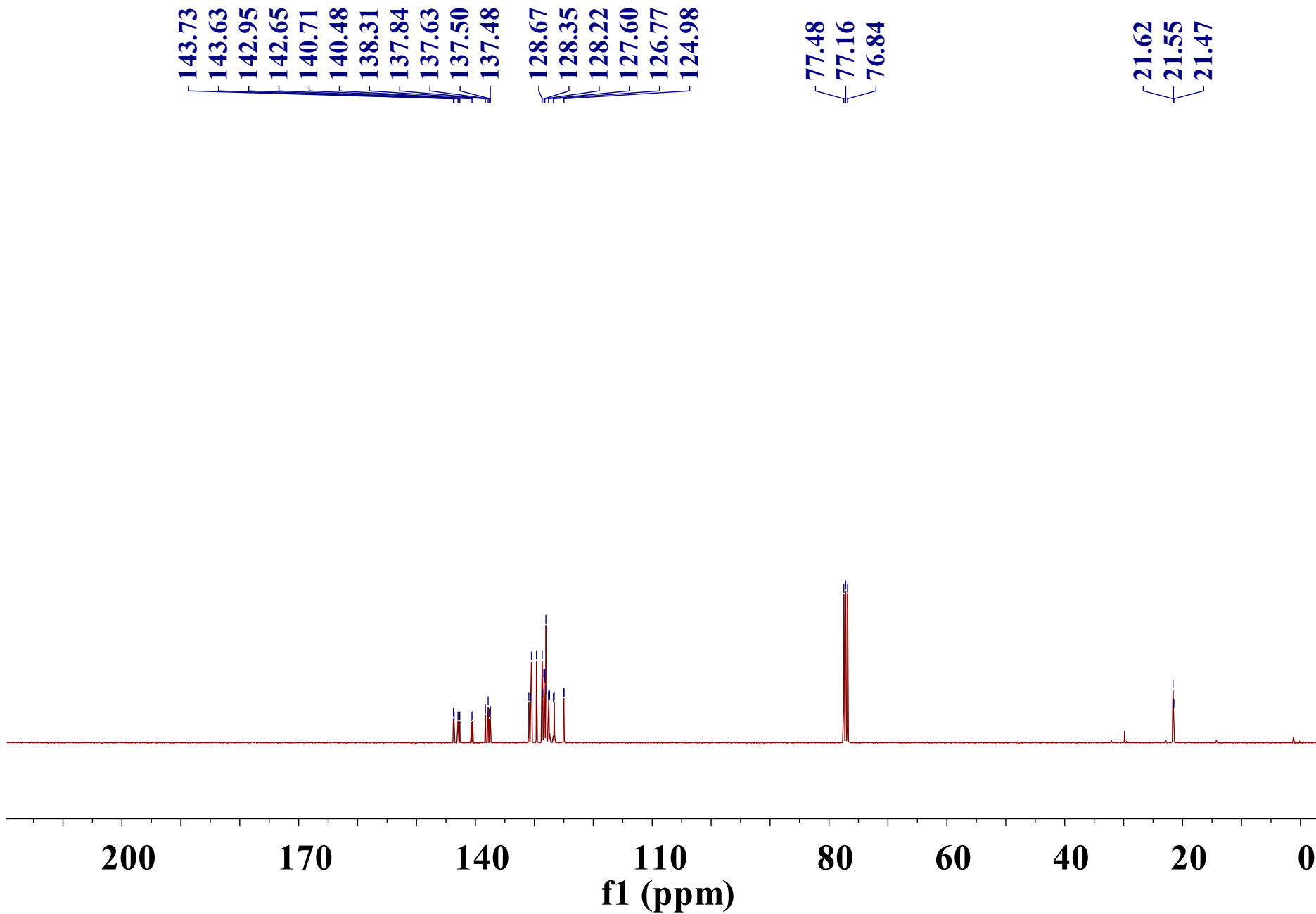
4i ¹³C NMR spectrum



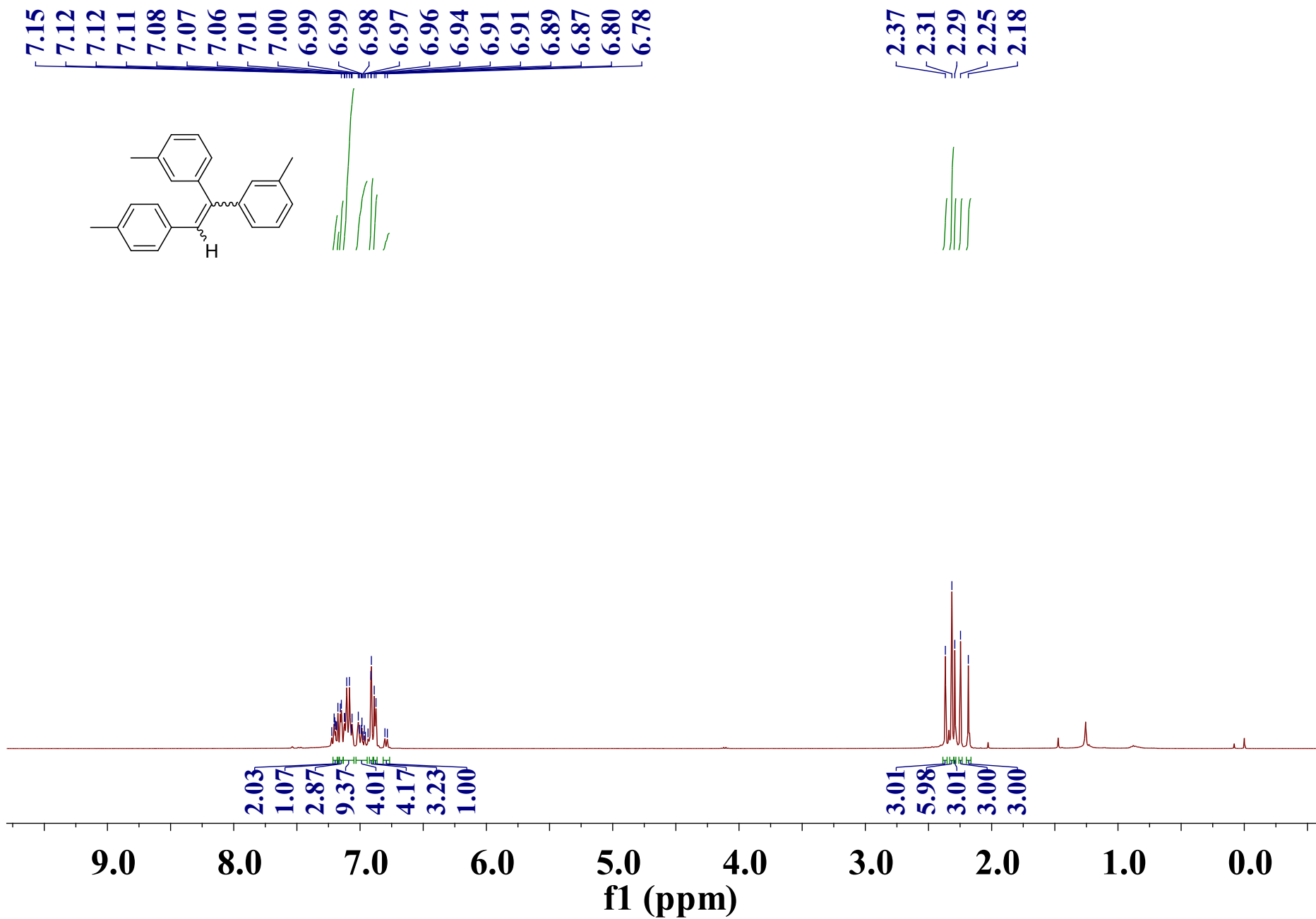
4j ¹H NMR spectrum



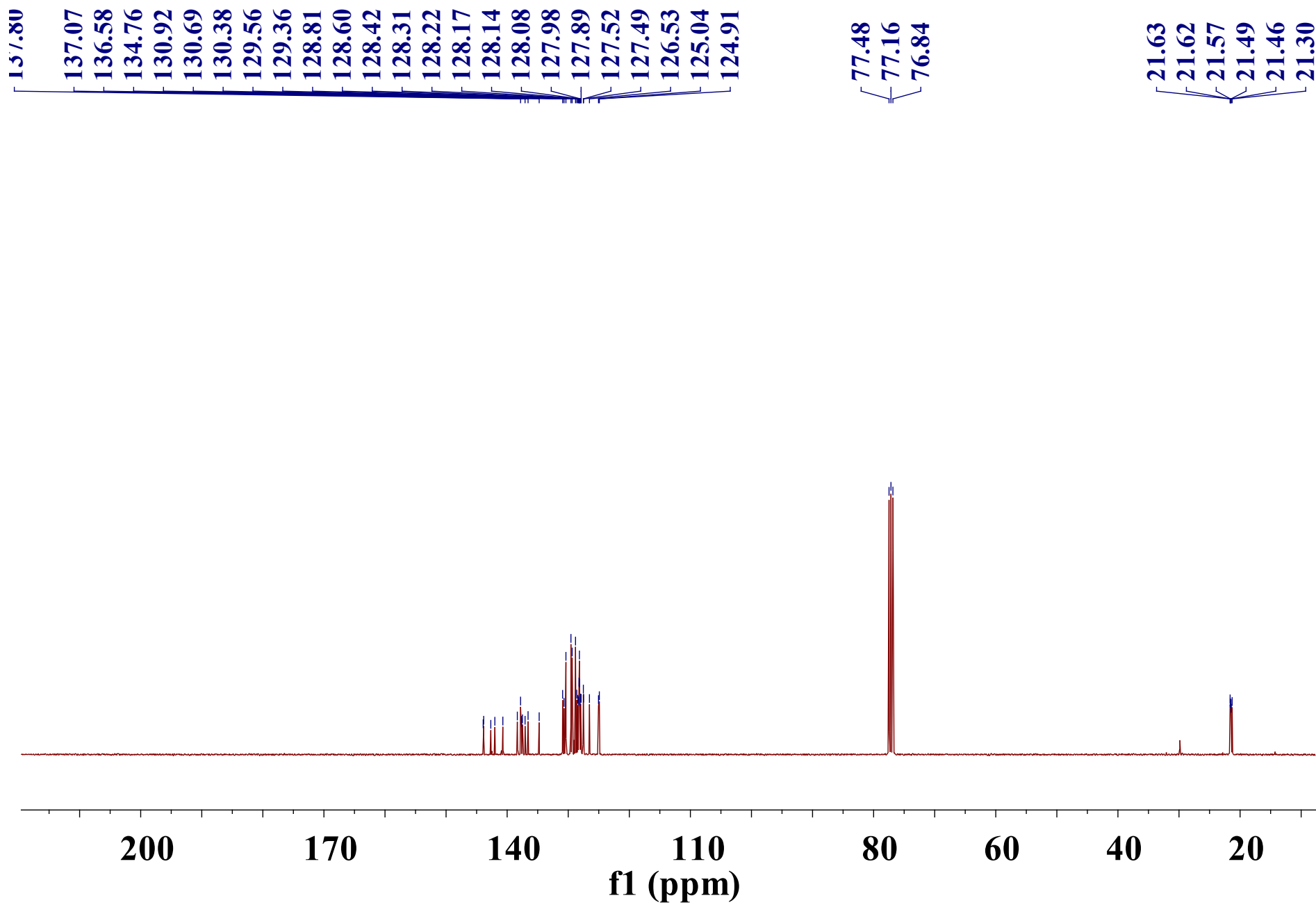
4j ¹³C NMR spectrum



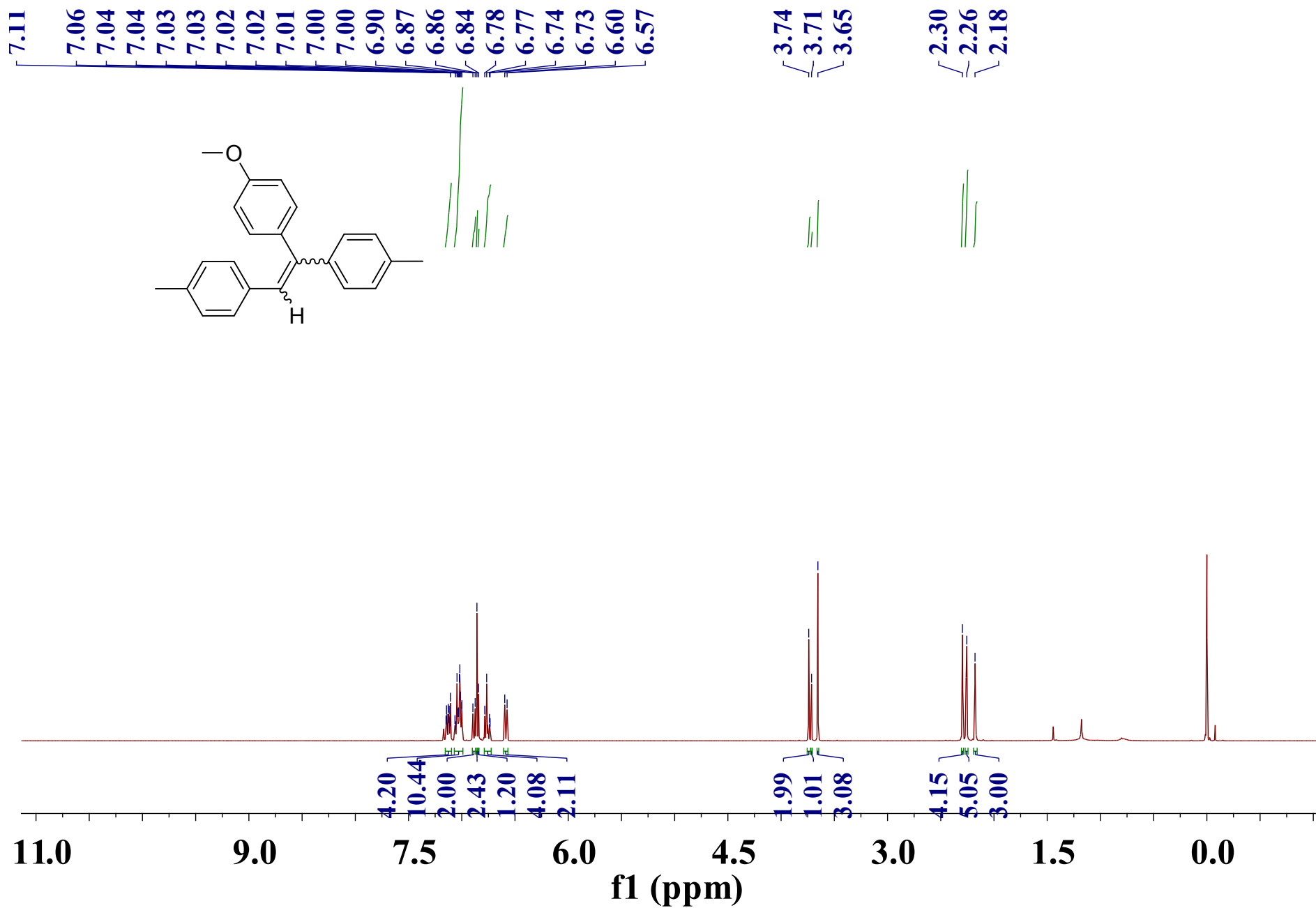
4k ¹³C NMR spectrum



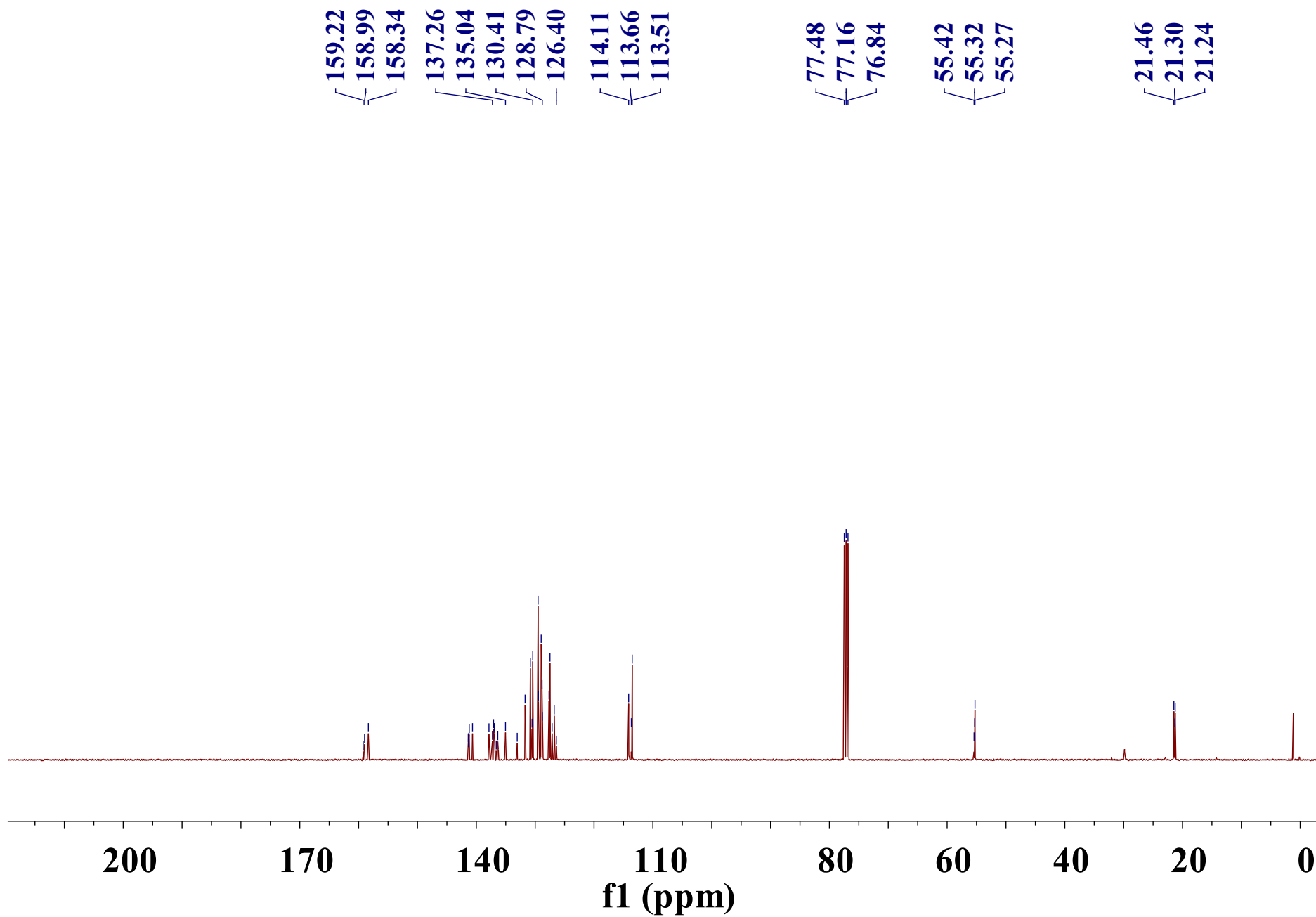
4k ¹³C NMR spectrum



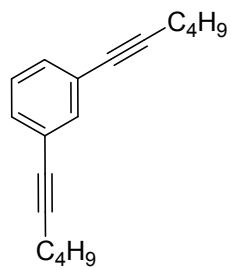
41 ¹H NMR spectrum



4l ¹³C NMR spectrum



7 ¹³C NMR spectrum



7.43
7.28
7.26
7.19
7.18
7.16

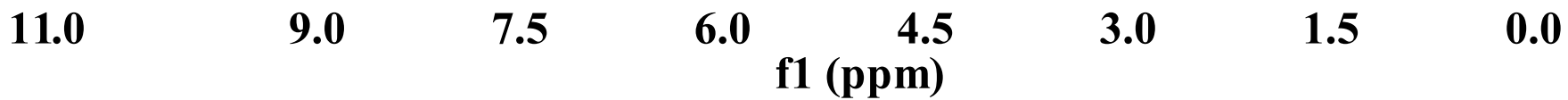
2.41
2.39
2.38
1.54
1.45
0.96
0.94
0.93

0.93
2.09
1.07

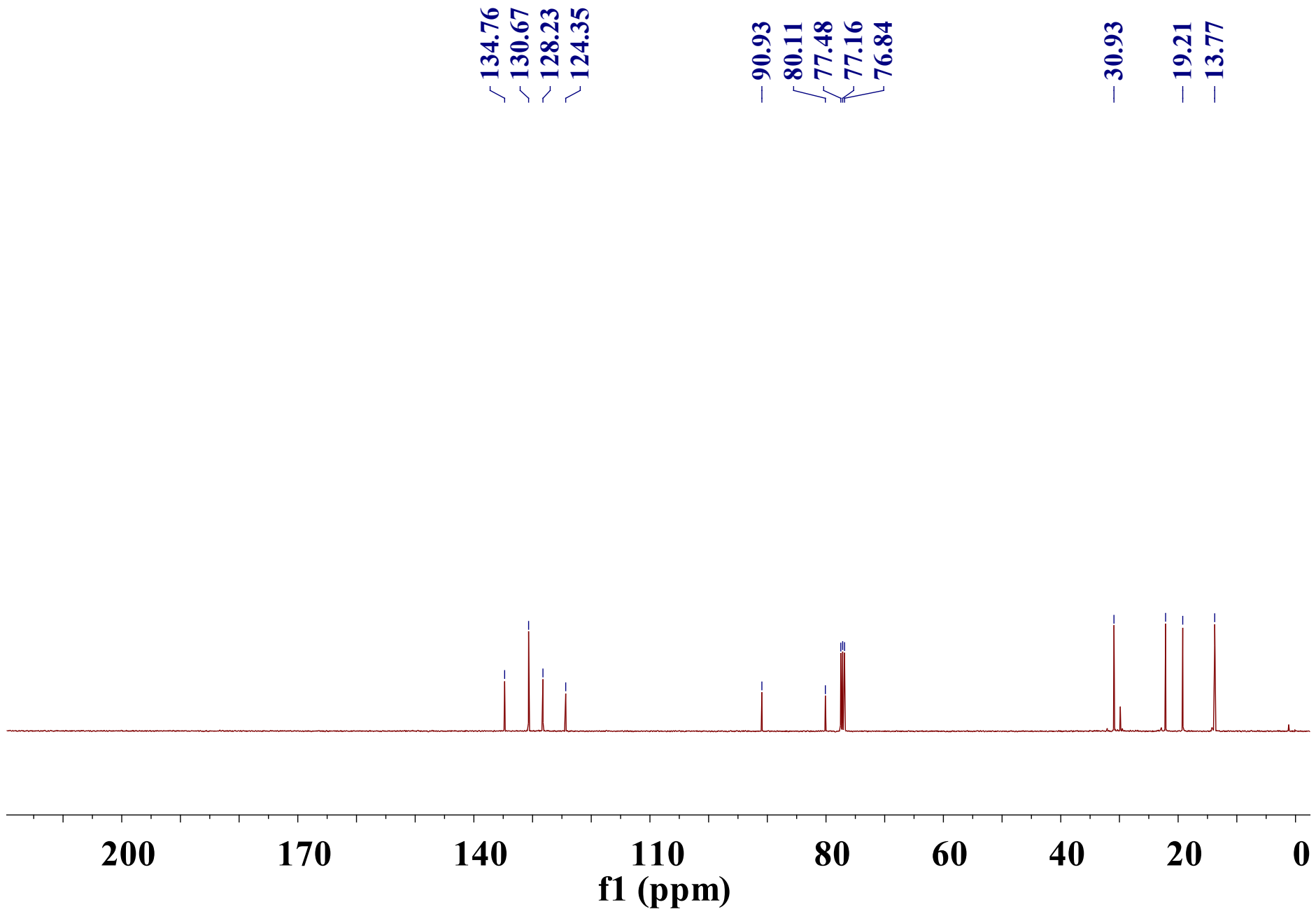
4.00

4.31
4.21

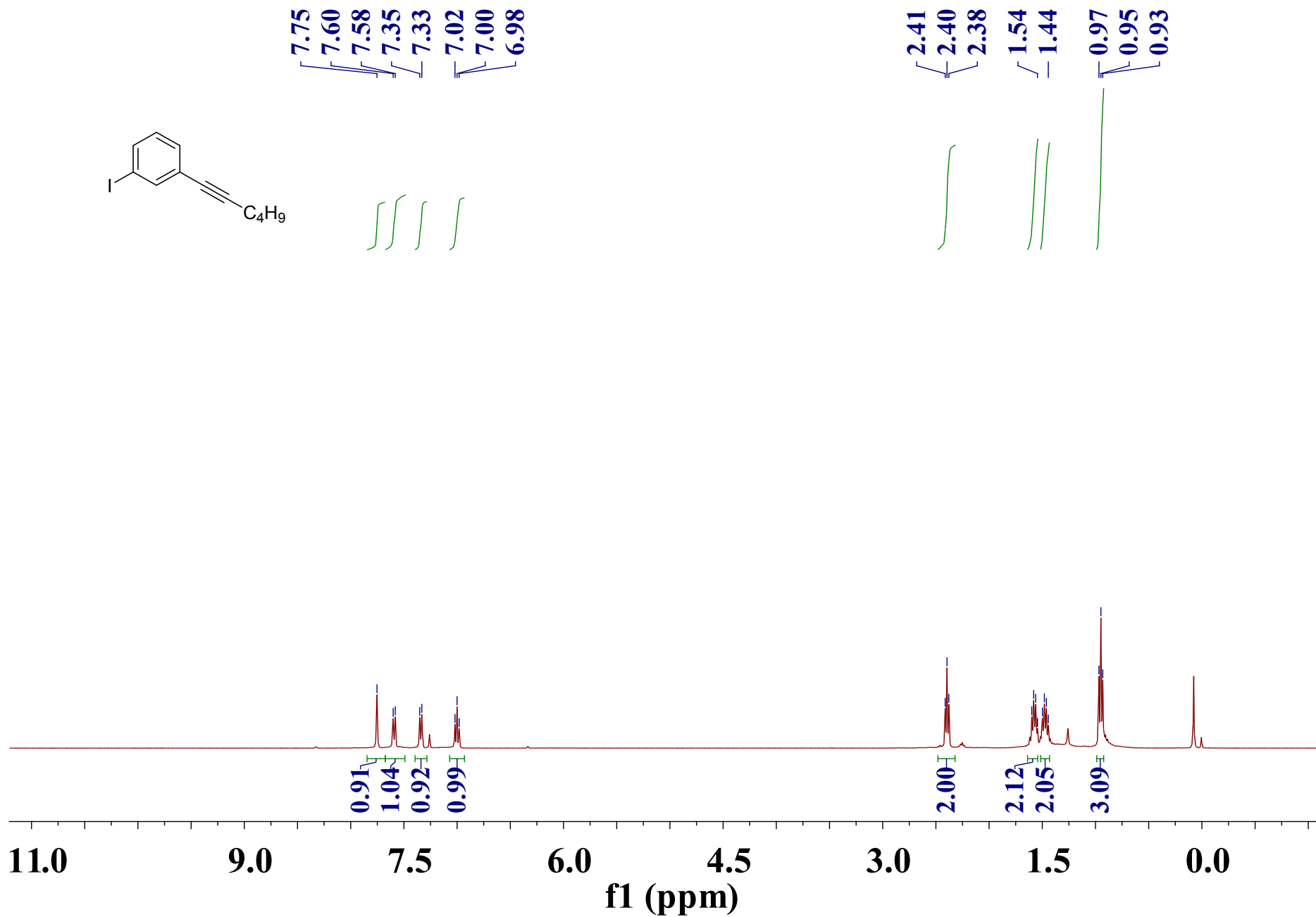
6.04



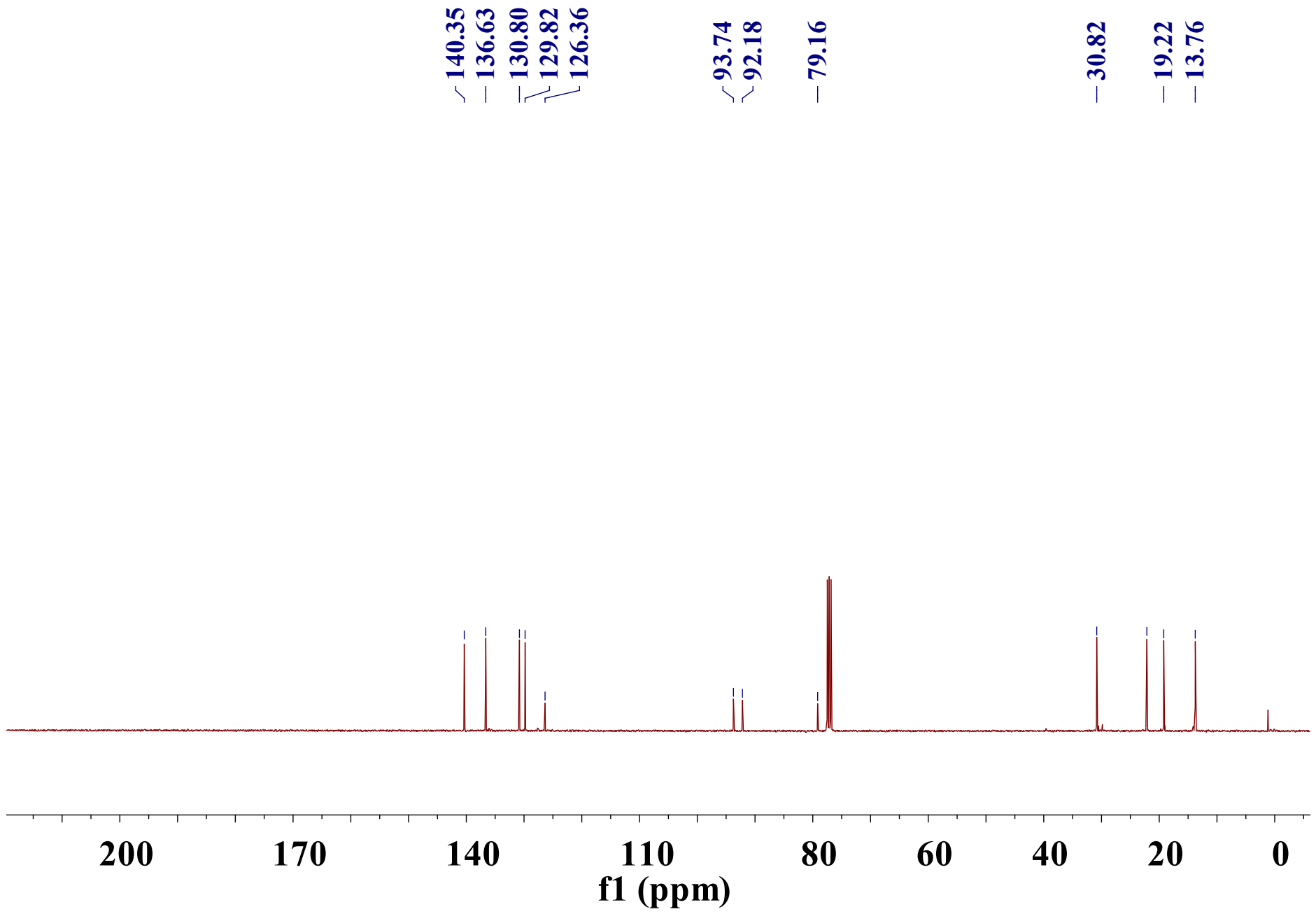
7 ¹³C NMR spectrum



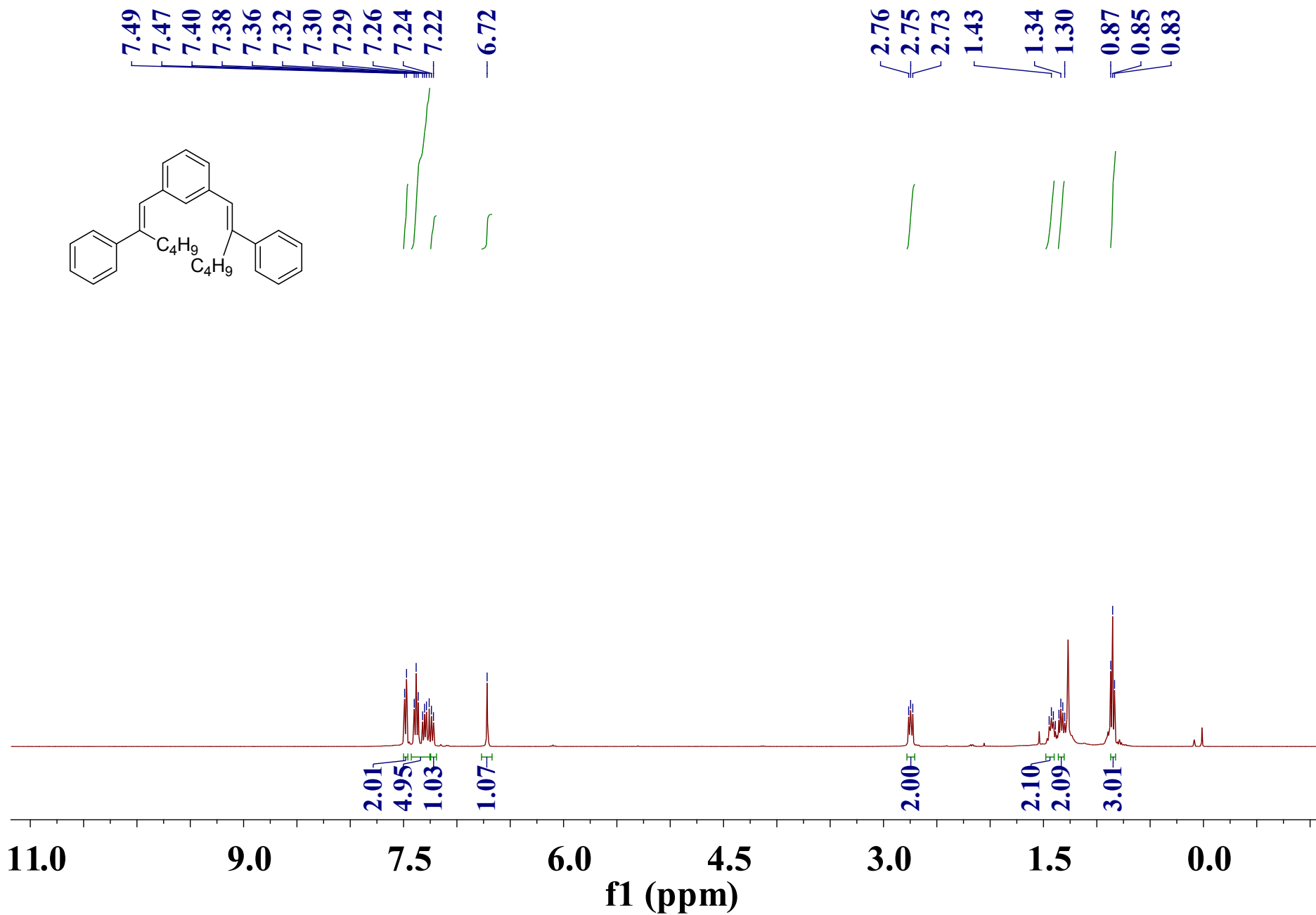
8 ¹H NMR spectrum



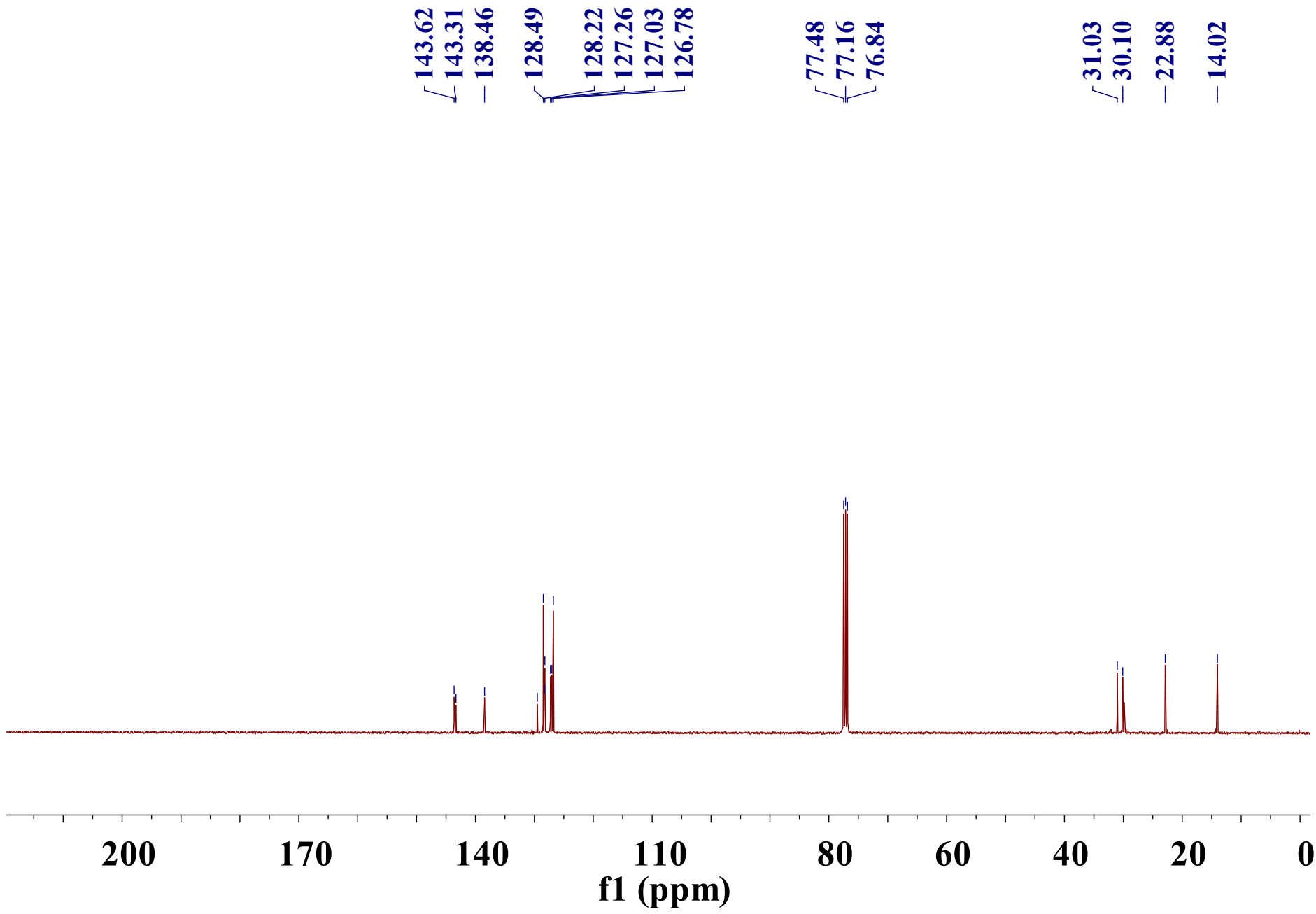
8 ¹³C NMR spectrum



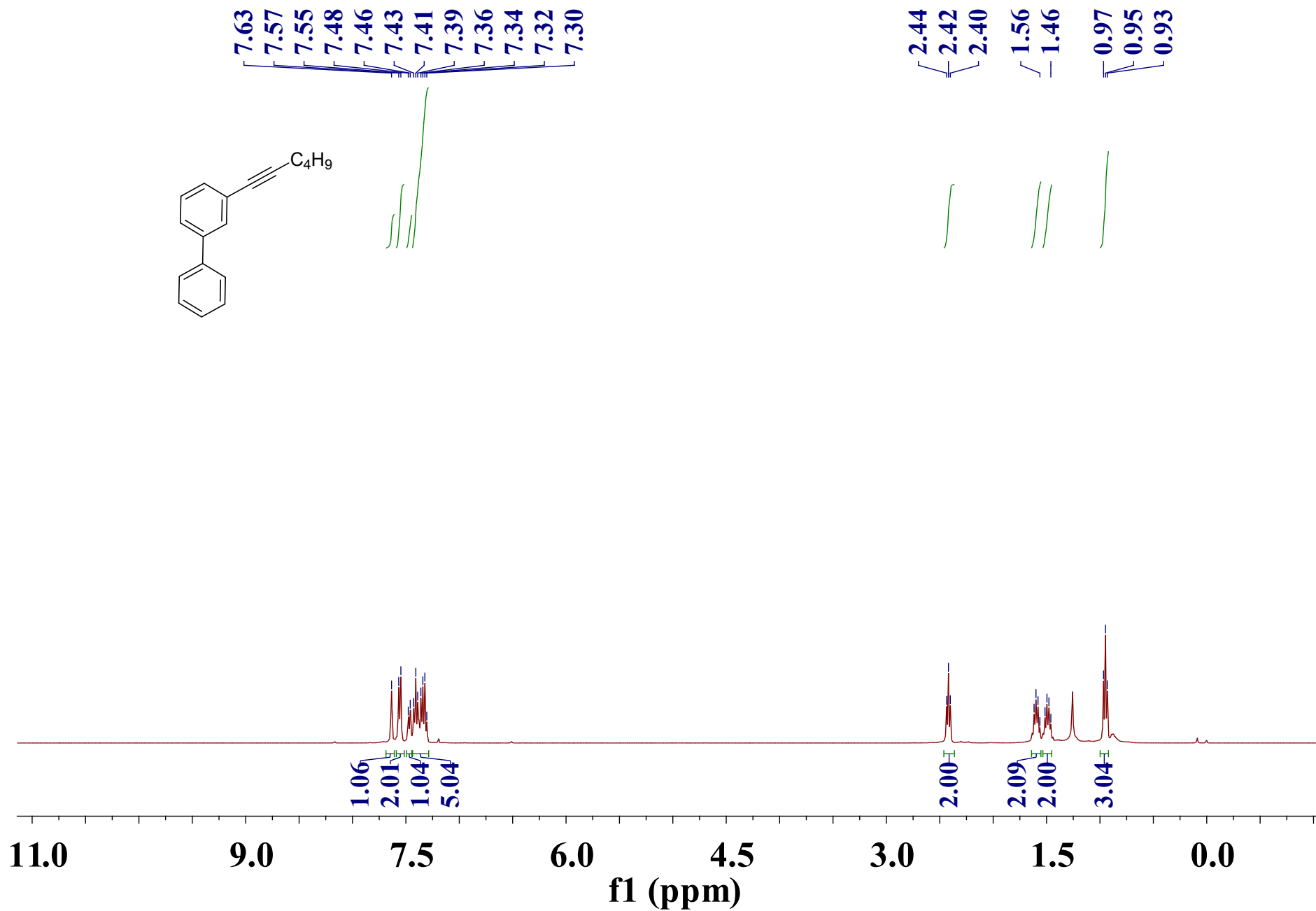
9 ¹H NMR spectrum



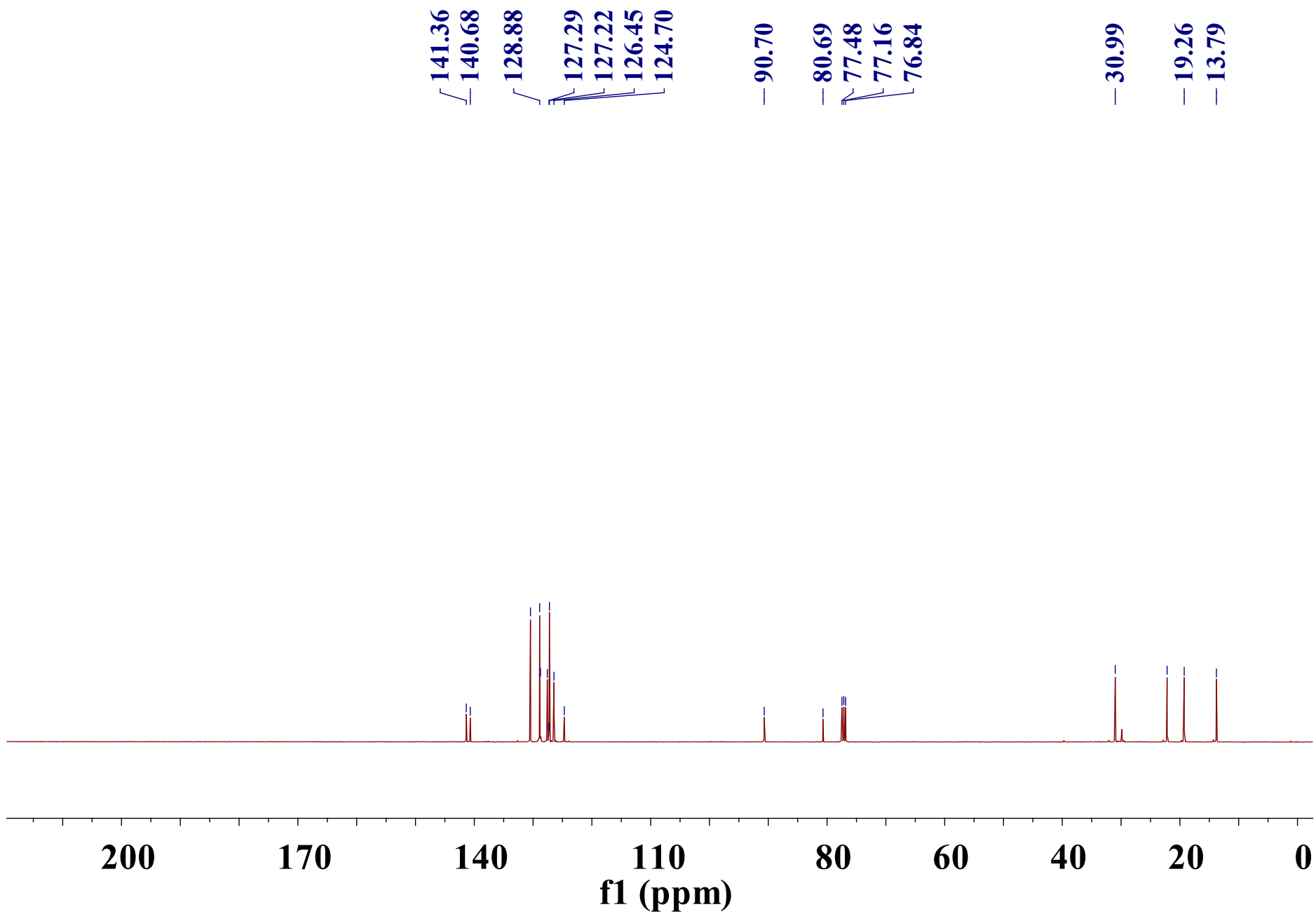
9 ¹³C NMR spectrum



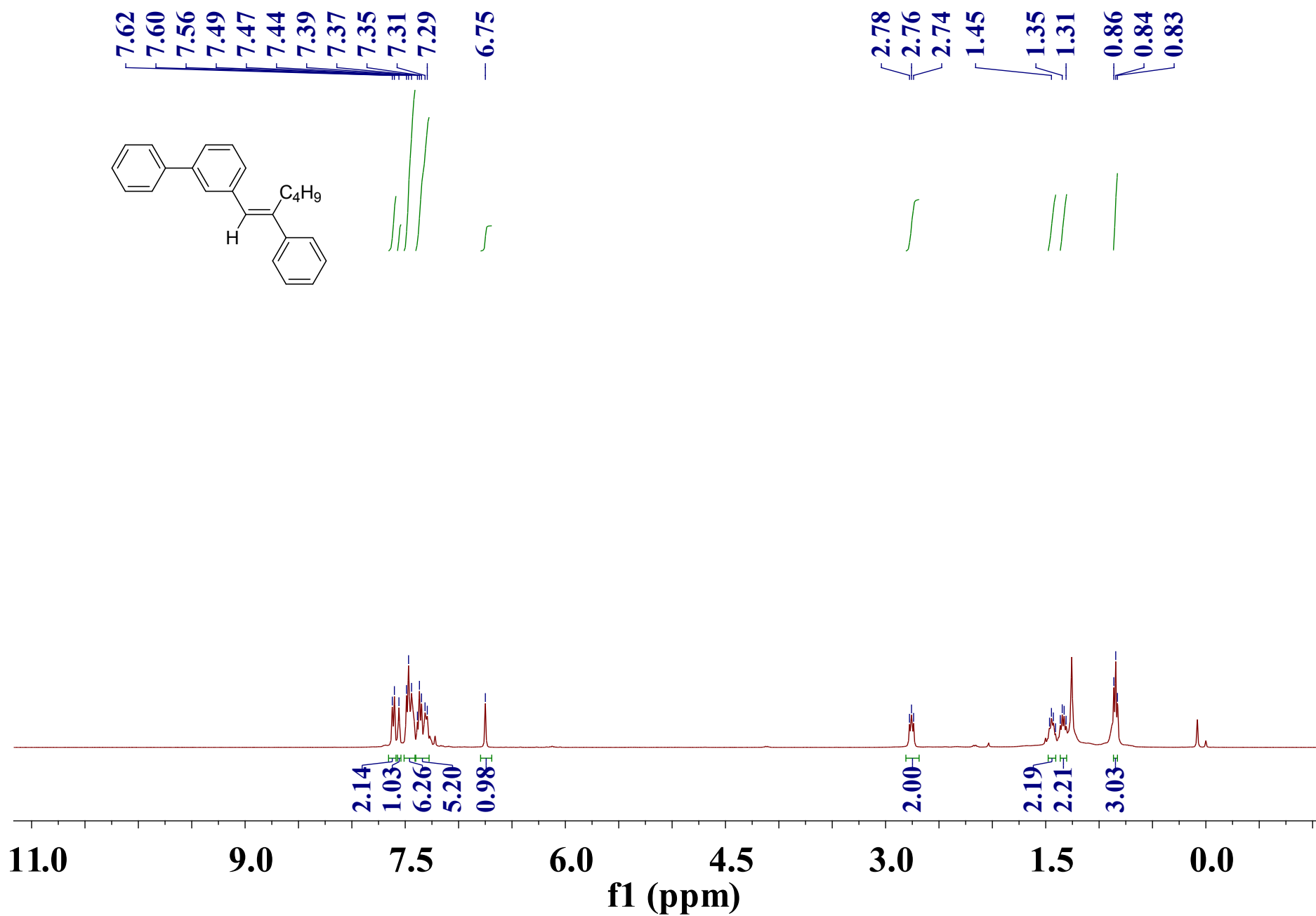
11 ^{11}B NMR spectrum



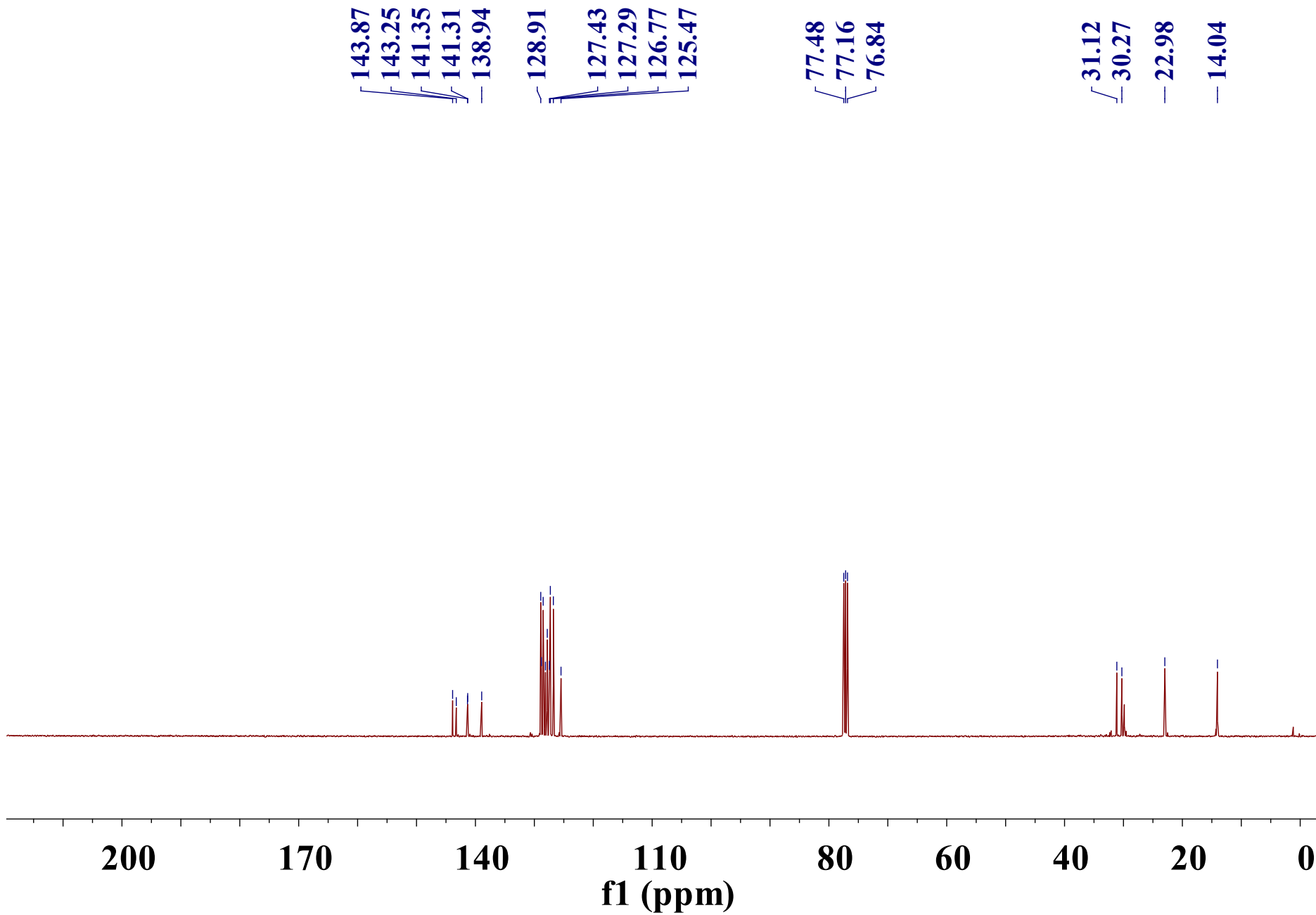
11 ¹³C NMR spectrum



12 ^{13}C NMR spectrum



12 ¹³C NMR spectrum



13 ¹H NMR spectrum

