Supplementary Materials for

Solvent-free and Catalyst-free Direct Alkylation of Alkenes

Meng-Yao Li,[a†] Jiatong Li,[a†] Ao Gu,[a] Xiao-Mei Nong,[a] Shuyang Zhai,[a] Zhu-Ying Yue,[a] Chen-Guo Feng,[b*] Yingbin Liu,[a*] and Guo-Qiang Lin[b]

Corresponding authors:

Chen-Guo Feng, fengcg@shutcm.edu.cn;

Yingbin Liu, laoniulyb@shsmu.edu.cn

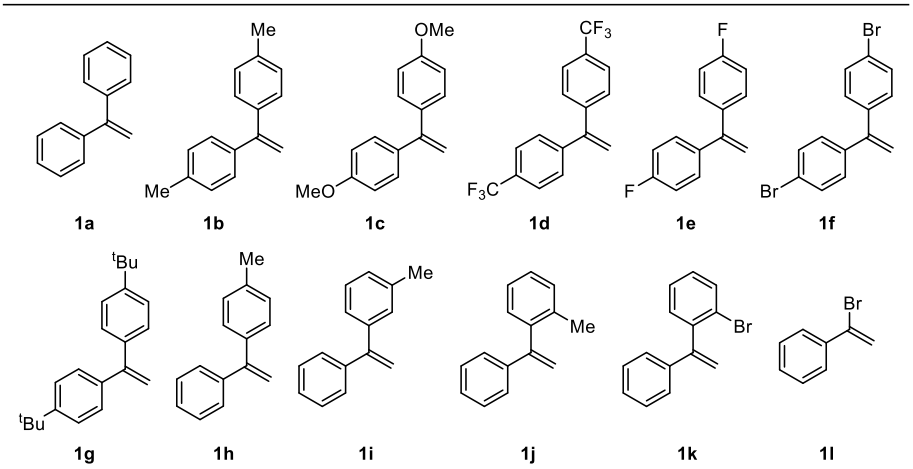
† These authors contributed equally to this work.

Table of Contents

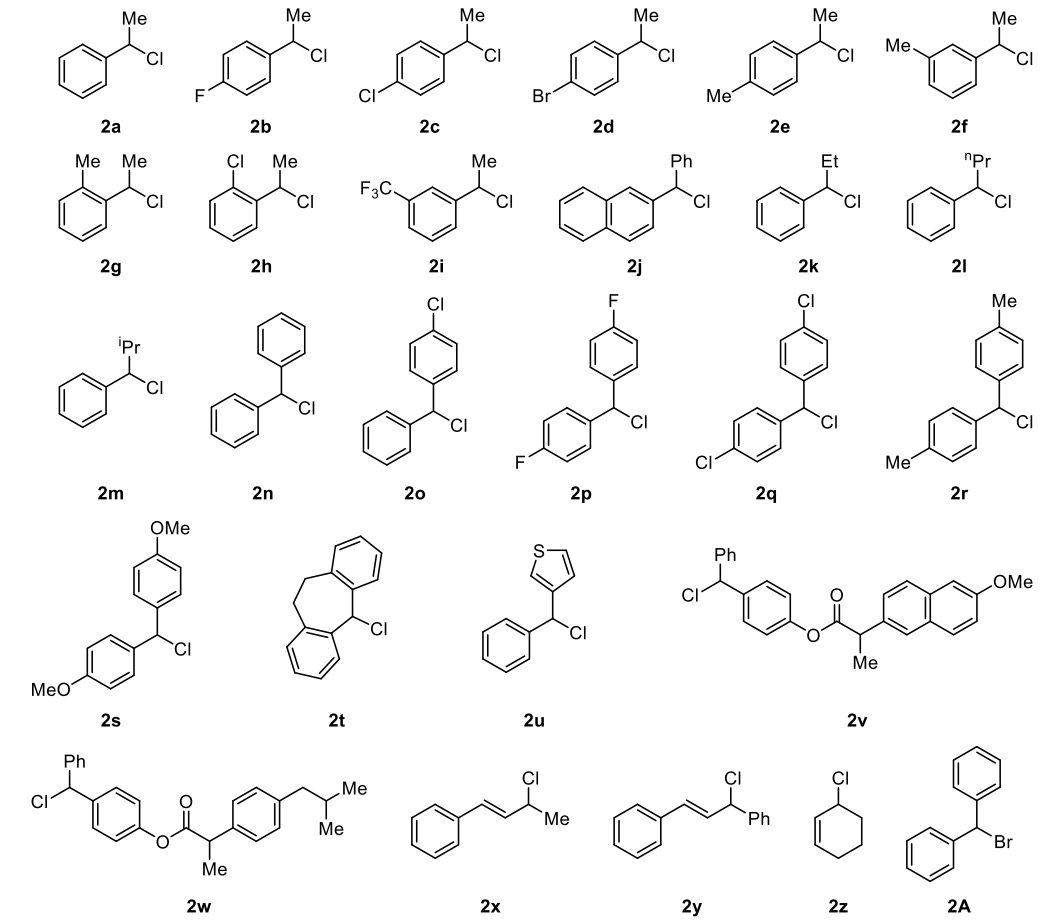
S1. General Information

All the solvents were dried using standard procedure and distilled before use. All commercially available chemical resources were used as received unless otherwise stated. Reactions were monitored by thin layer chromatography (TLC) supplied by Yantai Jiangyou Silicon Material Company (China). Visualization was accomplished with UV light or basic aqueous phosphomolybdic acid (PMA). Chromatography was achieved using forced flow (flash chromatography) of the indicated solvent system on 300-400 mesh silica gel unless otherwise noted. Nuclear Magnetic Resonance (**NMR**) spectra were acquired on Agilent 400 or Bruker 400 instruments operating at 400, 100, and 376 MHz for ^{1}H, ^{13}C, and ^{19}F, respectively. Chemical shifts are reported in δ ppm referenced to an internal $SiMe_4$ standard (TMS: δ 0.000 ppm) for **^{1}H NMR**, chloroform-*d* (δ 77.16) for **^{13}C NMR** unless otherwise noted. Multiplicities are reported using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, quintet = quint, heptet = hept, m = multiplet, br = broad resonance. High-resolution mass spectra (**HRMS**) and Low-resolution mass spectrometry (**LRMS**) were acquired through the National Center for Organic Mass Spectrometry in Shanghai, Shanghai Institute of Organic Chemistry (CAS) and determined on a Waters Micromass GCT Premie spectrometer. The alkene substrates **1a**, **1l** and the halide substrates **2a-2f**, **2h**, **2k**, **2m-2q**, **2t**, **2z**, **2A** were commercially available. The alkene substrates[1-5] **1b-1k** and the chloride substrates[6-7] **2g**, **2i**, **2j**, **2l**, **2u-2y** were prepared according to the literatures.

S2. Substrates list

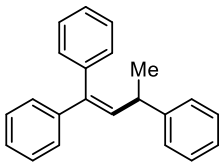


Scheme S1. Alkene substrates list.



Scheme S2. Halide substrates list.

S3. General procedures for alkylation of alkenes



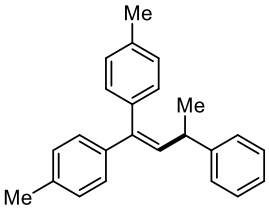
3aa

but-1-ene-1,1,3-triyltribenzene (3aa), white solid (90% yield), R_f = 0.34 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2a** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 80 °C and the stir was continued for 3 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.10 (m, 13H), 6.20 (d, *J* = 10.3 Hz, 1H), 3.59 (dq, *J* = 10.3, 7.0 Hz, 1H), 1.37 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 146.33, 142.51, 140.27, 140.22, 134.30, 129.96, 128.63, 128.40, 128.22, 127.40, 127.20, 127.13, 127.08, 126.14, 39.43, 22.49 ppm.

EI-MS m/z (%): 284 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{22}H_{20}$ $[M]^+$: 284.1560, found: 284.1564.



3ab

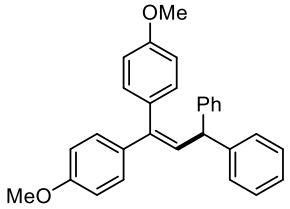
4,4'-(3-phenylbut-1-ene-1,1-diyl)bis(methylbenzene) (3ab), colorless oil (85% yield), R_f = 0.32 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1b** (0.6 mmol) and **2a** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 80 °C and the stir was continued

for 3 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.24 (m, 2H), 7.24-7.14 (m, 5H), 7.13-7.02 (m, 6H), 6.14 (d, *J* = 10.3 Hz, 1H), 3.60 (dq, *J* = 10.3, 6.9 Hz, 1H), 2.38 (s, 3H), 2.30 (s, 3H), 1.37 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 146.59, 140.03, 139.99, 137.40, 136.80, 136.70, 133.31, 129.84, 129.03, 128.89, 128.57, 127.33, 127.10, 126.04, 39.39, 22.59, 21.40, 21.18 ppm.

EI-MS m/z (%): 374 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{29}H_{26}$ $[M]^+$: 374.2020, found: 374.2031.



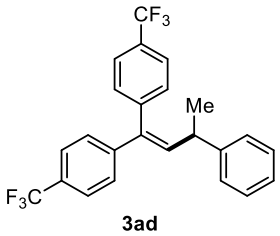
3ac

4,4'-(3,3-diphenylprop-1-ene-1,1-diyl)bis(methoxybenzene) (3ac), colorless oil (94% yield), R_f = 0.30 (silica, petroleum ether/ethyl acetate = 50:1 ***v/v***, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1c** (0.6 mmol) and **2n** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.24 (m, 4H), 7.22-7.14 (m, 8H), 7.12-7.04 (m, 2H), 6.93-6.85 (m, 2H), 6.83-6.76 (m, 2H), 6.40 (d, *J* = 10.5 Hz, 1H), 4.82 (d, *J* = 10.5 Hz, 1H), 3.83 (s, 3H), 3.78 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.07, 158.87, 144.98, 140.77, 135.46, 132.32, 131.06, 129.31, 128.80, 128.65, 128.60, 128.50, 127.72, 126.67, 126.29, 113.74, 113.58, 77.48, 77.16, 76.84, 55.43, 55.37, 50.71 ppm.

EI-MS m/z (%): 406 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{29}H_{26}O_2$ $[M]^+$: 406.1927, found: 406.1937.



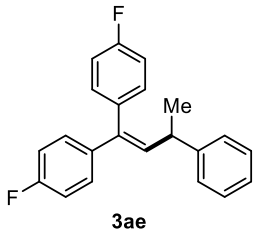
3ad

4,4'-(3-phenylbut-1-ene-1,1-diyl)bis((trifluoromethyl)benzene) (3ad), white solid (69% yield), R_f = 0.33 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1d** (0.6 mmol) and **2a** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 120 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.2 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.37-7.25 (m, 6H), 7.25-7.20 (m, 1H), 7.20-7.14 (m, 2H), 6.36 (d, *J* = 10.5 Hz, 1H), 3.54 (dq, *J* = 10.3, 6.7 Hz, 1H), 1.41 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.38, 145.14, 143.18, 138.15, 137.33, 130.28, 129.92 (q, *J* = 32.3 Hz), 129.48 (q, *J* = 32.2 Hz), 128.89, 127.60, 126.95, 126.57, 125.67 (q, *J* = 3.8 Hz), 125.37 (q, *J* = 3.8 Hz), 124.28 (q, *J* = 270.7 Hz), 39.68, 22.48 ppm; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.54, -62.55 ppm.

EI-MS m/z (%): 420 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{24}H_{18}F_6$ $[M]^+$: 420.1307, found: 420.1313.



3ae

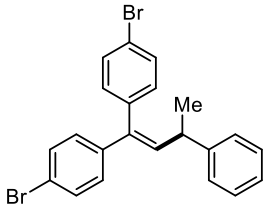
4,4'-(3-phenylbut-1-ene-1,1-diyl)bis(fluorobenzene) (3ae), white solid (74% yield), R_f = 0.33 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1e** (0.6 mmol) and **2a** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature.

Then the temperature was increased to 120 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.26 (m, 2H), 7.23-7.02 (m, 9H), 6.99-6.89 (m, 2H), 6.15 (d, *J* = 10.3 Hz, 1H), 3.55 (dq, *J* = 10.3, 6.9 Hz, 1H), 1.38 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.28 (d, *J* = 245.2 Hz), 162.19 (d, *J* = 245.2 Hz), 146.12, 138.50 (d, *J* = 3.1 Hz), 138.39, 135.87 (d, *J* = 3.4 Hz), 134.52 (d, *J* = 1.5 Hz), 131.47 (d, *J* = 8.0 Hz), 128.93 (d, *J* = 8.0 Hz), 128.73, 126.99, 126.29, 115.47 (d, *J* = 21.0 Hz), 115.10 (d, *J* = 21.4 Hz), 39.55, 22.61 ppm; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -114.99, -115.56 ppm.

EI-MS m/z (%): 320 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{22}H_{18}F_2$ $[M]^+$: 320.1371, found: 320.1365.



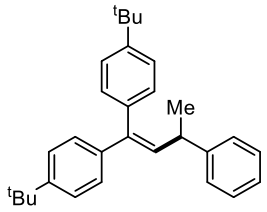
3af

4,4'-(3-phenylbut-1-ene-1,1-diyl)bis(bromobenzene) (3af), white solid (66% yield), R_f = 0.31 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1f** (0.6 mmol) and **2a** (1.2 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57-7.46 (m, 2H), 7.42-7.32 (m, 2H), 7.32-7.26 (m, 2H), 7.24-7.11 (m, 3H), 7.09-6.96 (m, 4H), 6.21 (d, *J* = 10.4 Hz, 1H), 3.54 (dq, *J* = 10.3, 6.9 Hz, 1H), 1.37 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.75, 140.89, 138.51, 138.22, 135.29, 131.75, 131.58, 131.39, 128.98, 128.77, 126.95, 126.37, 121.61, 121.40, 39.59, 22.53 ppm.

EI-MS m/z (%): 440 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{22}H_{18}Br_2$ $[M]^+$: 439.9770, found: 439.9773.



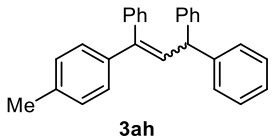
3ag

4,4'-(3-phenylbut-1-ene-1,1-diyl)bis(tert-butylbenzene) (3ag), white solid (53% yield), R_f = 0.27 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1g** (0.6 mmol) and **2a** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, J = 8.3 Hz, 2H), 7.32-7.20 (m, 6H), 7.20-7.14 (m, 3H), 7.14-7.07 (m, 2H), 6.16 (d, J = 10.4 Hz, 1H), 3.63 (dq, J = 10.3, 6.9 Hz, 1H), 1.38 (d, J = 6.9 Hz, 3H), 1.36 (s, 9H), 1.29 (s, 9H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 150.00, 149.83, 146.56, 139.92, 139.84, 137.17, 133.49, 129.53, 128.53, 127.14, 127.01, 126.00, 125.15, 125.08, 39.20, 34.70, 34.57, 31.57, 31.46, 22.47 ppm.

EI-MS m/z (%): 396 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{30}H_{36}$ $[M]^+$: 396.2812, found: 396.2818.



3ah

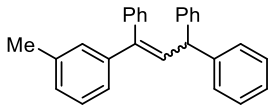
(3-(*p*-tolyl)prop-2-ene-1,1,3-triyl)tribenzene (3ah), colorless oil (97% yield), R_f = 0.23 (silica, petroleum ether, UV and $KMnO_4$ detection);

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1h** (0.6 mmol) and **2a** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and the stir was continued

for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure products.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.23 (m, 18.6H), 7.22-7.18 (m, 5.6H), 7.18-7.12 (m, 16.6H), 7.08-7.06 (m, 2.8H),7.06-7.03 (m, 2H) 6.51 (d, *J* = 10.7 Hz, 1H), 6.50 (d, *J* = 10.5 Hz, 1.4H), 4.84 (d, *J* = 10.5 Hz, 1.4H), 4.80 (d, *J* = 10.7 Hz, 1H), 2.38 (s, 4.2H), 2.31 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.76, 142.63, 141.69, 141.59, 139.99, 139.58, 137.19, 137.05, 136.81, 130.97, 130.31, 129.91, 129.83, 129.12, 128.97, 128.61, 128.52, 128.50, 128.39, 128.22, 127.66, 127.50, 127.35, 127.31, 126.34, 50.67, 50.63, 21.41, 21.21 ppm.

EI-MS m/z (%): 360 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{28}H_{24}$ $[M]^+$: 360.1873, found: 360.1874.



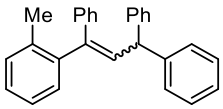
3ai

(3-(*m*-tolyl)prop-2-ene-1,1,3-triyl)tribenzene (3ai), colorless oil (83% yield), R_f = 0.23 (silica, petroleum ether, UV and $KMnO_4$ detection);

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1i** (0.6 mmol) and **2a** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure products.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.13 (m, 32H), 7.13-7.07 (m, 2H), 7.03 (dd, *J* = 7.3, 1.8 Hz, 2H), 7.00-6.94 (m, 2H), 6.54 (d, *J* = 2.1 Hz, 1H), 6.51 (d, *J* = 2.3 Hz, 1H), 4.82 (d, *J* = 3.8 Hz, 1H), 4.80 (d, *J* = 3.9 Hz, 1H), 2.30 (s, 3H), 2.28 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.76, 144.70, 142.47, 142.36, 141.91, 141.84, 139.92, 139.70, 137.93, 137.82, 131.05, 131.00, 130.62, 129.91, 128.62, 128.59, 128.55, 128.51, 128.47, 128.39, 128.24, 128.19, 128.16, 127.63, 127.58, 127.56, 127.39, 127.37, 127.33, 127.00, 126.35, 124.91, 50.66, 50.64, 21.57 ppm.

EI-MS m/z (%): 360 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{28}H_{24}$ $[M]^+$: 360.1873, found: 360.1880.



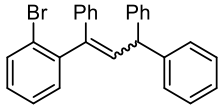
3aj

(3-(*o*-tolyl)prop-2-ene-1,1,3-triyl)tribenzene (3aj), colorless oil (60% yield), R_f = 0.23 (silica, petroleum ether, UV and $KMnO_4$ detection);

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1j** (0.6 mmol) and **2a** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure products.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.17 (m, 14+3.2H), 7.17-7.12 (m, 2+0.6H), 7.12-7.06 (m, 3H), 6.69 (d, *J* = 10.4 Hz, 1H), 6.16 (d, *J* = 10.7 Hz, 0.2H), 5.11 (d, *J* = 10.4 Hz, 0.2H), 4.58 (d, *J* = 10.4 Hz, 1H), 1.99 (s, 0.5H), 1.92 (s, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.85, 144.65, 144.40, 143.54, 141.83, 141.18, 140.88, 139.89, 138.83, 137.02, 136.39, 133.35, 131.04, 130.44, 130.42, 130.30, 130.20, 129.33, 128.70, 128.57, 128.56, 128.49, 128.48, 128.45, 128.22, 127.70, 127.45, 127.34, 127.20, 126.58, 126.41, 126.35, 126.33, 125.82, 125.70, 50.75, 50.29, 20.64, 19.84 ppm.

EI-MS m/z (%): 360 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{28}H_{24}$ $[M]^+$: 360.1873, found: 360.1883.



3ak

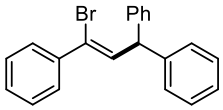
(3-(2-bromophenyl)prop-2-ene-1,1,3-triyl)tribenzene (3ak), white solid (65% yield), R_f = 0.21 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1k** (0.6 mmol) and **2a** (1.2 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature.

Then the temperature was increased to 100 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure products.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.9 Hz, 1H), 7.44-7.01 (m, 18H), 6.69 (d, *J* = 10.4 Hz, 1H), 6.20 (d, *J* = 10.7 Hz, 0.16H), 5.10 (d, *J* = 10.7 Hz, 0.16H), 4.56 (d, *J* = 10.4 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.06, 142.34, 140.92, 140.17, 140.03, 133.30, 132.21, 132.01, 129.53, 129.25, 128.68, 128.65, 128.62, 128.60, 128.52, 128.48, 128.45, 128.16, 127.57, 127.51, 127.40, 127.33, 126.71, 126.48, 126.40, 124.37, 50.96 ppm.

DART-MS m/z (%): 425 $(M+H)^+$; **HRMS** (DART): m/z Exact mass calcd for $C_{27}H_{22}Br$ $[M+H]^+$: 425.0905, found: 425.0910.



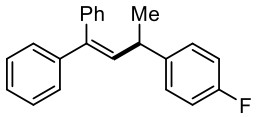
3al

(*Z*)-(3-bromoprop-2-ene-1,1,3-triyl)tribenzene (3al), white solid (66% yield), R_f = 0.27 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1l** (0.6 mmol) and **2A** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73-7.50 (m, 2H), 7.37-7.28 (m, 7H), 7.28-7.21 (m, 6H), 6.67 (d, *J* = 9.4 Hz, 1H), 5.36 (d, *J* = 9.4 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 142.70, 139.67, 133.27, 128.67, 128.61, 128.32, 128.25, 127.77, 126.63, 126.18, 53.65 ppm.

EI-MS m/z (%): 348 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{21}H_{17}Br$ $[M]^+$: 348.0508, found: 348.0517.



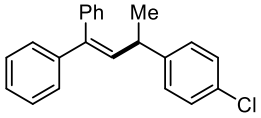
3ba

(3-(4-fluorophenyl)but-1-ene-1,1-diyl)dibenzene (3ba), white solid (72% yield), R_f = 0.34 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2b** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 120 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.29 (m, 3H), 7.29-7.19 (m, 5H), 7.19-7.08 (m, 4H), 7.01-6.89 (m, 2H), 6.16 (d, J = 10.3 Hz, 1H), 3.58 (dq, J = 10.3, 6.8 Hz, 1H), 1.36 (d, J = 7.0 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 161.37 (d, J = 244.1 Hz), 142.35, 142.00 (d, J = 3.1 Hz), 140.50, 140.11, 133.99, 129.86, 128.45, 128.37, 128.26, 127.38, 127.28, 127.24, 115.31 (d, J = 21.0 Hz), 38.71, 22.58 ppm; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -117.46 ppm.

EI-MS m/z (%): 302 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{22}H_{19}F$ $[M]^+$: 302.1465, found: 302.1473.



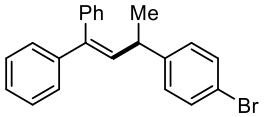
3bb

(3-(4-chlorophenyl)but-1-ene-1,1-diyl)dibenzene (3bb), white solid (82% yield), R_f = 0.33 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2c** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.29 (m, 3H), 7.28-7.18 (m, 7H), 7.18-7.07 (m, 4H), 6.15 (d, *J* = 10.3 Hz, 1H), 3.56 (dq, *J* = 10.2, 6.9 Hz, 1H), 1.36 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.86, 142.28, 140.81, 140.05, 133.57, 131.77, 129.84, 128.70, 128.46, 128.44, 128.27, 127.39, 127.33, 127.29, 38.90, 22.47 ppm.

EI-MS m/z (%): 318 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{22}H_{19}Cl$ $[M]^+$: 318.1170, found: 318.1174.



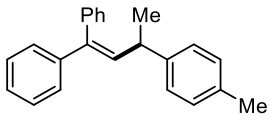
3bc

(3-(4-bromophenyl)but-1-ene-1,1-diyl)dibenzene (3bc), white solid (60% yield), R_f = 0.31 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2d** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.30 (m, 4H), 7.30-7.18 (m, 6H), 7.18-7.13 (m, 2H), 7.12-7.00 (m, 2H), 6.14 (d, *J* = 10.3 Hz, 1H), 3.55 (dq, *J* = 10.3, 7.0 Hz, 1H), 1.36 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.40, 142.26, 140.87, 140.04, 133.46, 131.65, 129.83, 128.86, 128.46, 128.27, 127.39, 127.33, 127.30, 119.84, 38.97, 22.42 ppm.

EI-MS m/z (%): 362 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{22}H_{19}Br$ $[M]^+$: 362.0665, found: 362.0661.



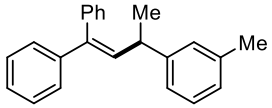
3bd

(3-(*p*-tolyl)but-1-ene-1,1-diyl)dibenzene (3bd), white solid (98% yield), R_f = 0.31 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2e** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 80 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.28 (m, 3H), 7.27-7.14 (m, 7H), 7.10 (s, 4H), 6.20 (d, *J* = 10.4 Hz, 1H), 3.56 (dq, *J* = 10.4, 6.9 Hz, 1H), 2.31 (s, 3H), 1.36 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.17, 142.42, 140.12, 139.89, 135.47, 134.37, 129.83, 129.17, 128.23, 128.05, 127.26, 127.02, 126.94, 126.79, 38.87, 22.44, 21.00 ppm.

EI-MS m/z (%): 298 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{23}H_{22}$ $[M]^+$: 298.1716, found: 298.1717.



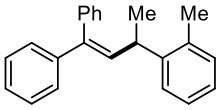
3be

(3-(*m*-tolyl)but-1-ene-1,1-diyl)dibenzene (3be), colorless oil (40% yield), R_f = 0.30 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2f** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and the stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.29 (m, 3H), 7.28-7.11 (m, 8H), 7.04-6.96 (m, 3H), 6.22 (d, *J* = 10.4 Hz, 1H), 3.56 (dq, *J* = 13.8, 6.9 Hz, 1H), 2.33 (s, 3H), 1.37 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 146.30, 142.56, 140.24, 140.13, 138.16, 134.40, 129.99, 128.51, 128.37, 128.21, 127.80, 127.41, 127.17, 127.10, 126.90, 124.14, 39.38, 22.61, 21.68 ppm.

EI-MS m/z (%): 298 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{23}H_{22}$ $[M]^+$: 298.1716, found: 298.1713.



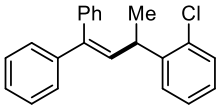
3bf

(3-(*o*-tolyl)but-1-ene-1,1-diyl)dibenzene (3bf), colorless oil (94% yield), $\boldsymbol{R_f}$ = 0.31 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2g** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 80 °C and stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.29 (m, 4H), 7.27-7.16 (m, 6H), 7.16-7.11 (m, 2H), 7.10-7.05 (m, 2H), 6.24 (d, J = 10.0 Hz, 1H), 3.75 (dq, J = 10.0, 6.9 Hz, 1H), 1.96 (s, 3H), 1.37 (d, J = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.12, 142.60, 140.47, 140.29, 135.38, 134.28, 130.47, 129.87, 128.31, 128.20, 127.47, 127.21, 127.12, 126.40, 126.08, 125.87, 35.48, 22.83, 19.35 ppm.

EI-MS m/z (%): 298 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{23}H_{22}$ $[M]^+$: 298.1716, found: 298.1713.



3bg

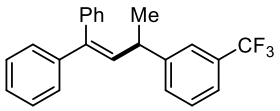
(3-(2-chlorophenyl)but-1-ene-1,1-diyl)dibenzene (3bg), colorless oil (40% yield), $\boldsymbol{R_f}$ = 0.28 (silica, petroleum ether, UV and $KMnO_4$ detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2h** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for

5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.18 (m, 11H), 7.14-7.06 (m, 3H), 6.26 (d, *J* = 9.9 Hz, 1H), 4.05 (dq, *J* = 9.8, 6.8 Hz, 1H), 1.36 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.27, 142.61, 141.72, 139.91, 133.27, 132.69, 129.85, 129.83, 128.26, 128.22, 128.17, 127.56, 127.26, 127.25, 127.23, 127.15, 36.41, 22.54 ppm.

EI-MS m/z (%): 318 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{22}H_{19}Cl$ $[M]^+$: 318.1170, found: 318.1166.



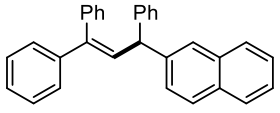
3bh

(3-(3-(trifluoromethyl)phenyl)but-1-ene-1,1-diyl)dibenzene (3bh), colorless oil (25% yield), R_f = 0.34 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2i** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 120 °C and stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford a pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.30 (m, 7H), 7.29-7.19 (qd, *J* = 7.2, 6.1, 2.8 Hz, 5H), 7.20-7.11 (m, 2H), 6.18 (d, *J* = 10.2 Hz, 1H), 3.65 (dq, *J* = 10.3, 6.9 Hz, 1H), 1.41 (d, *J* = 6.9 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 147.30, 142.11, 141.33, 139.96, 133.08, 130.86 (d, *J* = 31.8 Hz), 130.61 (q, *J* = 1.1 Hz), 129.79, 129.02, 128.53, 128.30, 127.42, 127.40, 127.38, 124.40 (q, *J* = 271.4 Hz), 123.75 (q, *J* = 3.8 Hz), 123.04 (q, *J* = 3.8 Hz), 39.37, 22.38 ppm; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -62.52 ppm.

EI-MS m/z (%): 352 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{23}H_{19}F_3$ $[M]^+$: 352.1443, found: 352.1431.



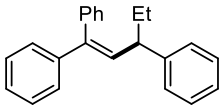
3bi

2-(1,3,3-triphenylallyl)naphthalene (3bi), colorless oil (89% yield), R_f = 0.24 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2j** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 3 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.69 (m, 3H), 7.64 (s, 1H), 7.48-7.40 (m, 2H), 7.39-7.33 (m, 3H), 7.30-7.15 (m, 13H), 6.63 (d, J = 10.5 Hz, 1H), 4.98 (d, J = 10.5 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.57, 142.36, 142.10, 141.99, 139.81, 133.69, 132.31, 130.90, 129.93, 128.69, 128.59, 128.48, 128.30, 127.94, 127.74, 127.70, 127.50, 127.45, 127.24, 126.62, 126.48, 126.15, 125.69, 50.88 ppm.

EI-MS m/z (%): 396 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{31}H_{24}$ $[M]^+$: 396.1878, found: 396.1875.



3bj

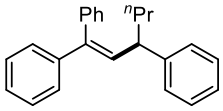
pent-1-ene-1,1,3-triyltribenzene (3bj), white solid (71% yield), R_f = 0.32 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2k** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.31 (m, 3H), 7.31-7.10 (m, 12H), 6.23 (d, J = 10.4 Hz, 1H), 3.30 (dt, J = 10.5, 7.4 Hz, 1H), 1.75 (p, J = 7.2 Hz, 2H), 0.84 (t, J = 7.4 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.48, 142.65, 141.40, 140.35, 133.19,

130.08, 128.62, 128.30, 128.21, 127.53, 127.36, 127.11, 126.11, 47.20, 30.45, 12.32 ppm.

EI-MS m/z (%): 298 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{23}H_{24}$ $[M]^+$: 298.1716, found: 298.1726.



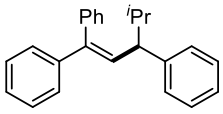
3bk

hex-1-ene-1,1,3-triyltribenzene (3bk), colorless oil (87% yield), R_f = 0.33 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2l** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.09 (m, 15H), 6.23 (d, *J* = 10.4 Hz, 1H), 3.40 (dt, *J* = 10.5, 7.4 Hz, 1H), 1.76-1.61 (m, 2H), 1.39-1.25 (m, 1H), 1.23-1.11 (m, 1H), 0.81 (t, *J* = 7.3 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.69, 142.62, 141.13, 140.30, 133.38, 130.06, 128.63, 128.27, 128.20, 127.48, 127.35, 127.12, 127.09, 126.07, 45.28, 39.89, 20.86, 14.18 ppm.

EI-MS m/z (%): 312 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{24}H_{24}$ $[M]^+$: 312.1873, found: 312.1869.



3bl

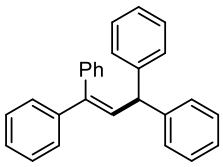
(4-methylpent-1-ene-1,1,3-triyl)tribenzene (3bl), white solid (53% yield), R_f = 0.32 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2m** (1.2 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature.

Then the temperature was increased to 100 °C and stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.31 (m, 3H), 7.31-7.14 (m, 8H), 7.14-7.06 (m, 4H), 6.32 (d, *J* = 10.8 Hz, 1H), 3.02 (dd, *J* = 10.8, 8.9 Hz, 1H), 1.98 (dp, *J* = 9.0, 6.7 Hz, 1H), 0.97 (d, *J* = 6.7 Hz, 3H), 0.67 (d, *J* = 6.7 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.95, 142.79, 141.48, 140.43, 132.45, 130.13, 128.54, 128.24, 128.20, 128.04, 127.31, 127.06, 127.04, 126.00, 53.18, 34.56, 21.08, 20.92 ppm.

DART-MS m/z (%): 313 $(M+H)^+$; **HRMS** (DART): m/z Exact mass calcd for $C_{24}H_{25}$ $[M+H]^+$: 313.1945, found: 313.1948.



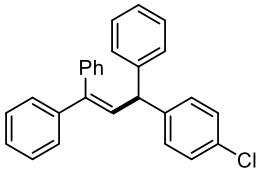
3bm

prop-1-ene-1,1,3,3-tetrayltetrabenzene (3bm), white solid (99% yield), R_f = 0.26 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2n** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 80 °C and stir was continued for 3 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.07 (m, 20H), 6.54 (d, *J* = 10.6 Hz, 1H), 4.81 (d, *J* = 10.6 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.64, 142.35, 141.74, 139.79, 131.13, 129.93, 128.64, 128.50, 128.44, 128.27, 127.62, 127.43, 127.39, 126.39, 50.64 ppm.

EI-MS m/z (%): 346 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{27}H_{22}$ $[M]^+$: 346.1716, found: 346.1723.



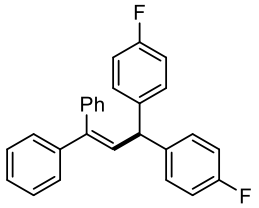
3bn

(3-(4-chlorophenyl)prop-1-ene-1,1,3-triyl)tribenzene (3bn), white solid 68% yield), R_f = 0.25 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2o** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.32 (m, 3H), 7.32-7.18 (m, 10H), 7.18-7.10 (m, 4H), 7.08 (d, *J* = 8.3 Hz, 2H), 6.47 (d, *J* = 10.5 Hz, 1H), 4.77 (d, *J* = 10.5 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.98, 143.02, 142.02, 141.95, 139.46, 132.02, 130.31, 129.66, 129.64, 128.59, 128.33, 128.22, 128.14, 127.43, 127.38, 127.36, 126.44, 49.93 ppm.

EI-MS m/z (%): 380 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{27}H_{21}Cl$ $[M]^+$: 380.1326, found: 380.1320.



3bo

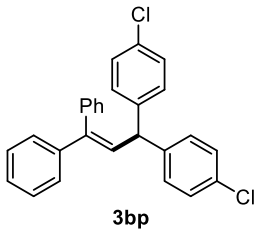
4,4'-(3,3-diphenylprop-2-ene-1,1-diyl)bis(fluorobenzene) (3bo), colorless oil (96% yield), R_f = 0.23 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2p** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for

5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.31 (m, 3H), 7.28-7.20 (m, 5H), 7.18-7.11 (m, 2H), 7.11-7.04 (m, 4H), 7.02-6.90 (m, 4H), 6.43 (d, *J* = 10.5 Hz, 1H), 4.76 (d, *J* = 10.5 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 161.60 (d, *J* = 244.9 Hz), 142.06 (d, *J* = 7.2 Hz), 140.17 (d, *J* = 3.4 Hz), 139.58, 130.59, 129.85, 129.77, 129.75, 128.54, 128.35, 127.60, 127.58, 115.51 (d, *J* = 21.4 Hz), 49.18 ppm; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -115.39 ppm.

EI-MS m/z (%): 382 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{27}H_{20}F_2$ $[M]^+$: 382.1528, found: 382.1536.



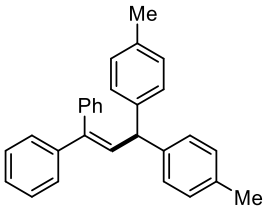
3bp

4,4'-(3,3-diphenylprop-2-ene-1,1-diyl)bis(chlorobenzene) (3bp), colorless oil (92% yield), R_f = 0.23 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2q** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.32 (m, 3H), 7.25-7.20 (m, 9H), 7.16-7.08 (m, 2H), 7.08-7.01 (m, 4H), 6.40 (d, *J* = 10.4 Hz, 1H), 4.74 (d, *J* = 10.4 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 142.68, 142.64, 141.89, 139.46, 132.44, 129.83, 129.73, 129.70, 128.89, 128.57, 128.36, 127.68, 127.59, 49.52 ppm.

EI-MS m/z (%): 414 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{27}H_{20}Cl_2$ $[M]^+$: 414.0937, found: 414.0927.



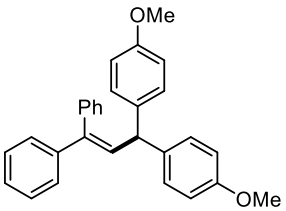
3bq

4,4'-(3,3-diphenylprop-2-ene-1,1-diyl)bis(methylbenzene) (3bq), white solid (98% yield), R_f = 0.27 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2r** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.28 (m, 3H), 7.27-7.23 (m, 3H), 7.23-7.21 (m, 2H), 7.19-7.14 (m, 2H), 7.09 (d, *J* = 8.1 Hz, 4H), 7.04 (d, *J* = 8.1 Hz, 4H), 6.51 (d, *J* = 10.6 Hz, 1H), 4.74 (d, *J* = 10.6 Hz, 1H), 2.31 (s, 6H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 142.49, 141.85, 141.28, 139.90, 135.81, 131.54, 129.96, 129.30, 128.38, 128.31, 128.22, 127.62, 127.34, 127.27, 49.88, 21.17 ppm.

EI-MS m/z (%): 374 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{29}H_{26}$ $[M]^+$: 374.2029, found: 374.2039.



3br

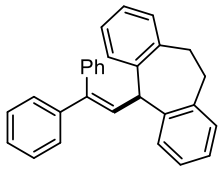
4,4'-(3,3-diphenylprop-2-ene-1,1-diyl)bis(methoxybenzene) (3br), white solid (81% yield), R_f = 0.30 (silica, petroleum ether/ethyl acetate = 50:1 ***v/v***, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2s** (0.66 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 5 h. After it

cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.29 (m, 3H), 7.29-7.20 (m, 5H), 7.20-7.11 (m, 2H), 7.06 (d, *J* = 8.5 Hz, 4H), 6.89-6.69 (m, 4H), 6.47 (d, *J* = 10.6 Hz, 1H), 4.70 (d, *J* = 10.6 Hz, 1H), 3.78 (s, 6H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.10, 142.47, 141.07, 139.89, 137.09, 131.71, 129.94, 129.34, 128.39, 128.24, 127.61, 127.36, 127.29, 113.99, 55.39, 48.99 ppm.

EI-MS m/z (%): 406 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{29}H_{26}O_2$ $[M]^+$: 406.1927, found: 406.1938.



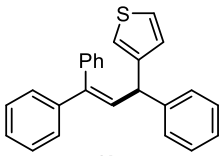
3bs

5-(2,2-diphenylvinyl)-10,11-dihydro-5H-dibenzo[a,d][7]annulene (3bs), white solid (82% yield), R_f = 0.26 (silica, petroleum ether/ethyl acetate = 50:1 ***v/v***, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2t** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.31 (m, 3H), 7.29-7.18 (m, 5H), 7.13 (dt, *J* = 5.5, 1.9 Hz, 2H), 7.07 (dq, *J* = 13.7, 3.4 Hz, 8H), 6.96 (d, *J* = 10.0 Hz, 1H), 5.02 (d, *J* = 10.0 Hz, 1H), 3.70-3.19 (m, 2H), 3.19-2.69 (m, 2H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 142.40, 141.70, 141.28, 139.84, 139.20, 130.57, 130.35, 130.05, 128.83, 128.47, 128.25, 127.40, 127.39, 127.31, 126.68, 126.22, 50.09, 33.24 ppm.

EI-MS m/z (%): 372 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{29}H_{24}$ $[M]^+$: 372.1873, found: 372.1862.

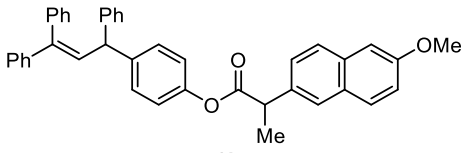


3bt

3-(1,3,3-triphenylallyl)thiophene (3bt), colorless oil (68% yield), R_f = 0.22 (silica, petroleum ether, UV and PMA detection);

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2u** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 3 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.09 (m, 16H), 6.95 (dt, *J* = 2.7, 1.1 Hz, 1H), 6.83 (dd, *J* = 5.0, 1.2 Hz, 1H), 6.50 (d, *J* = 10.5 Hz, 1H), 4.82 (d, *J* = 10.5 Hz, 1H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.18, 144.16, 142.28, 141.56, 139.72, 130.74, 129.91, 128.71, 128.47, 128.43, 128.28, 128.18, 128.01, 127.62, 127.57, 127.47, 127.42, 126.55, 125.74, 121.24, 46.76 ppm.



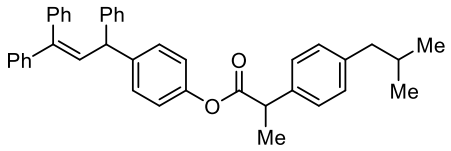
3bu

4-(1,3,3-triphenylallyl)phenyl 2-(6-methoxynaphthalen-2-yl)propanoate (3bu), colorless oil (67% yield), R_f = 0.38 (silica, petroleum ether/ethyl acetate = 15:1 v/v, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2v** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 3 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73-7.59 (m, 3H), 7.41 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.30-7.22 (m, 3H), 7.21-6.99 (m, 16H), 6.83 (d, *J* = 8.6 Hz, 2H), 6.39 (d, *J* = 10.5 Hz, 1H), 4.69 (d, *J* = 10.5 Hz, 1H), 4.00 (q, *J* = 7.1 Hz, 1H), 3.83 (s, 3H), 1.60 (d, *J* = 7.1 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.40, 157.87, 149.34, 144.34, 142.22, 142.08, 141.91, 139.68, 135.30, 133.94, 130.81, 129.84, 129.46, 129.32, 129.13, 128.67, 128.45, 128.27, 127.60, 127.49, 127.47, 127.43, 126.48, 126.27, 121.43, 119.23, 105.75, 55.46, 50.07, 45.72, 18.65 ppm.

EI-MS m/z (%): 574 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{41}H_{34}O_3$ $[M]^+$: 574.2508, found: 574.2503.



3bv

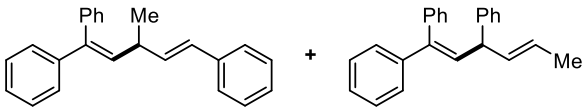
4-(1,3,3-triphenylallyl)phenyl 2-(4-isobutylphenyl)propanoate (3bv), colorless oil (74% yield), R_f = 0.22 (silica, petroleum ether/ethyl acetate = 30:1 v/v, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2w** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 3 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.03 (m, 21H), 6.95 (d, *J* = 8.1 Hz, 2H), 6.51 (d, *J* = 10.5 Hz, 1H), 4.82 (d, *J* = 10.5 Hz, 1H), 3.96 (q, *J* = 6.9 Hz, 1H), 2.50 (d, *J* = 7.1 Hz, 2H), 1.89 (dt, *J* = 13.3, 6.6 Hz, 1H), 1.63 (d, *J* = 7.0 Hz, 3H), 0.94 (d, *J* = 6.5 Hz, 6H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.46, 149.37, 144.37, 142.23, 142.03, 141.91, 140.94, 139.69, 137.39, 130.83, 129.85, 129.62, 129.31, 128.68, 128.46, 128.28, 127.61, 127.48, 127.44, 127.34, 126.49, 121.45, 50.08, 45.39, 45.18, 30.33, 22.53, 18.63 ppm.

EI-MS m/z (%): 550 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{40}H_{38}O_2$ $[M]^+$:

550.2872, found: 550.2869.



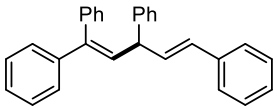
3bw **3bw'**

(*E*)-(3-methylpenta-1,4-diene-1,1,5-triyl)tribenzene (3bw) and **(*E*)-hexa-1,4-diene-1,1,3-triyltribenzene (3bw'),** colorless oil (87% yield, 3bw/3bw' = 1:0.6), R_f = 0.53 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2x** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 3 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.09 (m, 15+9H), 6.38 (d, *J* = 16.3 Hz, 1H), 6.32-6.23 (m, 1+0.6H), 6.05 (d, *J* = 10.0 Hz, 1H), 5.71 (ddd, *J* = 15.3, 6.3, 1.5 Hz, 0.6H), 5.62-5.50 (m, 0.6H), 4.19 (dd, *J* = 10.1, 6.5 Hz, 0.6H), 3.45 – 3.04 (m, 1H), 1.77 (dt, *J* = 6.3, 1.3 Hz, 1.8H), 1.26 (d, *J* = 6.8 Hz, 3H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.32, 142.59, 142.54, 141.07, 140.99, 140.25, 140.03, 137.86, 134.90, 133.53, 132.92, 130.98, 129.92, 129.88, 128.63, 128.36, 128.25, 128.22, 127.80, 127.51, 127.43, 127.28, 127.21, 127.17, 127.13, 126.31, 126.21, 125.94, 48.13, 37.24, 21.35, 18.24 ppm.

EI-MS m/z (%): 310 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{24}H_{22}$ $[M]^+$: 310.1722, found: 310.1726.



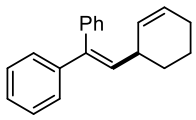
3bx

(*E*)-penta-1,4-diene-1,1,3,5-tetrayltetrabenzene (3bx), colorless oil (88% yield), R_f = 0.46 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2y** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 3 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.05 (m, 1H), 6.40-6.28 (m, 2H), 6.23 (d, *J* = 10.2 Hz, 1H), 4.27 (dd, *J* = 10.2, 4.0 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.68, 142.35, 141.98, 139.89, 137.56, 132.55, 130.40, 129.99, 129.90, 128.79, 128.68, 128.47, 128.29, 127.94, 127.57, 127.45, 127.40, 126.61, 126.40, 48.43 ppm.

EI-MS m/z (%): 372 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{29}H_{24}$ $[M]^+$: 372.1878, found: 372.1870.



3by

(2-(cyclohex-2-en-1-yl)ethene-1,1-diyl)dibenzene (3by), colorless oil (66% yield), R_f = 0.38 (silica, petroleum ether, UV and PMA detection).

To an oven-dried, 10 mL Teflon-lined screw-capped Pyrex test tube, **1a** (0.6 mmol) and **2z** (1.8 mmol) were added without argon protection. A magnetic stir bar was added to the tube carefully and the mixture was stirred slowly for 5 min at room temperature. Then the temperature was increased to 100 °C and stir was continued for 5 h. After it cooled down to room temperature, it was purified by flash chromatography on silica gel to afford pure product.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.34 (m, 2H), 7.33-7.28 (m, 1H), 7.27-7.13 (m, 7H), 5.94 (d, *J* = 10.4 Hz, 1H), 5.84-5.62 (m, 1H), 5.56-5.46 (m, 1H), 3.00-2.85 (m, 1H), 2.08-1.87 (m, 2H), 1.87-1.65 (m, 2H), 1.58-1.34 (m, 2H) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$) δ 142.70, 140.90, 140.32, 133.82, 130.17, 129.95, 128.35, 128.20, 127.83, 127.34, 127.06, 127.02, 36.12, 29.70, 24.99, 21.01 ppm.

EI-MS m/z (%): 260 (M^+); **HRMS** (EI): m/z Exact mass calcd for $C_{20}H_{20}$ $[M]^+$: 260.1560, found: 260.1552.

S4. NMR characterization of substrate interactions

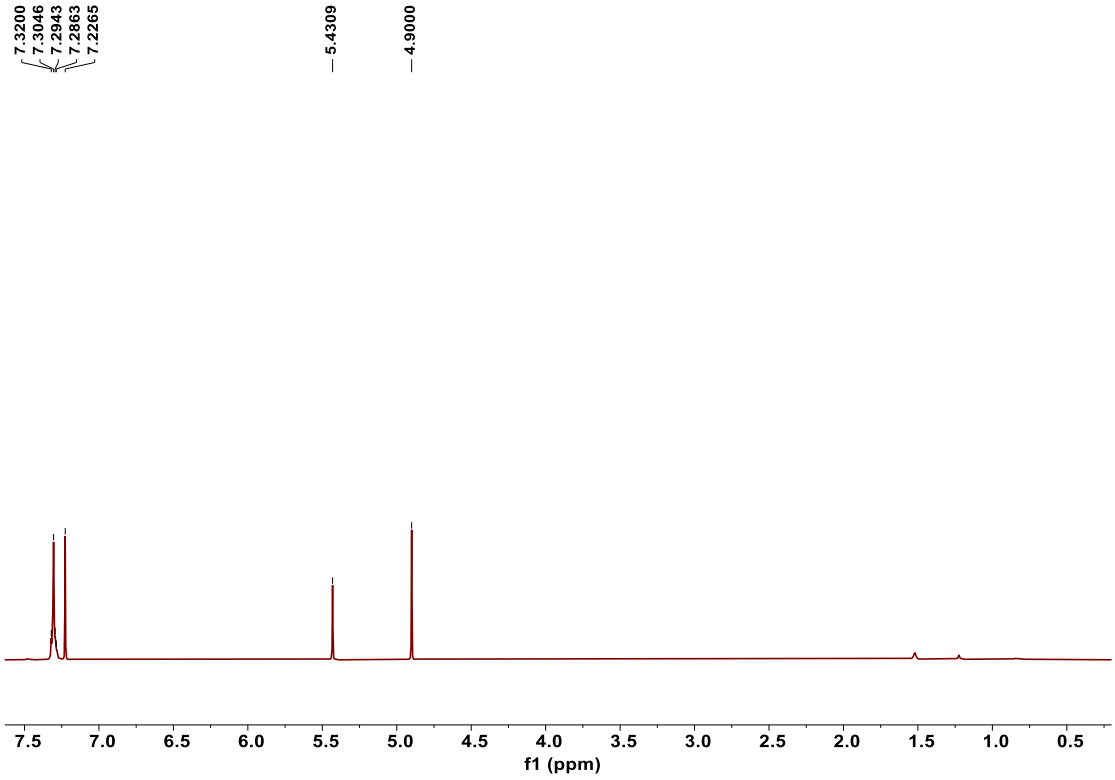


Figure S1. ^{1}H NMR spectrum of **1a** in the single-molecular state.

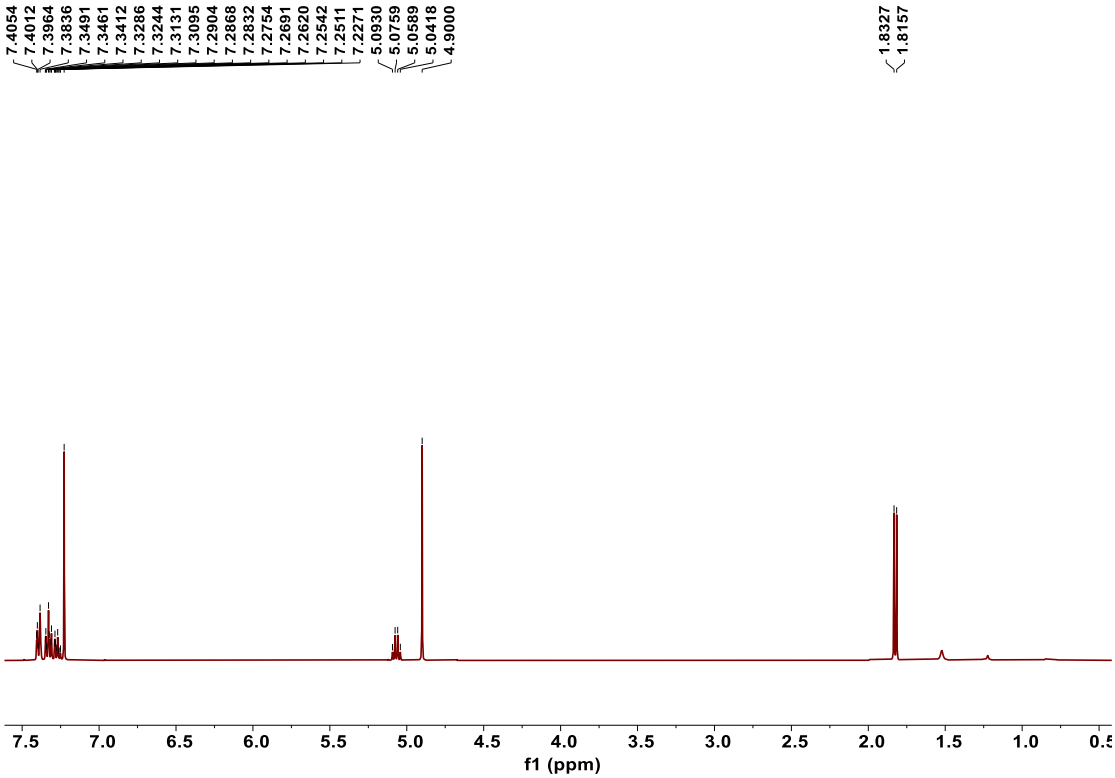


Figure S2. ^{1}H NMR spectrum of **2a** in the single-molecular state.

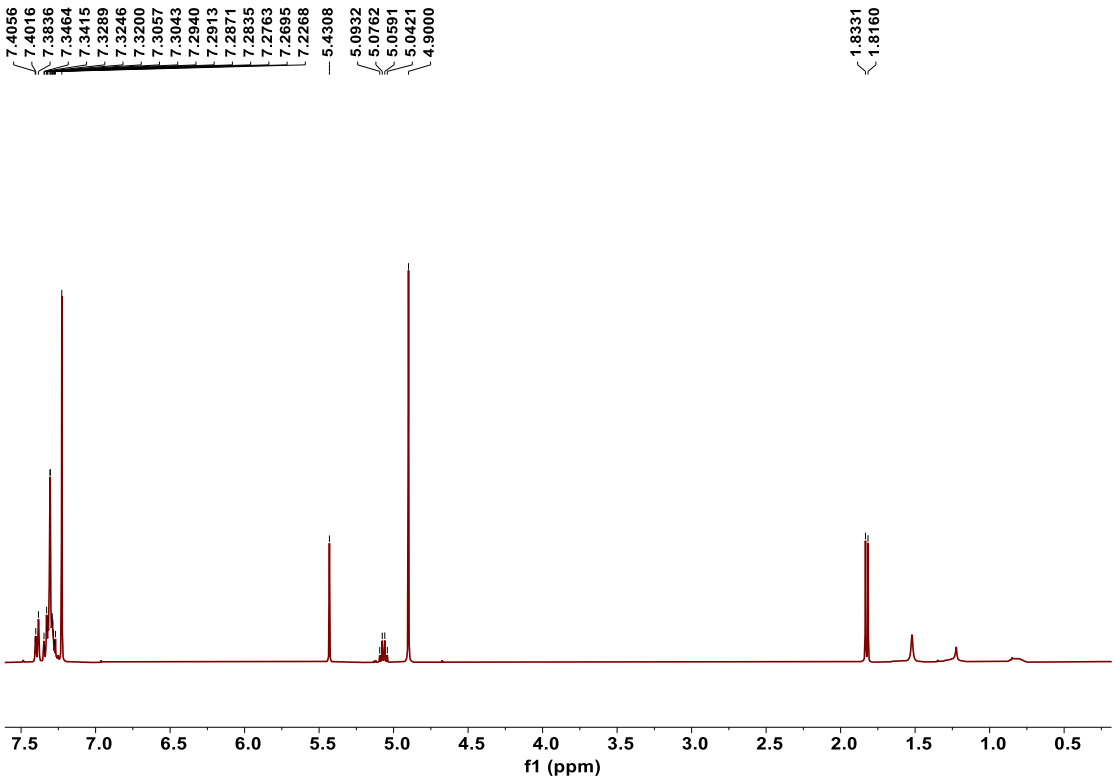


Figure S3. ^{1}H NMR spectrum of **1a**+**2a** in the single-molecular state. (V_{1a}/V_{2a} = 1:1)

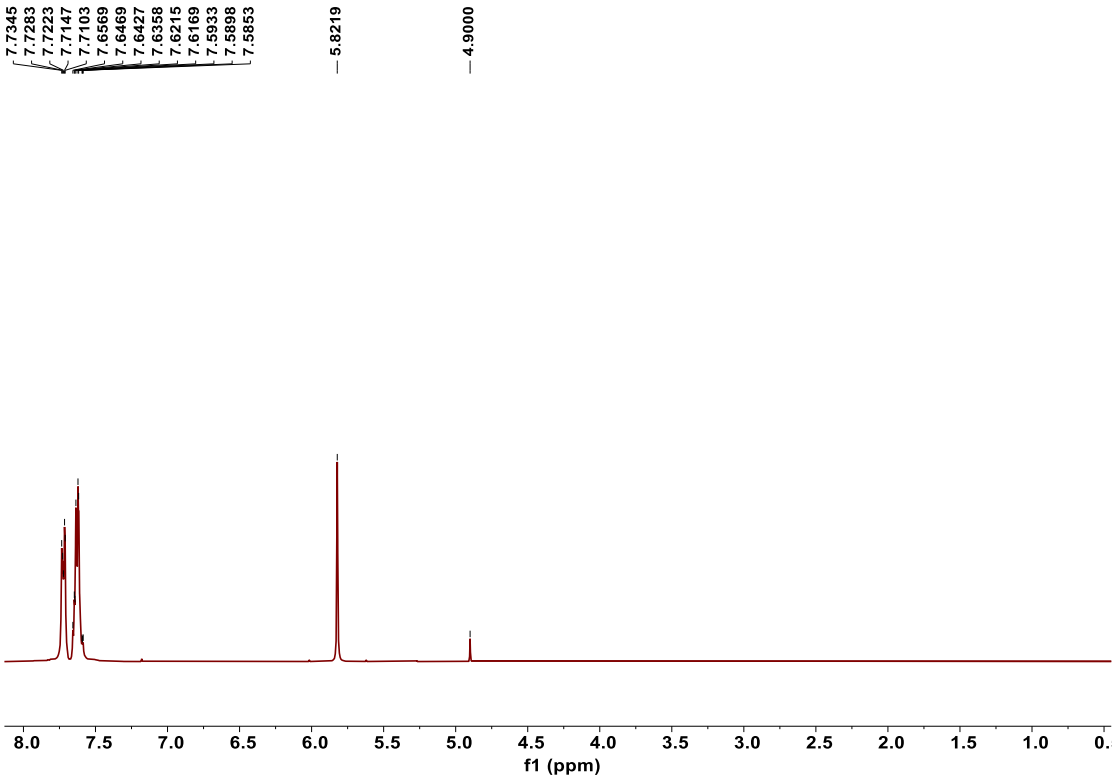


Figure S4. ^{1}H NMR spectrum of **1a** in the aggregate molecular state.

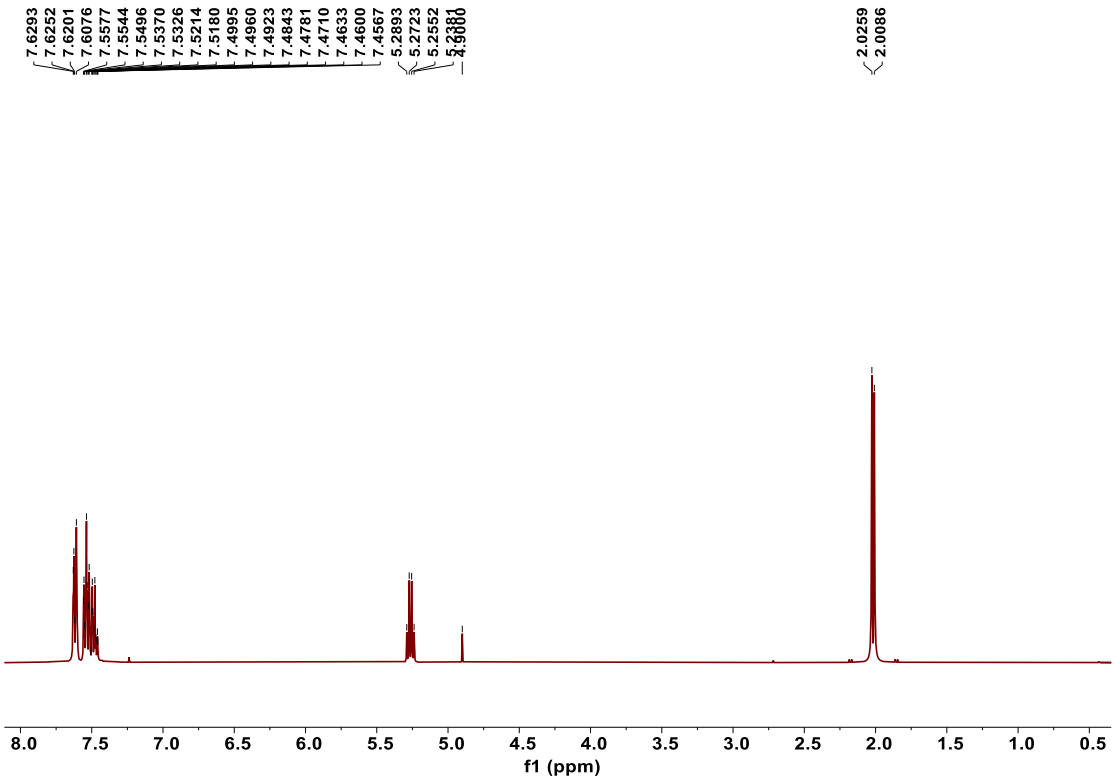


Figure S5. ^{1}H NMR spectrum of **2a** in the aggregate molecular state.

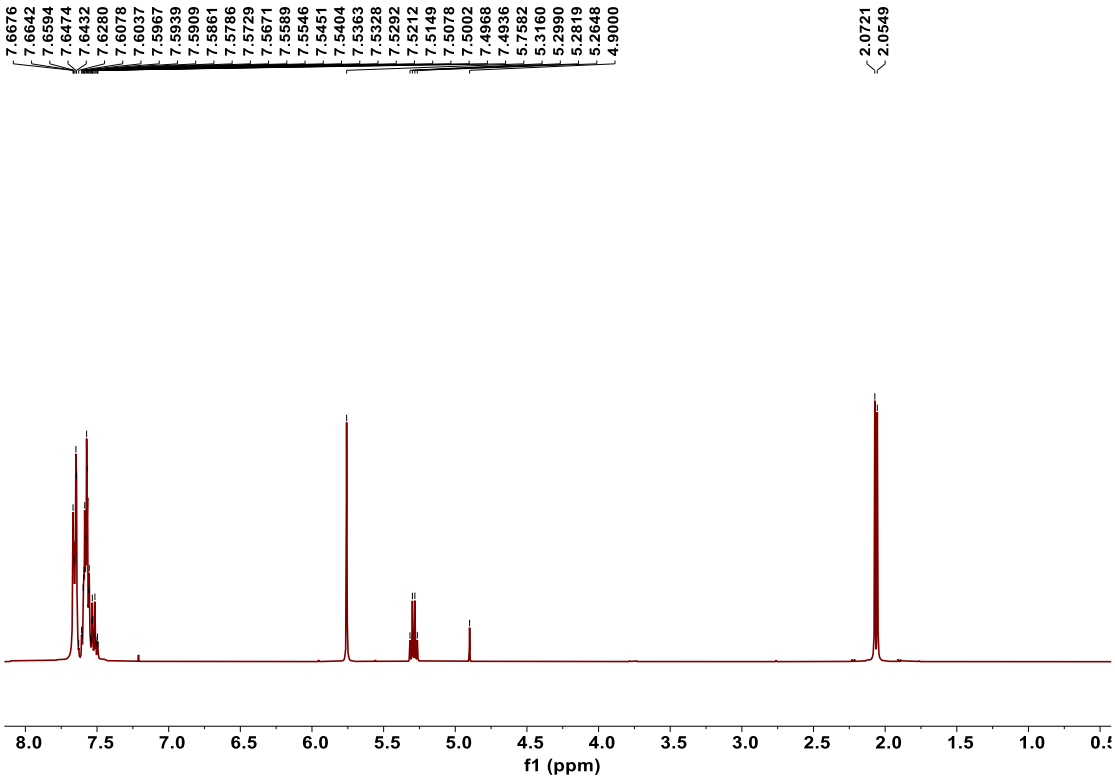


Figure S6. ^{1}H NMR spectrum of **1a**+**2a** in the aggregate molecular state. ($V_{\mathbf{1a}}/V_{\mathbf{2a}}$ = 1:1)

S5. Control experiments

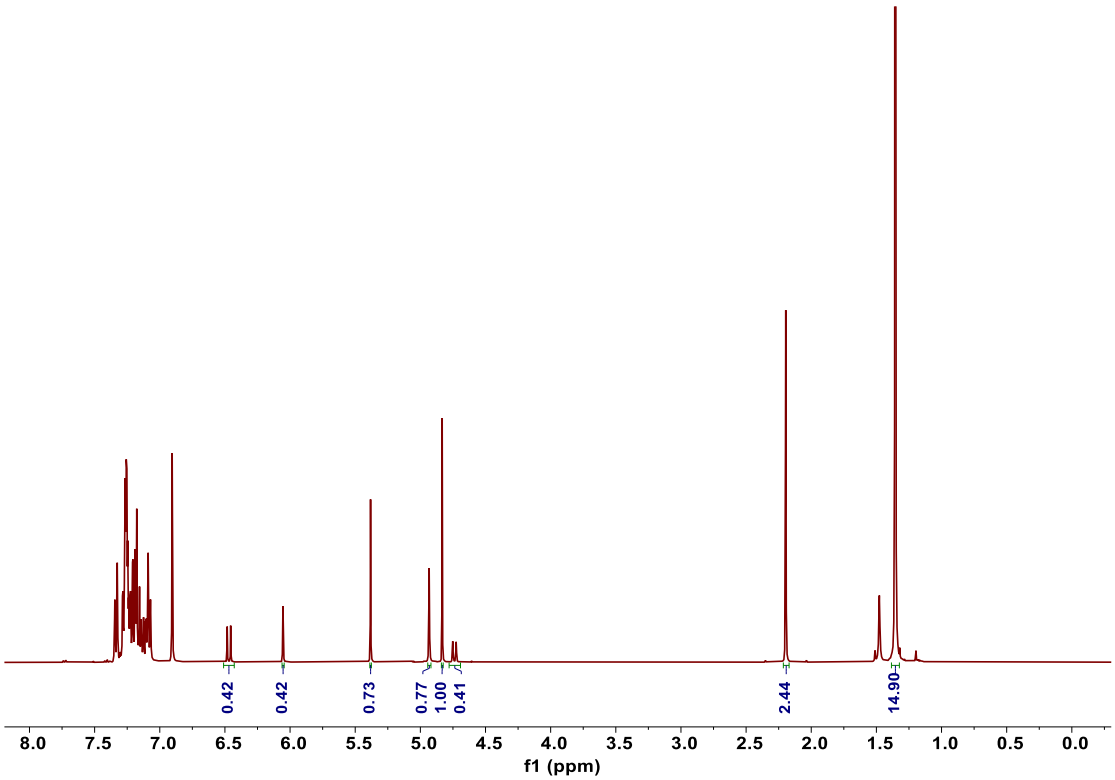


Figure S7. ^{1}**H NMR** spectrum of radical capture experiment

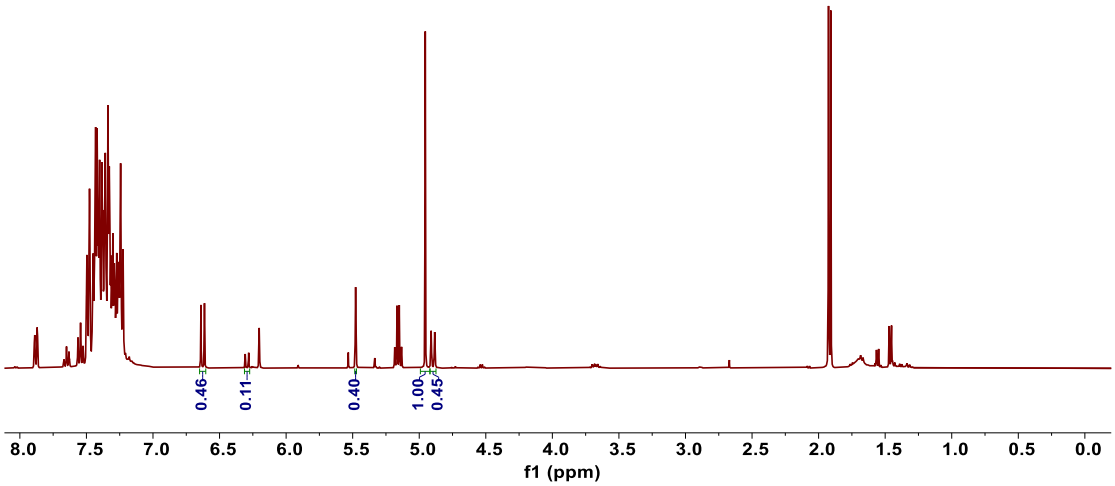
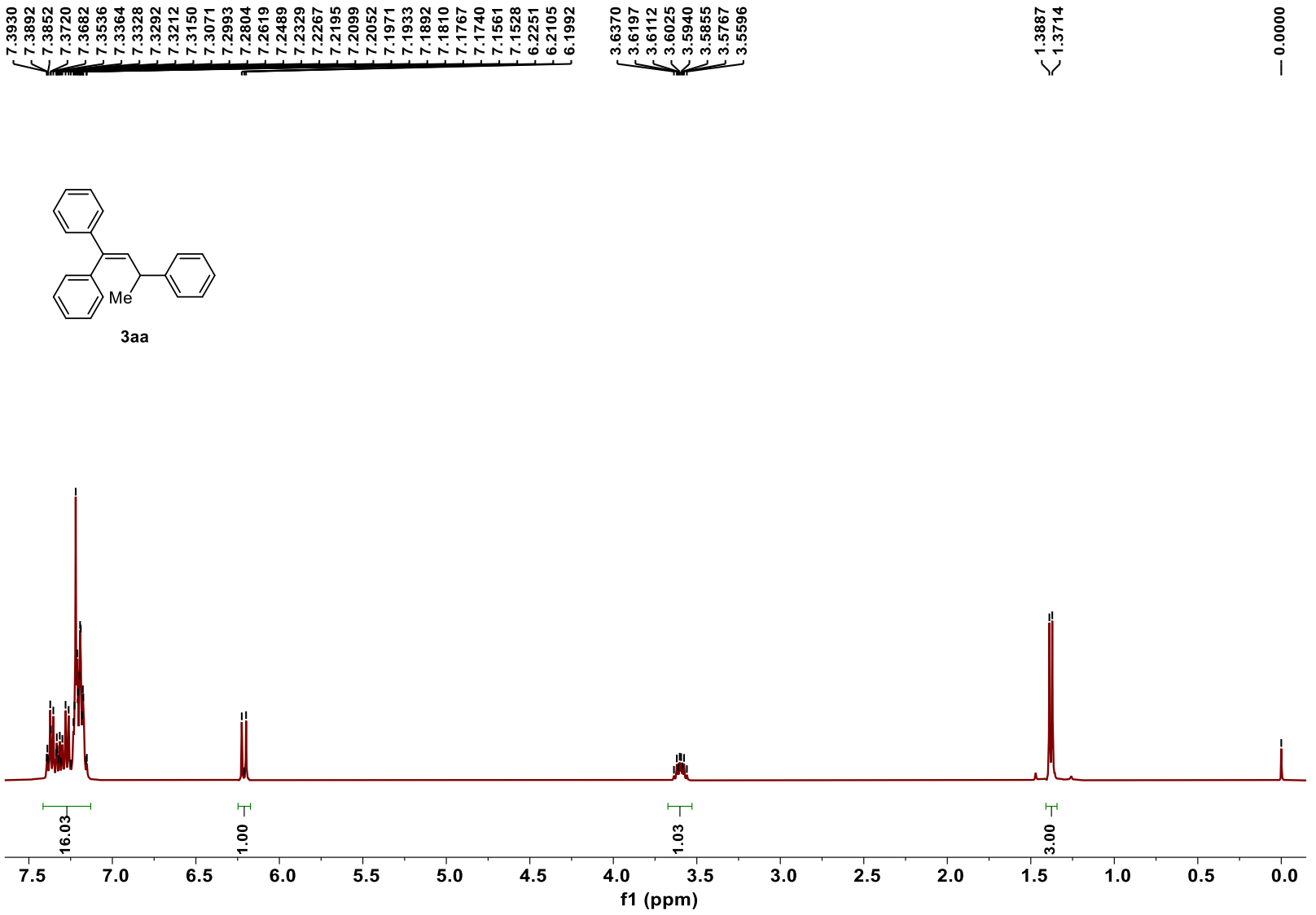
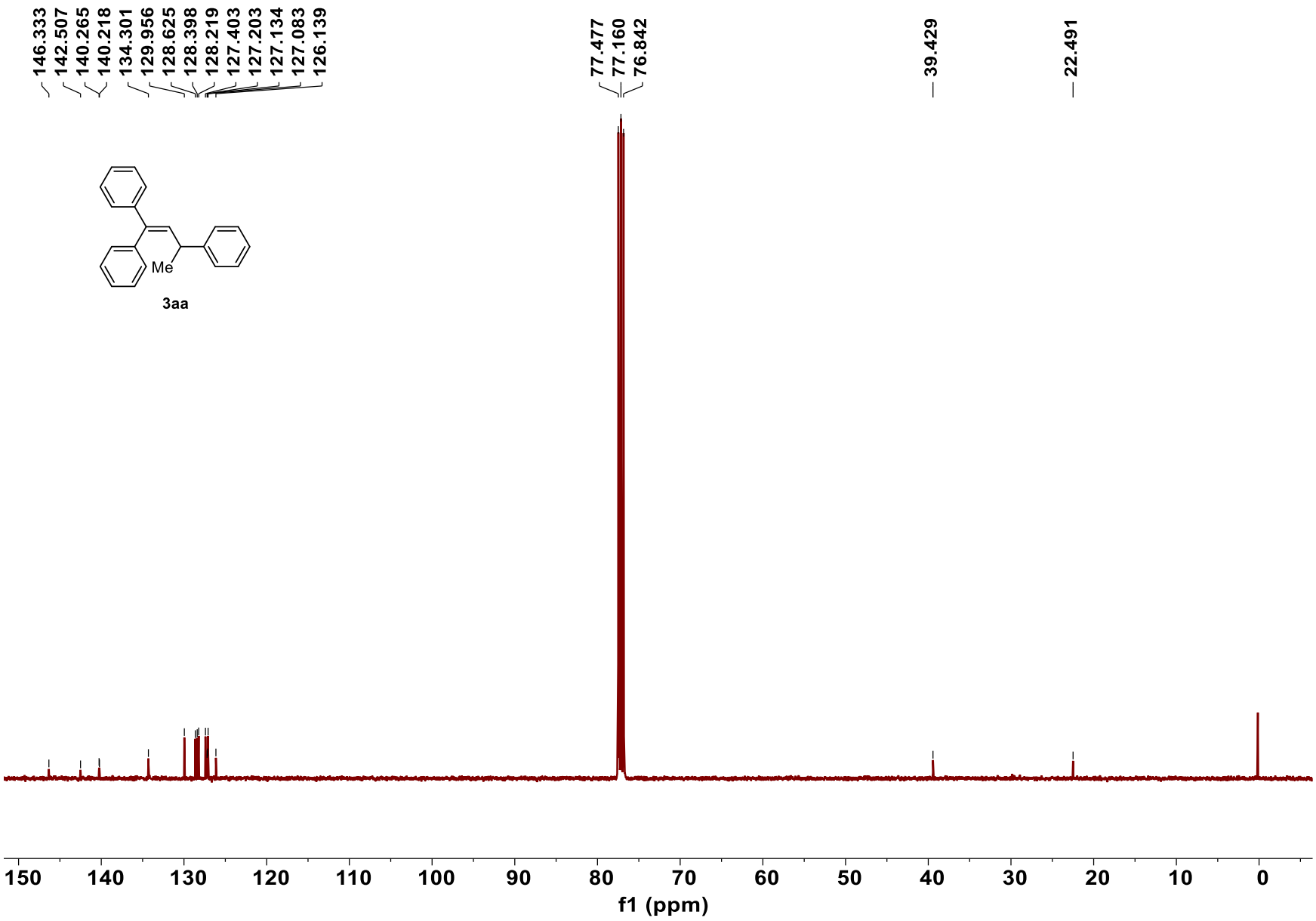
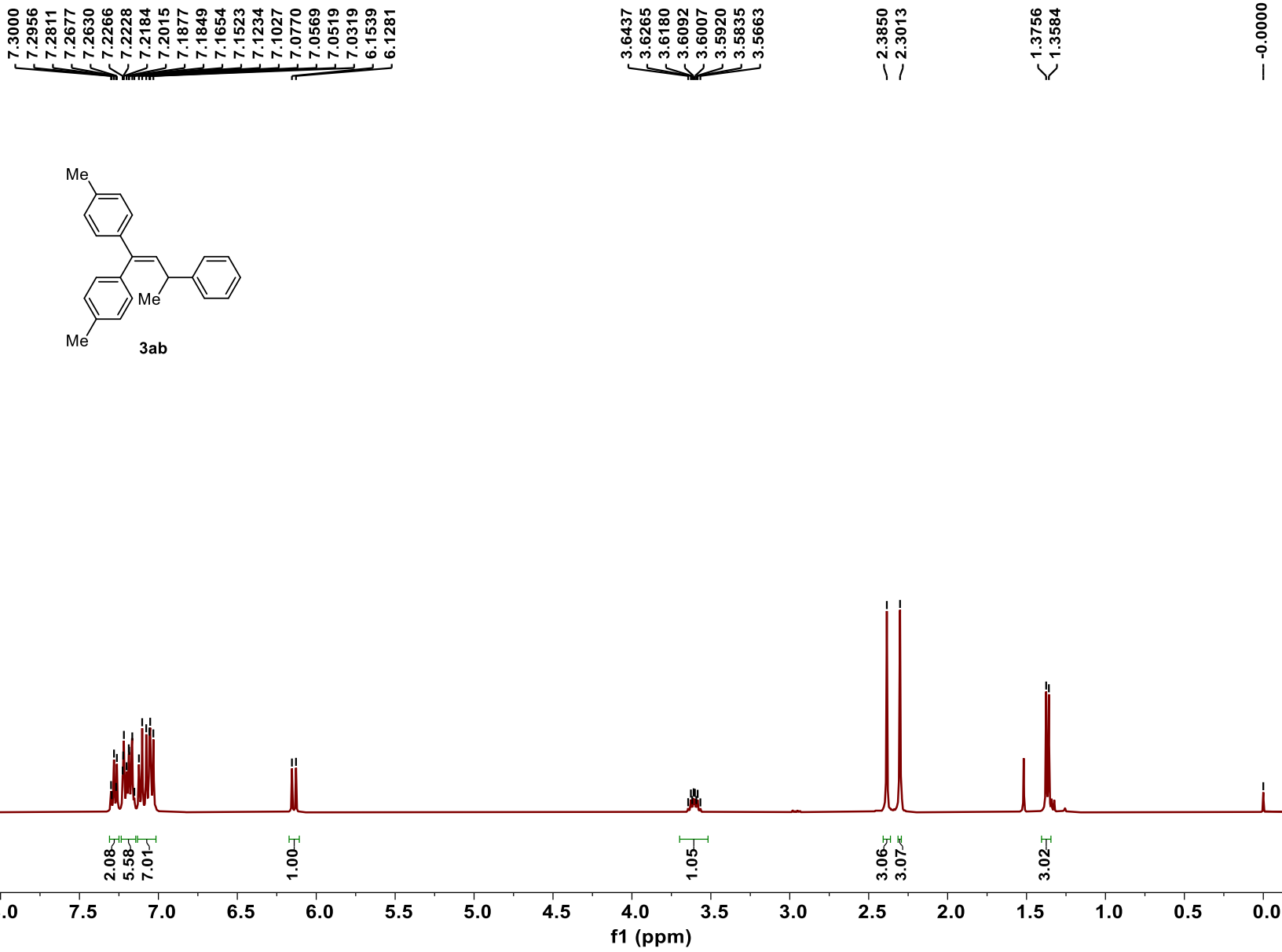


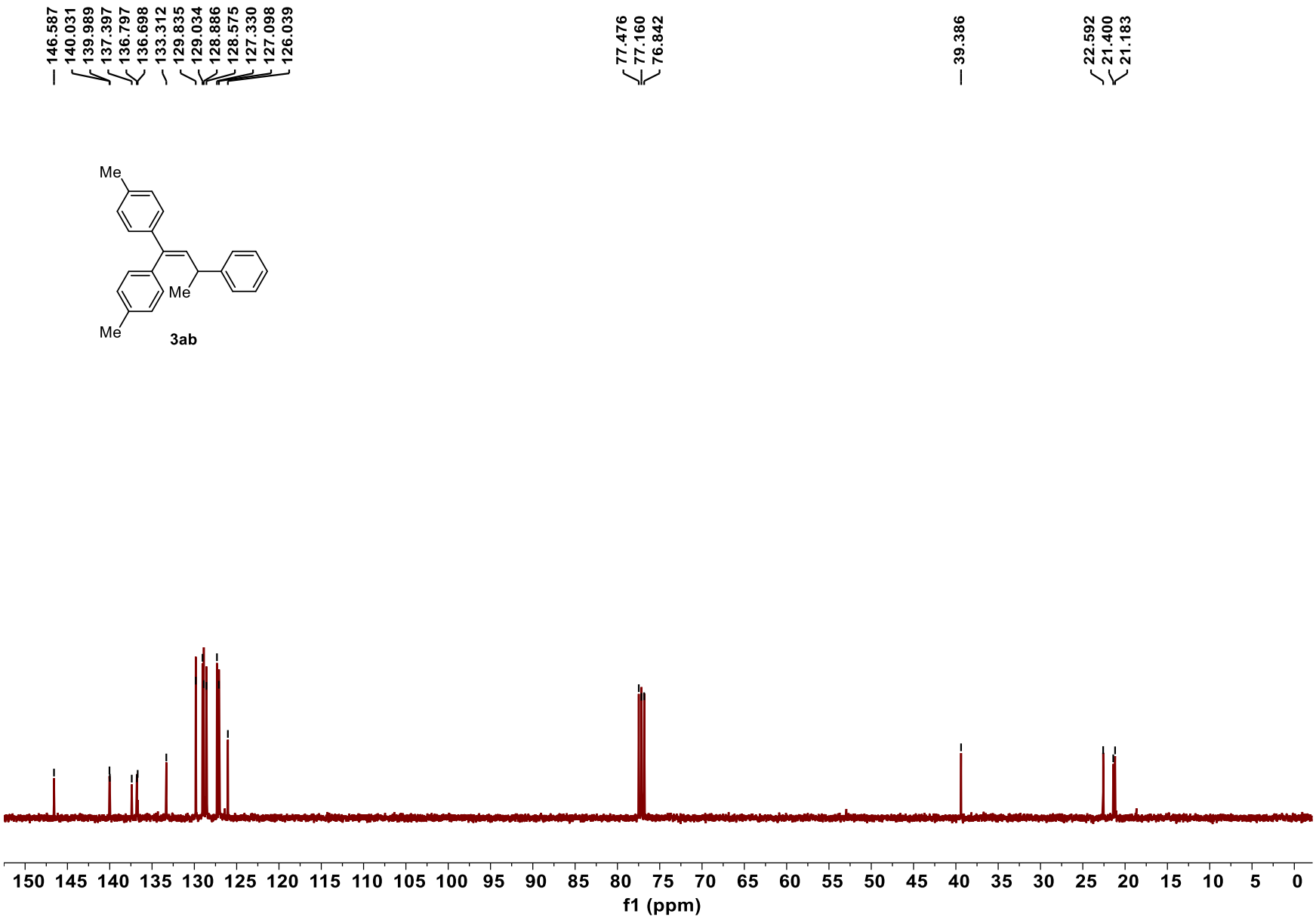
Figure S8. ^{1}**H NMR** spectrum of cation-exchange experiment

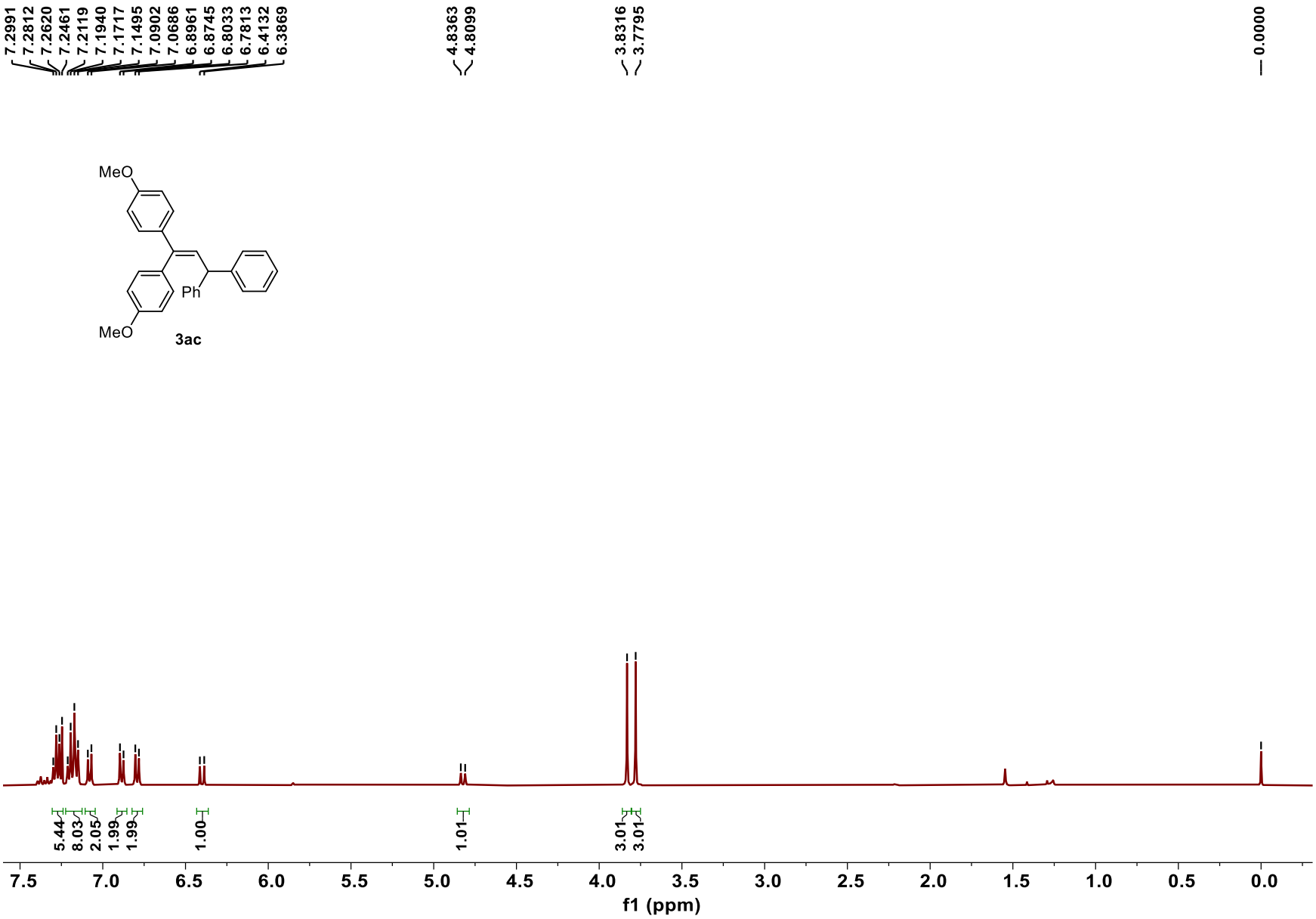
S6. NMR Spectra

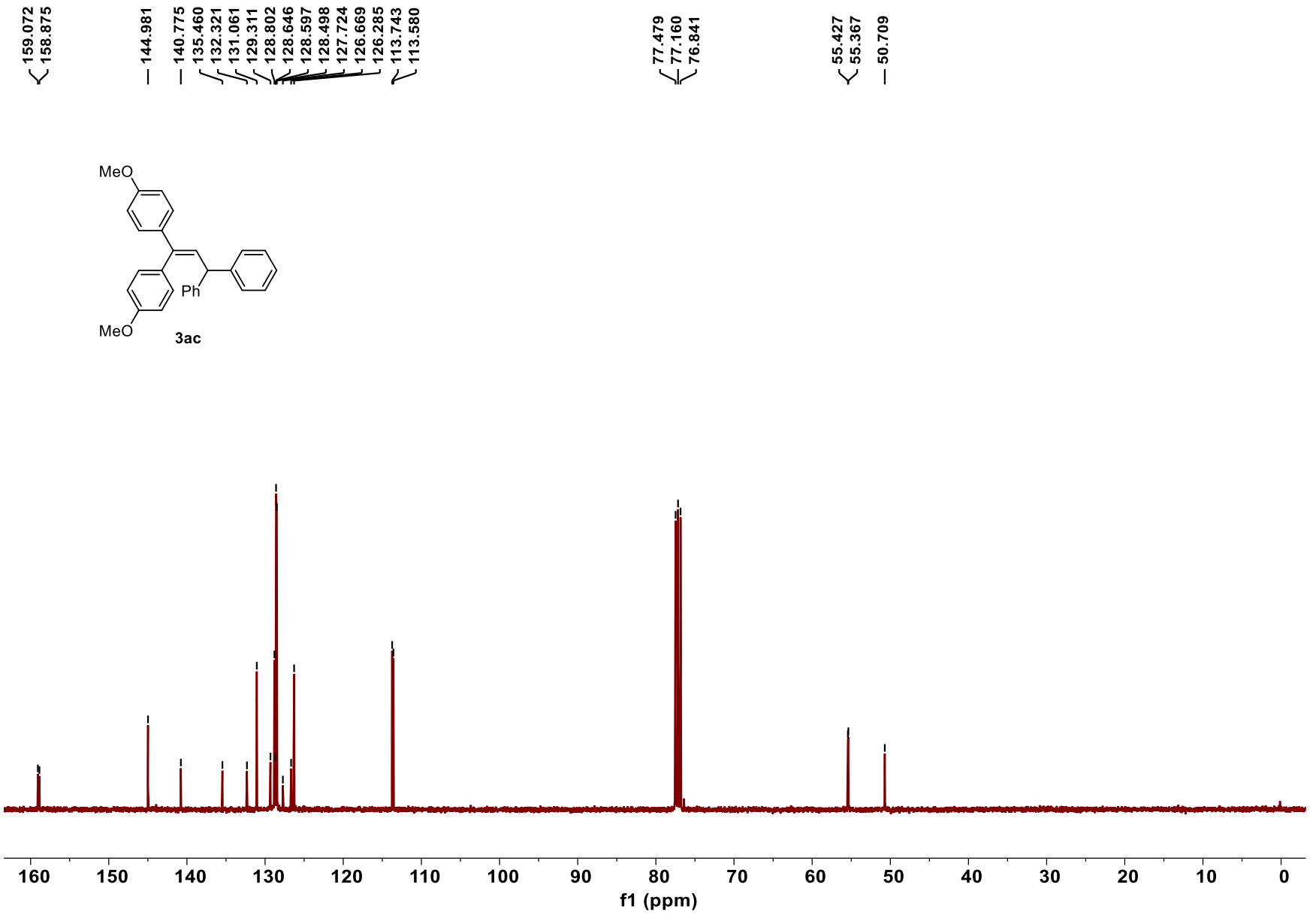


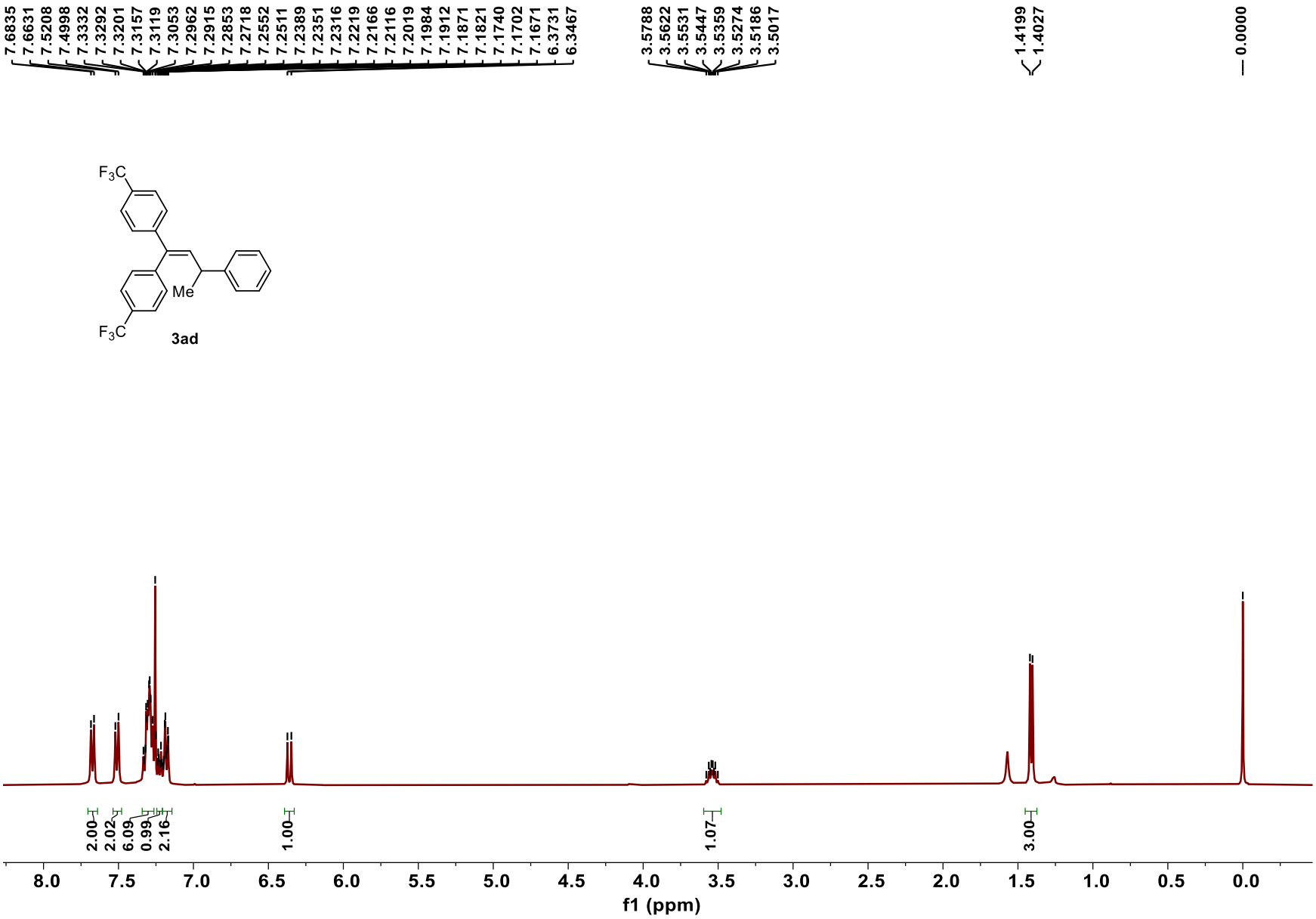

146.333
142.507
140.265
140.218
134.301
129.956
128.625
128.398
128.219
127.403
127.203
127.134
127.083
126.139
77.477
77.160
76.842
39.429
22.491
Me
3aa
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

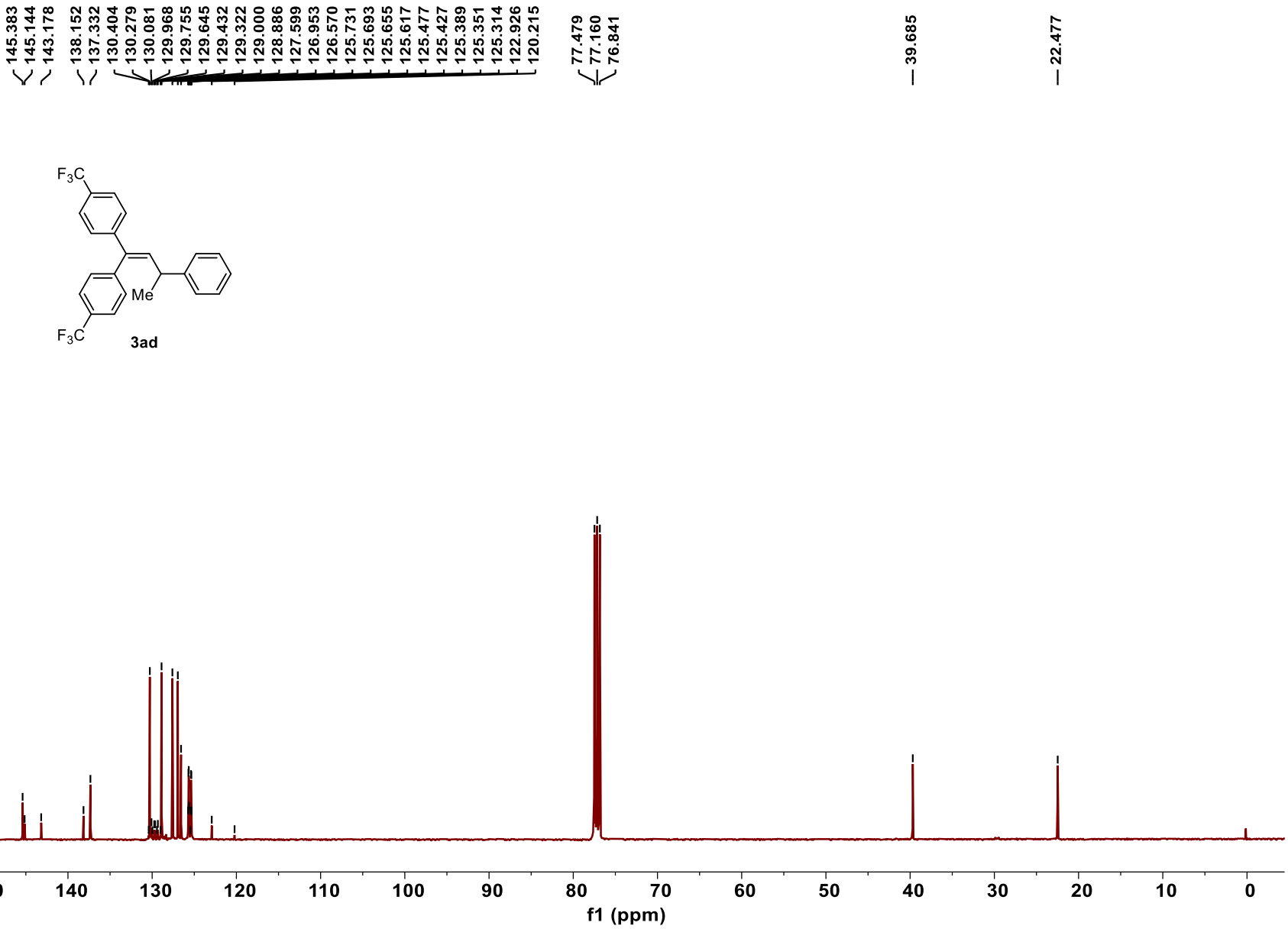

7.3000
7.2956
7.2811
7.2677
7.2630
7.2266
7.2228
7.2184
7.2015
7.1877
7.1849
7.1654
7.1523
7.1234
7.1027
7.0770
7.0569
7.0519
7.0319
6.1539
6.1281
3.6437
3.6265
3.6180
3.6092
3.6007
3.5920
3.5835
3.5663
2.3850
2.3013
1.3756
1.3584
-0.0000
Me
Me
Me
3ab
2.08
5.58
7.01
1.00
1.05
3.06
3.07
3.02
8.0
7.5
7.0
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)


146.587
140.031
139.989
137.397
136.797
136.698
133.312
129.835
129.034
128.886
128.575
127.330
127.098
126.039
77.476
77.160
76.842
39.386
22.592
21.400
21.183
Me
Me
Me
3ab
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)


7.2991
7.2812
7.2620
7.2461
7.2119
7.1940
7.1717
7.1495
7.0902
7.0686
6.8961
6.8745
6.8033
6.7813
6.4132
6.3869
4.8363
4.8099
3.8316
3.7795
0.0000
MeO
Ph
MeO
3ac
5.44
8.03
2.05
1.99
1.99
1.00
1.01
3.01
3.01
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)

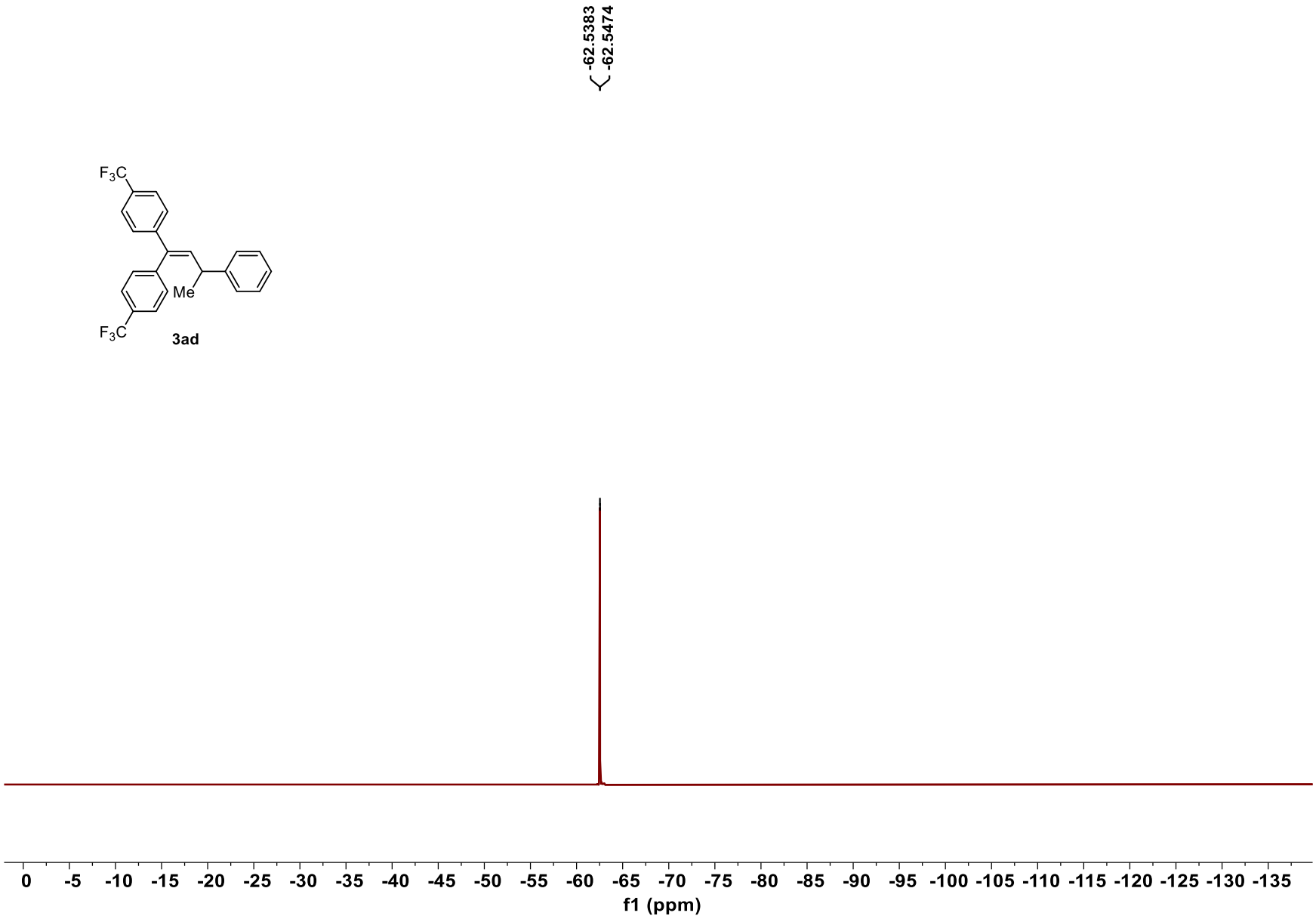

159.072
158.875
144.981
140.775
135.460
132.321
131.061
129.311
128.802
128.646
128.597
128.498
127.724
126.669
126.285
113.743
113.580
77.479
77.160
76.841
55.427
55.367
50.709
MeO
Ph
MeO
3ac
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

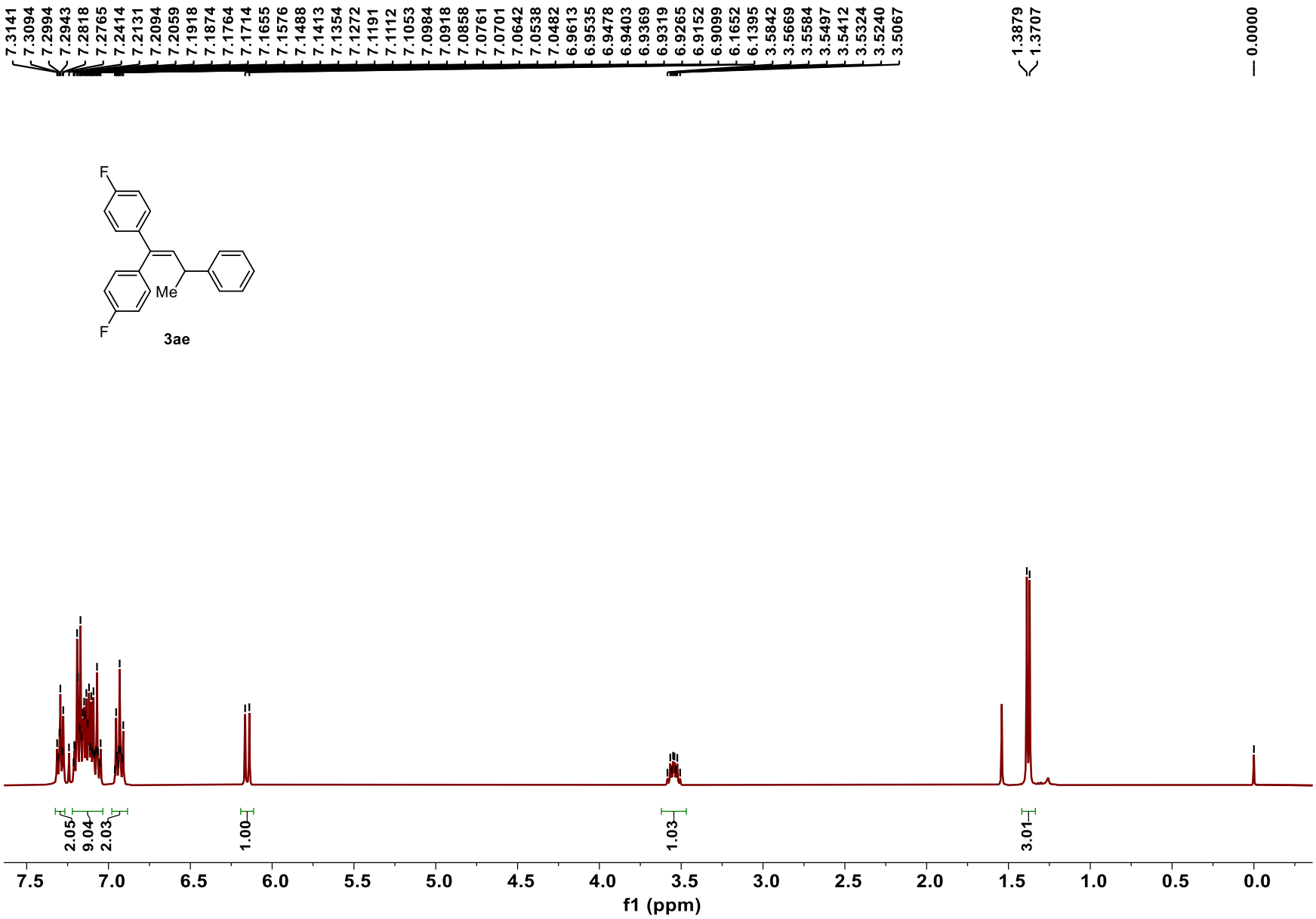

7.6835
7.6631
7.5208
7.4998
7.3332
7.3292
7.3201
7.3157
7.3119
7.3053
7.2962
7.2915
7.2853
7.2718
7.2552
7.2511
7.2389
7.2351
7.2316
7.2219
7.2166
7.2116
7.2019
7.1984
7.1912
7.1871
7.1821
7.1740
7.1702
7.1671
6.3731
6.3467
3.5788
3.5622
3.5531
3.5447
3.5359
3.5274
3.5186
3.5017
1.4199
1.4027
0.0000
F3C
Me
F3C
3ad
2.00
2.02
6.09
0.99
2.16
1.00
1.07
3.00
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)

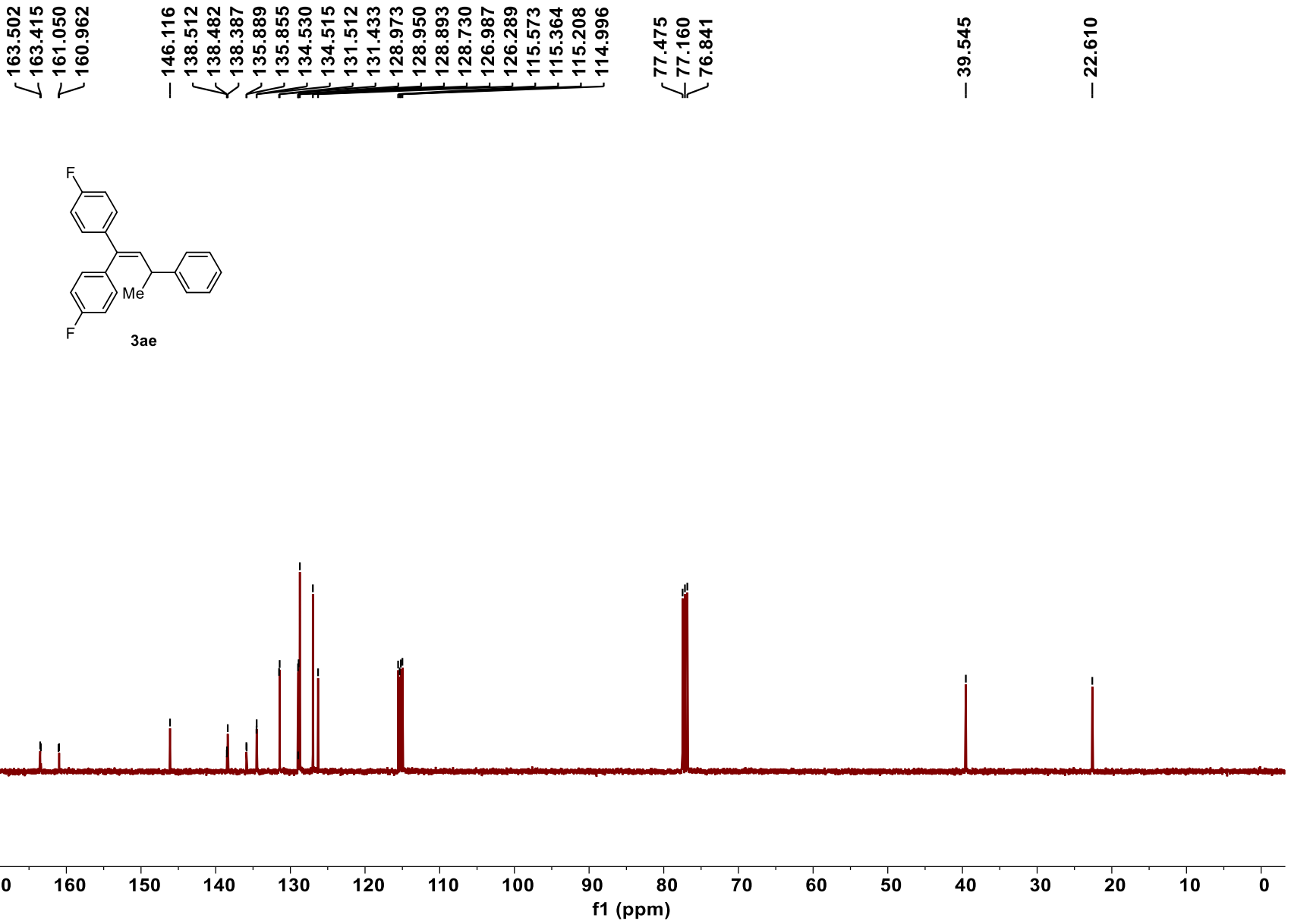

145.383
145.144
143.178
138.152
137.332
130.404
130.279
130.081
129.968
129.755
129.645
129.432
129.322
129.000
128.886
127.599
126.953
126.570
125.731
125.693
125.655
125.617
125.477
125.427
125.389
125.351
125.314
122.926
120.215
77.479
77.160
76.841
39.685
22.477
F3C
Me
F3C
3ad
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

F3C
Me
F3C

3ad

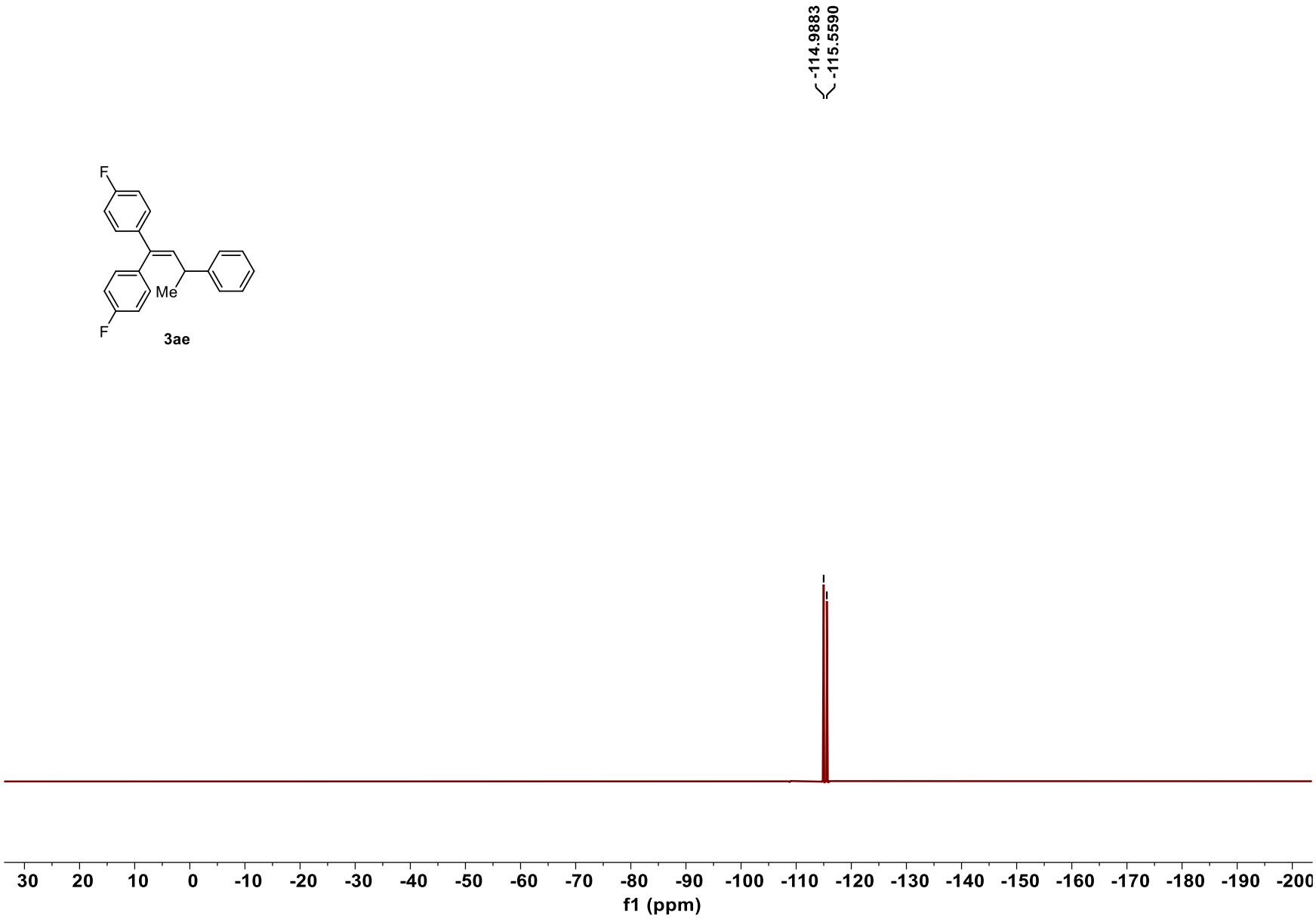

-62.5383
-62.5474
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135
f1 (ppm)

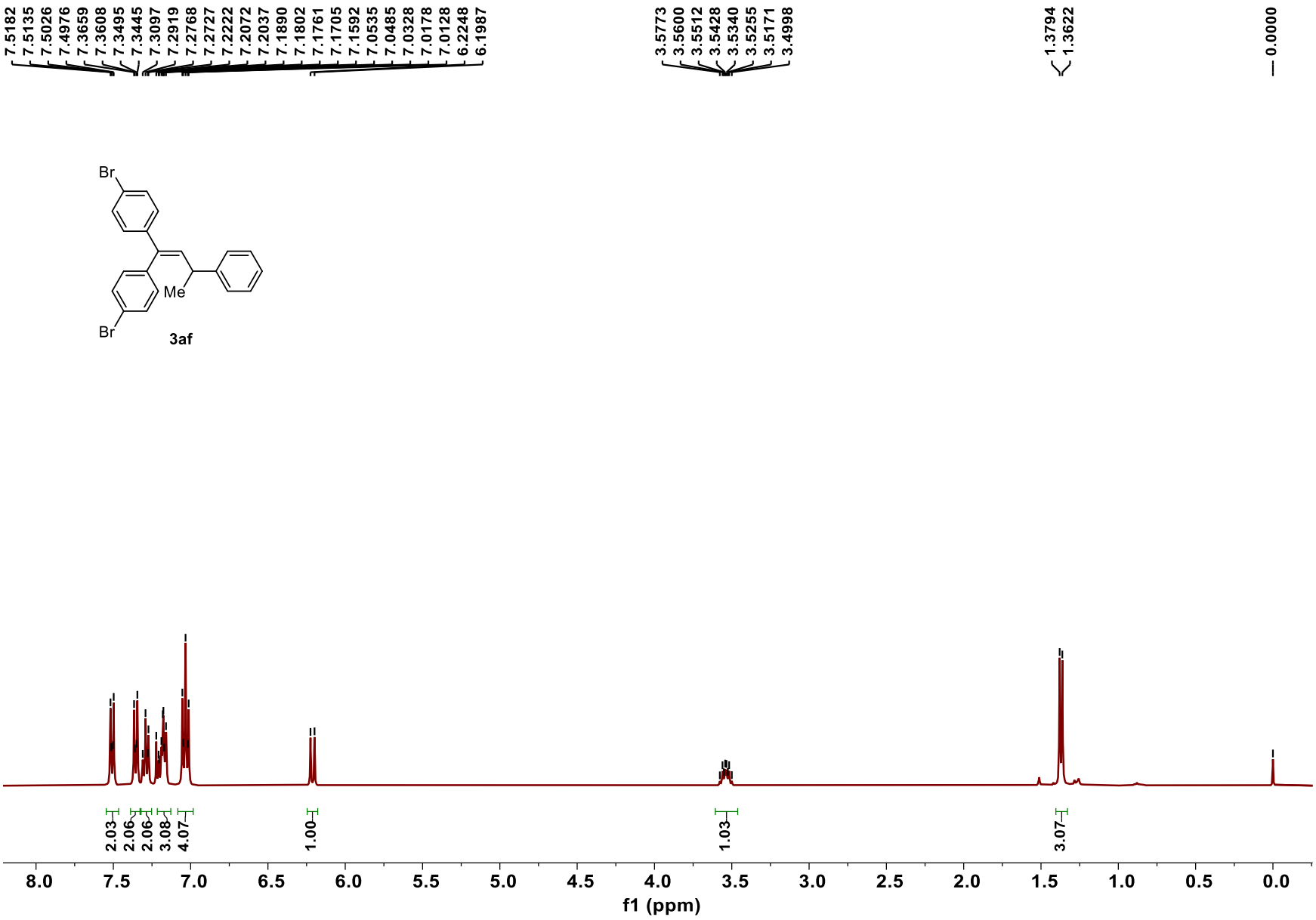

7.3141
7.3094
7.2994
7.2943
7.2818
7.2765
7.2414
7.2131
7.2094
7.2059
7.1918
7.1874
7.1764
7.1714
7.1655
7.1576
7.1488
7.1413
7.1354
7.1272
7.1191
7.1112
7.1053
7.0984
7.0918
7.0858
7.0761
7.0701
7.0642
7.0538
7.0482
6.9613
6.9535
6.9478
6.9403
6.9369
6.9319
6.9265
6.9152
6.9099
6.1652
6.1395
3.5842
3.5669
3.5584
3.5497
3.5412
3.5324
3.5240
3.5067
1.3879
1.3707
0.0000
F
Me
F
3ae
2.05
9.04
2.03
1.00
1.03
3.01
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)

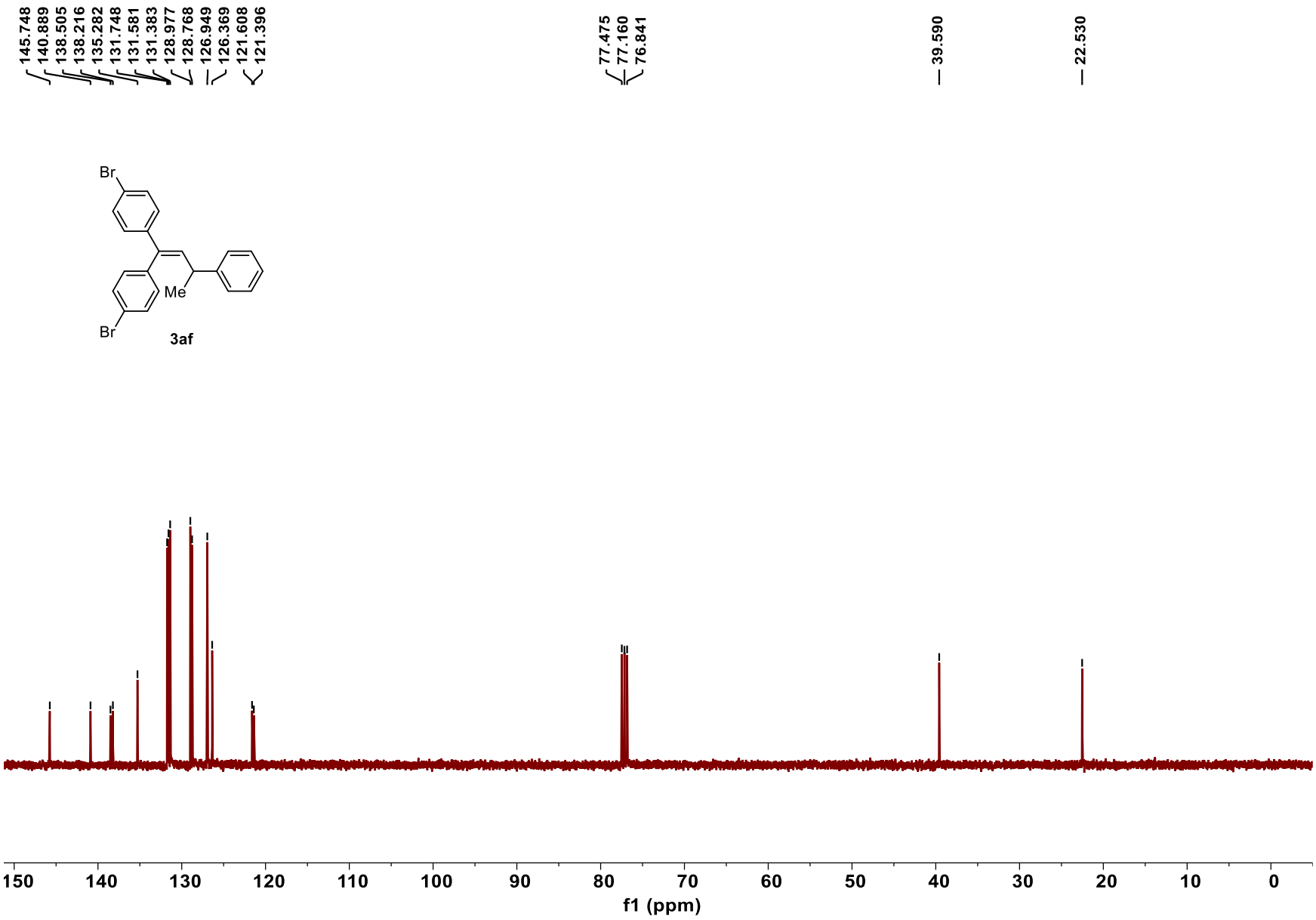

163.502
163.415
161.050
160.962
146.116
138.512
138.482
138.387
135.889
135.855
134.530
134.515
131.512
131.433
128.973
128.950
128.893
128.730
126.987
126.289
115.573
115.364
115.208
114.996
77.475
77.160
76.841
39.545
22.610
F
Me
F
3ae
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

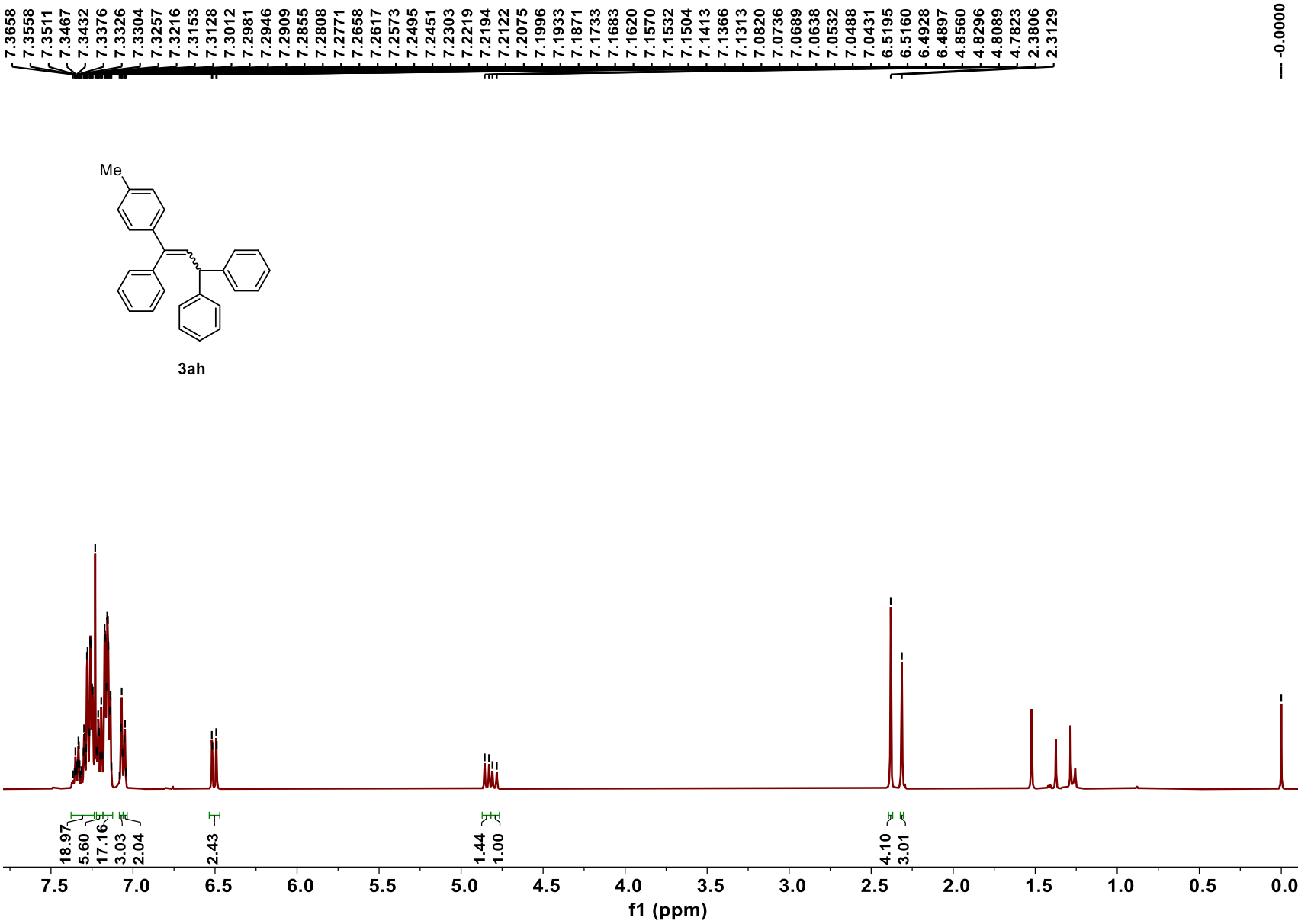
F
Me
F

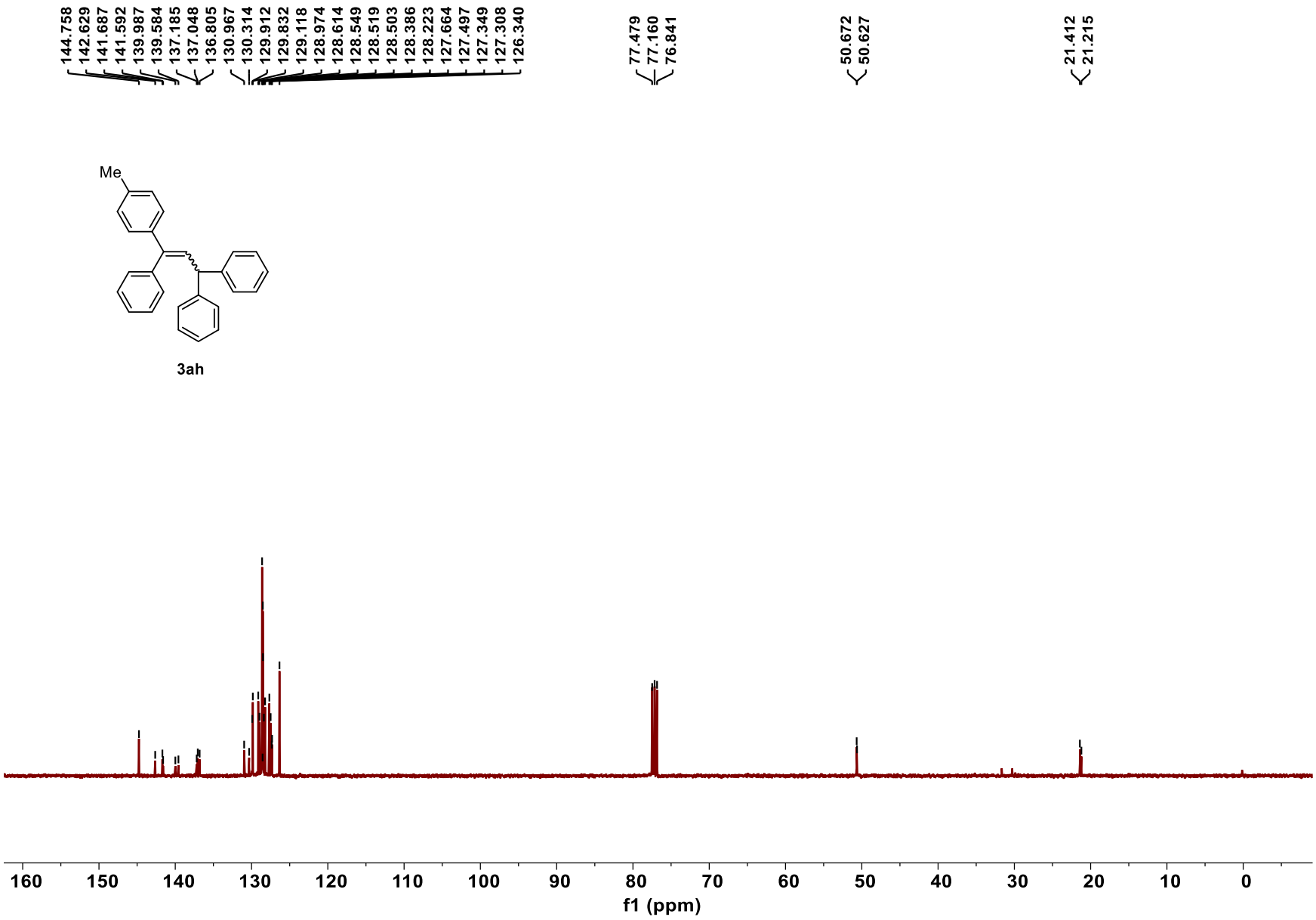
3ae

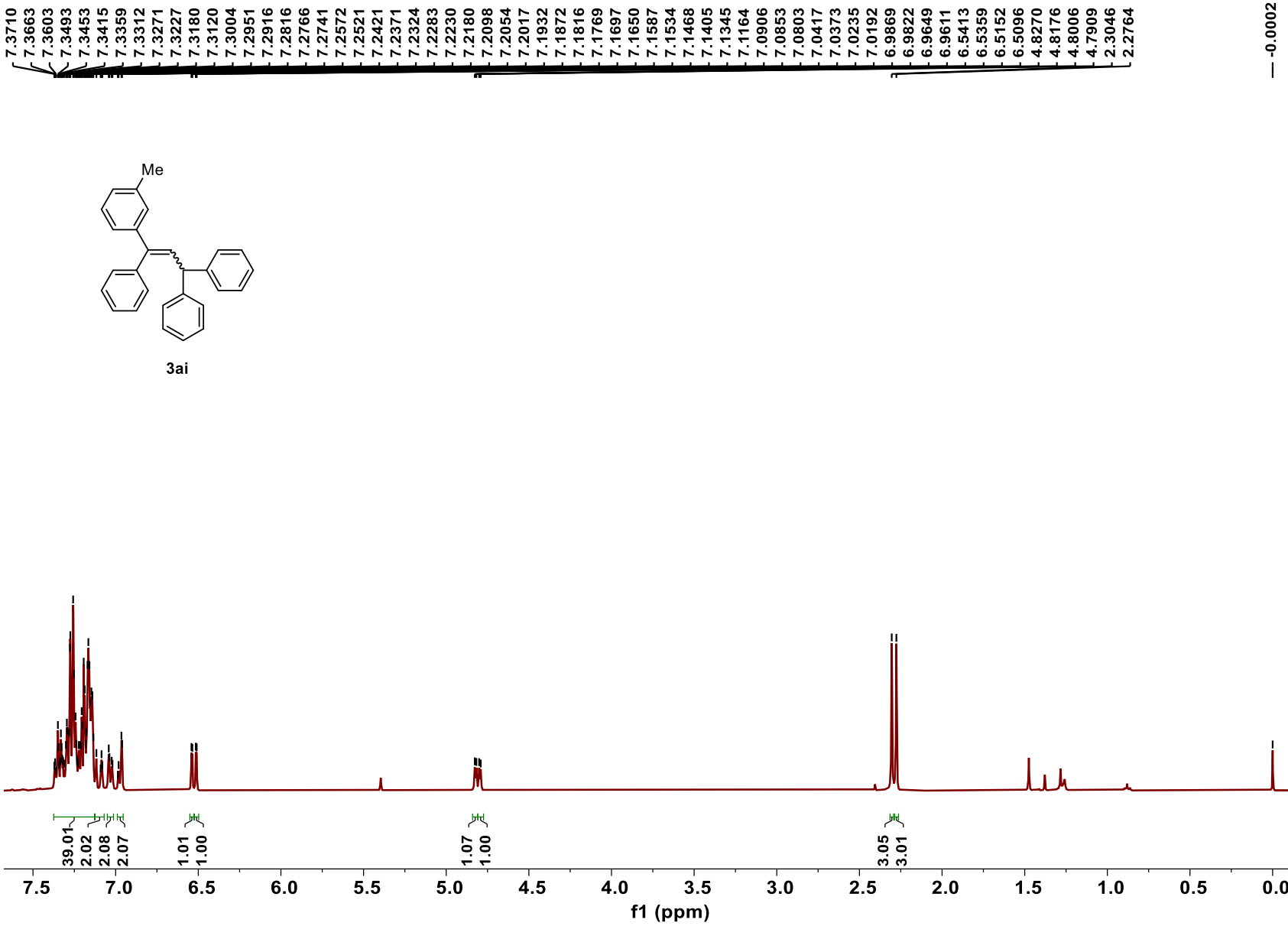

-114.9883
-115.5590
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

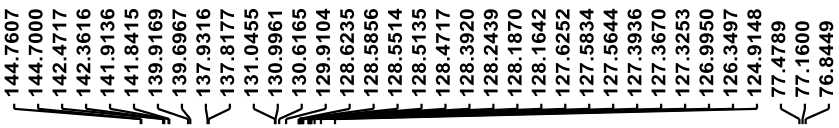

7.5182
7.5135
7.5026
7.4976
7.3659
7.3608
7.3495
7.3445
7.3097
7.2919
7.2768
7.2727
7.2222
7.2072
7.2037
7.1890
7.1802
7.1761
7.1705
7.1592
7.0535
7.0485
7.0328
7.0178
7.0128
6.2248
6.1987
3.5773
3.5600
3.5512
3.5428
3.5340
3.5255
3.5171
3.4998
1.3794
1.3622
0.0000
Br
Me
Br
3af
2.03
2.06
2.06
3.08
4.07
1.00
1.03
3.07
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)

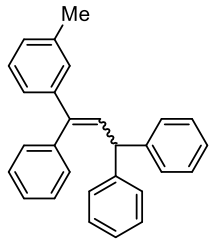

145.748
140.889
138.505
138.216
135.282
131.748
131.581
131.383
128.977
128.768
126.949
126.369
121.608
121.396
77.475
77.160
76.841
39.590
22.530
Br
Me
Br
3af
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.3658
7.3558
7.3511
7.3467
7.3432
7.3376
7.3326
7.3304
7.3257
7.3216
7.3153
7.3128
7.3012
7.2981
7.2946
7.2909
7.2855
7.2808
7.2771
7.2658
7.2617
7.2573
7.2495
7.2451
7.2303
7.2219
7.2194
7.2122
7.2075
7.1996
7.1933
7.1871
7.1733
7.1683
7.1620
7.1570
7.1532
7.1504
7.1413
7.1366
7.1313
7.0820
7.0736
7.0689
7.0638
7.0532
7.0488
7.0431
6.5195
6.5160
6.4928
6.4897
4.8560
4.8296
4.8089
4.7823
2.3806
2.3129
-0.0000
Me
3ah
18.97
5.60
17.16
3.03
2.04
2.43
1.44
1.00
4.10
3.01
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)

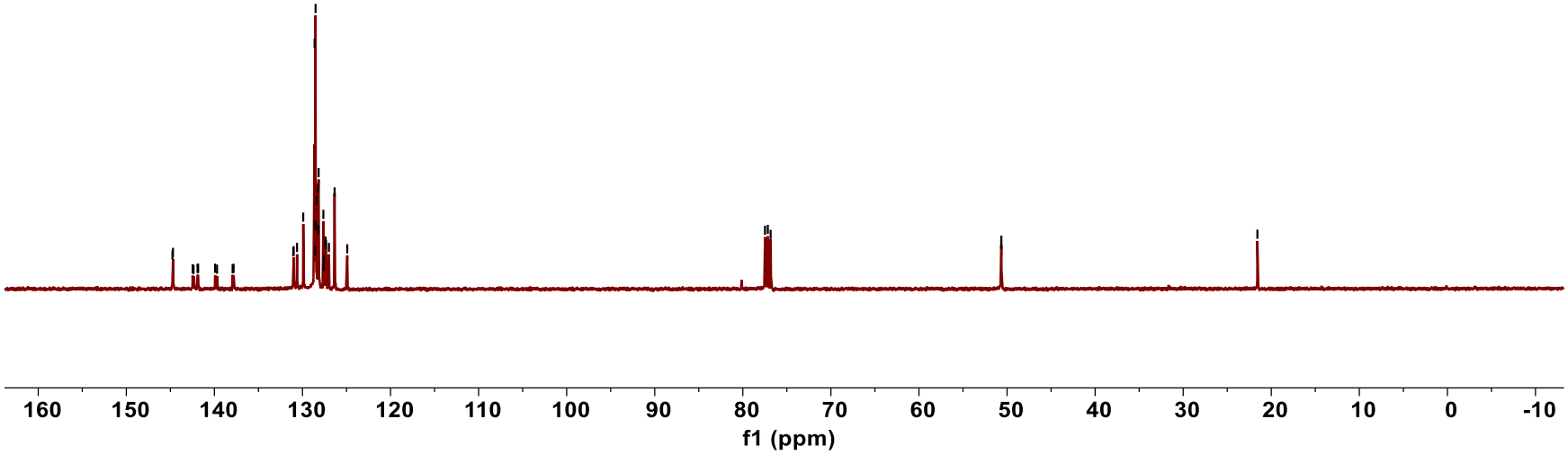

144.758
142.629
141.687
141.592
139.987
139.584
137.185
137.048
136.805
130.967
130.314
129.912
129.832
129.118
128.974
128.614
128.549
128.519
128.503
128.386
128.223
127.664
127.497
127.349
127.308
126.340
77.479
77.160
76.841
50.672
50.627
21.412
21.215
Me
3ah
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

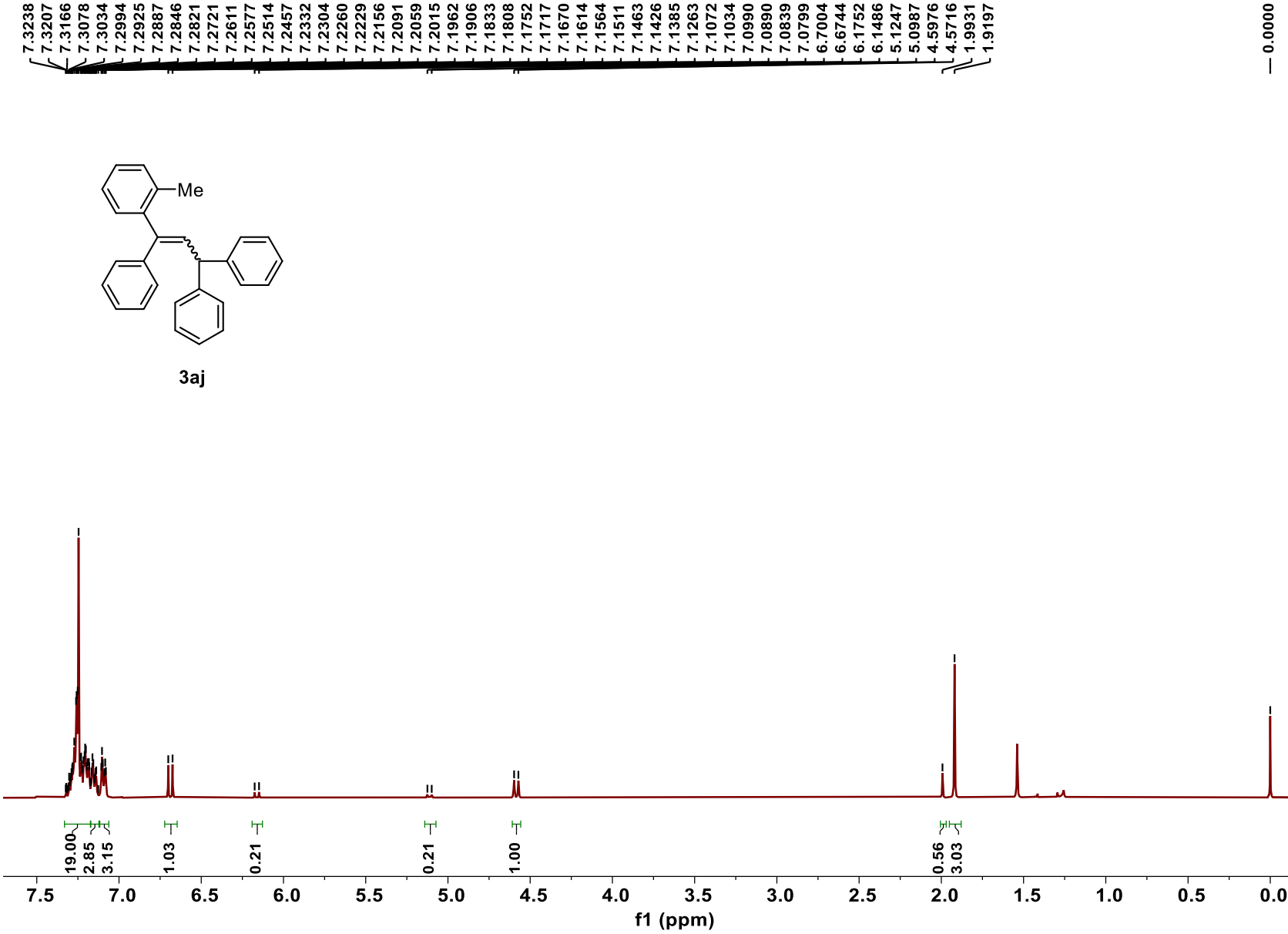

7.3710
7.3663
7.3603
7.3493
7.3453
7.3415
7.3359
7.3312
7.3271
7.3227
7.3180
7.3120
7.3004
7.2951
7.2916
7.2816
7.2766
7.2741
7.2572
7.2521
7.2421
7.2371
7.2324
7.2283
7.2230
7.2180
7.2098
7.2054
7.2017
7.1932
7.1872
7.1816
7.1769
7.1697
7.1650
7.1587
7.1534
7.1468
7.1405
7.1345
7.1164
7.0906
7.0853
7.0803
7.0417
7.0373
7.0235
7.0192
6.9869
6.9822
6.9649
6.9611
6.5413
6.5359
6.5152
6.5096
4.8270
4.8176
4.8006
4.7909
2.3046
2.2764
-0.0002
Me
3ai
39.01
2.02
2.08
2.07
1.01
1.00
1.07
1.00
3.05
3.01
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)

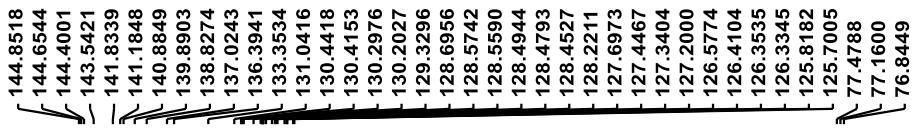

144.7607
144.7000
142.4717
142.3616
141.9136
141.8415
139.9169
139.6967
137.9316
137.8177
131.0455
130.9961
130.6165
129.9104
128.6235
128.5856
128.5514
128.5135
128.4717
128.3920
128.2439
128.1870
128.1642
127.6252
127.5834
127.5644
127.3936
127.3670
127.3253
126.9950
126.3497
124.9148
77.4789
77.1600
76.8449
50.6633
50.6443
21.5739

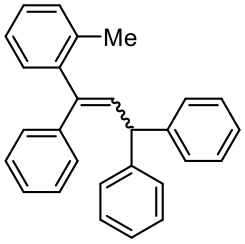

Me

3ai

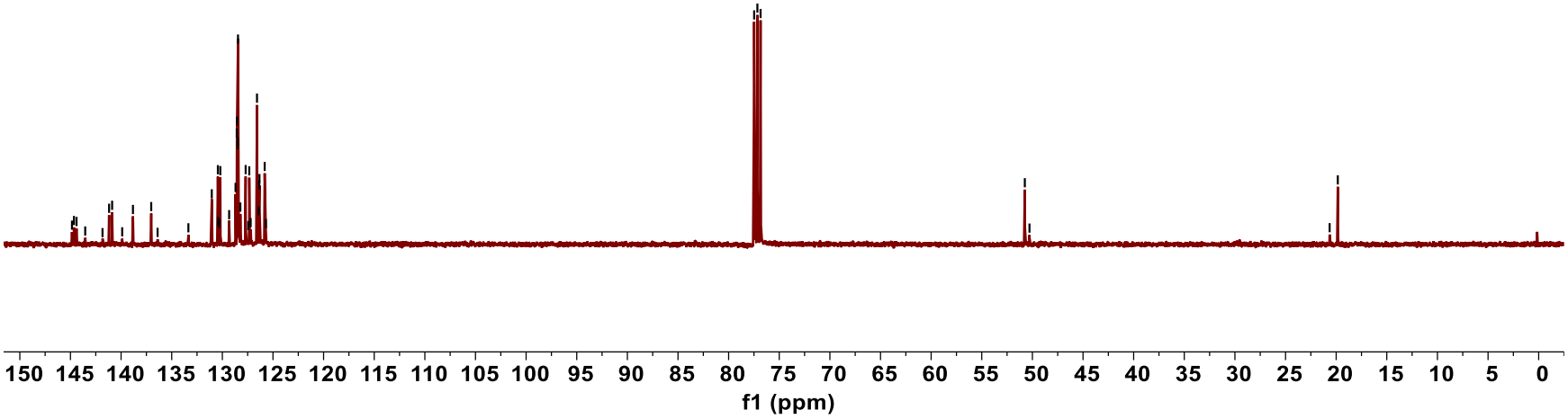

160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

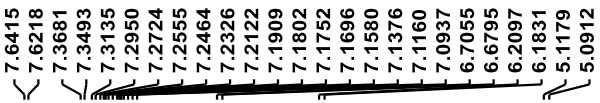

7.3238
7.3207
7.3166
7.3078
7.3034
7.2994
7.2925
7.2887
7.2846
7.2821
7.2721
7.2611
7.2577
7.2514
7.2457
7.2332
7.2304
7.2260
7.2229
7.2156
7.2091
7.2059
7.2015
7.1962
7.1906
7.1833
7.1808
7.1752
7.1717
7.1670
7.1614
7.1564
7.1511
7.1463
7.1426
7.1385
7.1263
7.1072
7.1034
7.0990
7.0890
7.0839
7.0799
6.7004
6.6744
6.1752
6.1486
5.1247
5.0987
4.5976
4.5716
1.9931
1.9197
0.0000
Me
3aj
19.00
2.85
3.15
1.03
0.21
0.21
1.00
0.56
3.03
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)

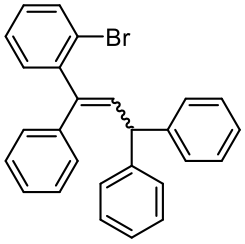

144.8518
144.6544
144.4001
143.5421
141.8339
141.1848
140.8849
139.8903
138.8274
137.0243
136.3941
133.3534
131.0416
130.4418
130.4153
130.2976
130.2027
129.3296
128.6956
128.5742
128.5590
128.4944
128.4793
128.4527
128.2211
127.6973
127.4467
127.3404
127.2000
126.5774
126.4104
126.3535
126.3345
125.8182
125.7005
77.4788
77.1600
76.8449
50.7468
50.2875
20.6438
19.8390


Me

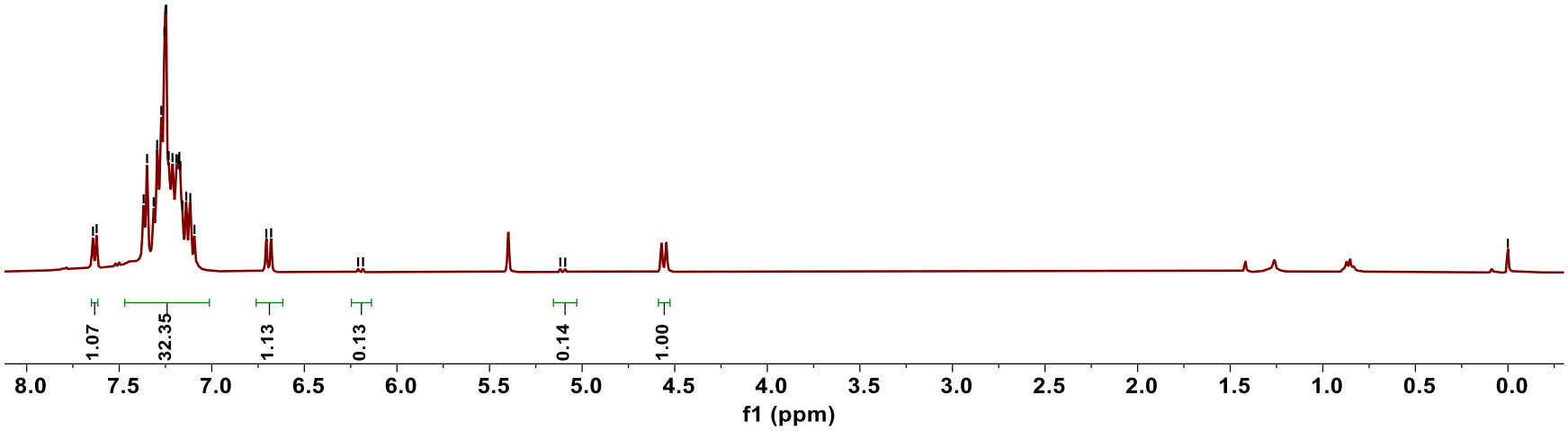
3aj

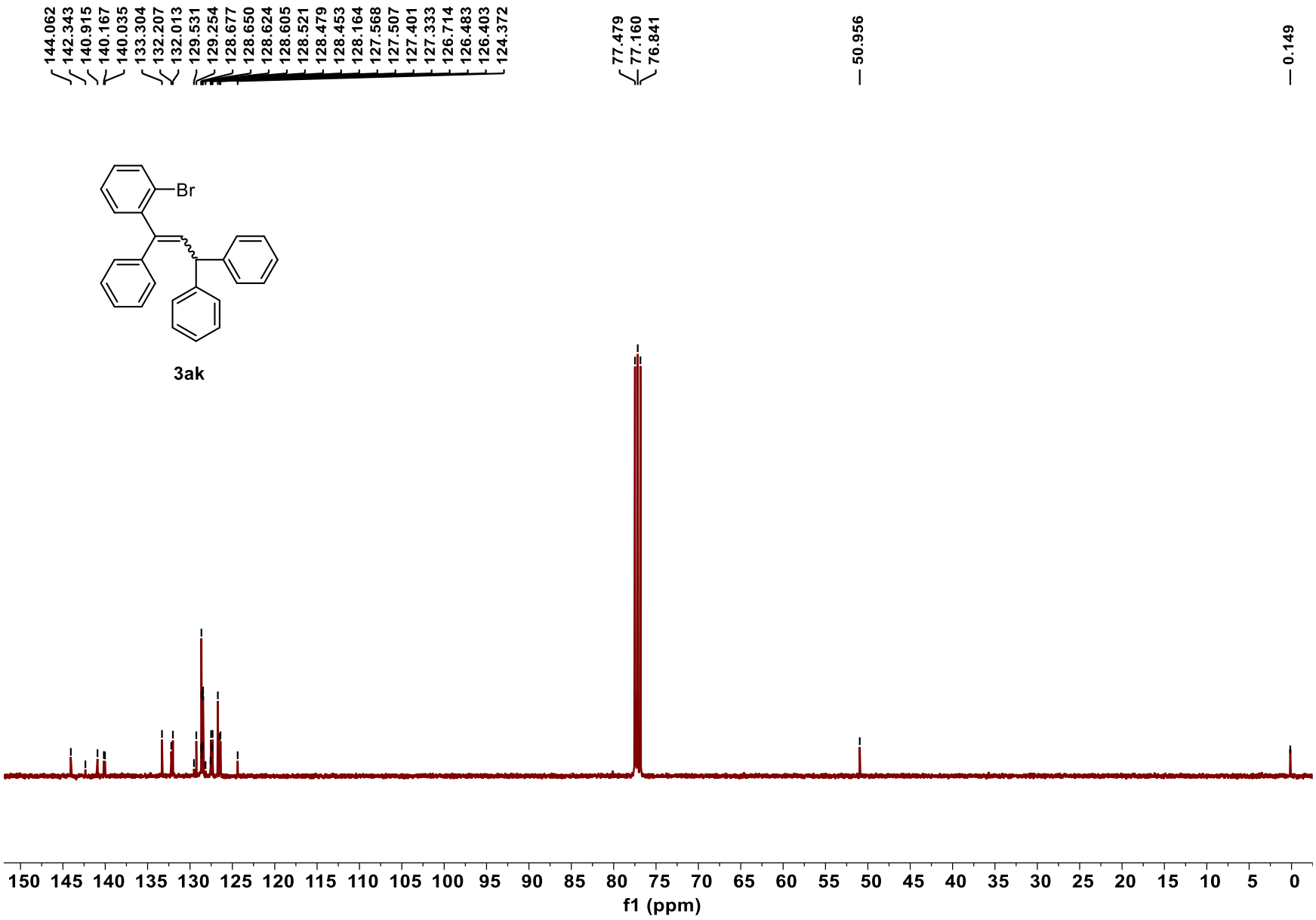

150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

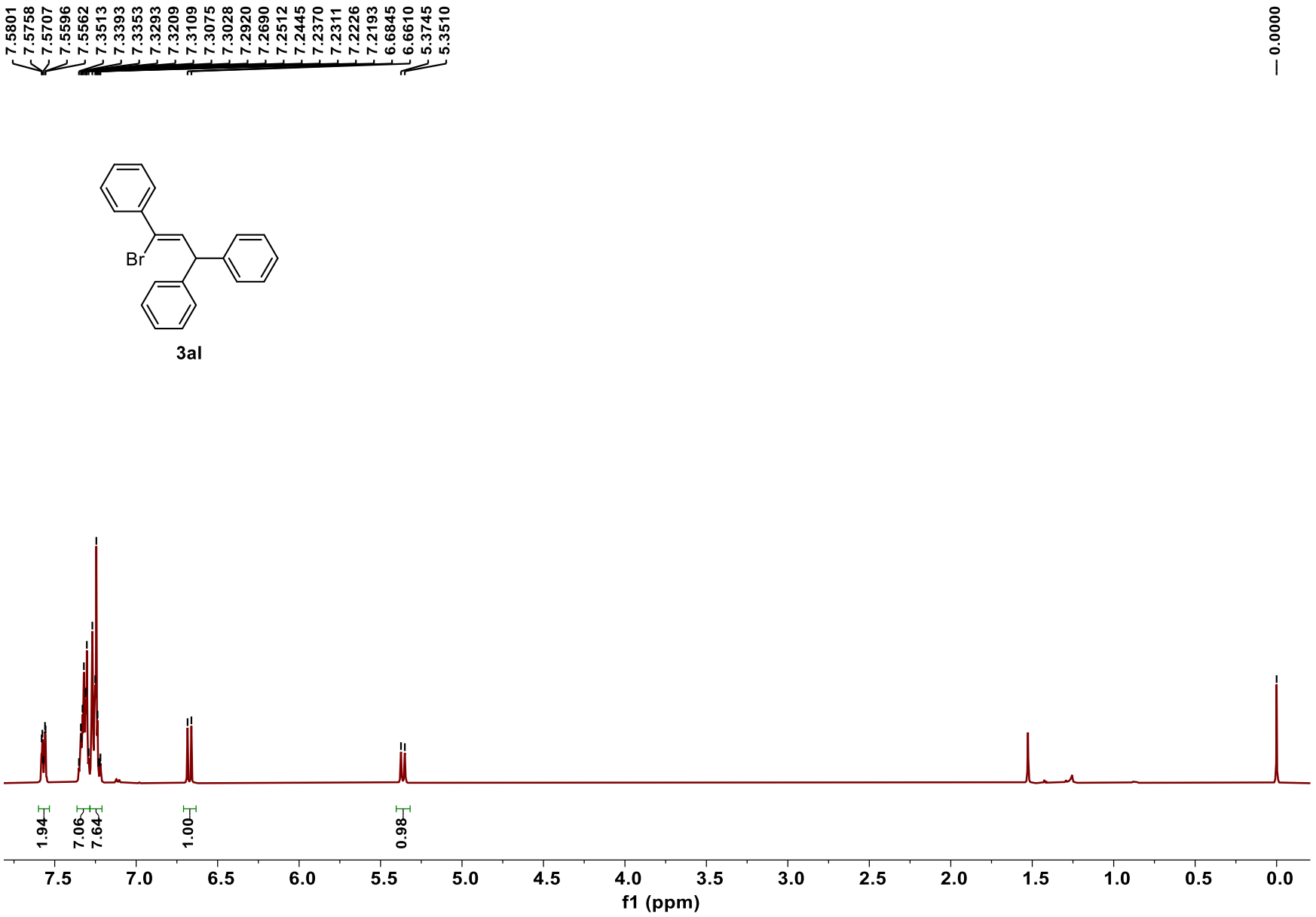

7.6415
7.6218
7.3681
7.3493
7.3135
7.2950
7.2724
7.2555
7.2464
7.2326
7.2122
7.1909
7.1802
7.1752
7.1696
7.1580
7.1376
7.1160
7.0937
6.7055
6.6795
6.2097
6.1831
5.1179
5.0912
0.0007

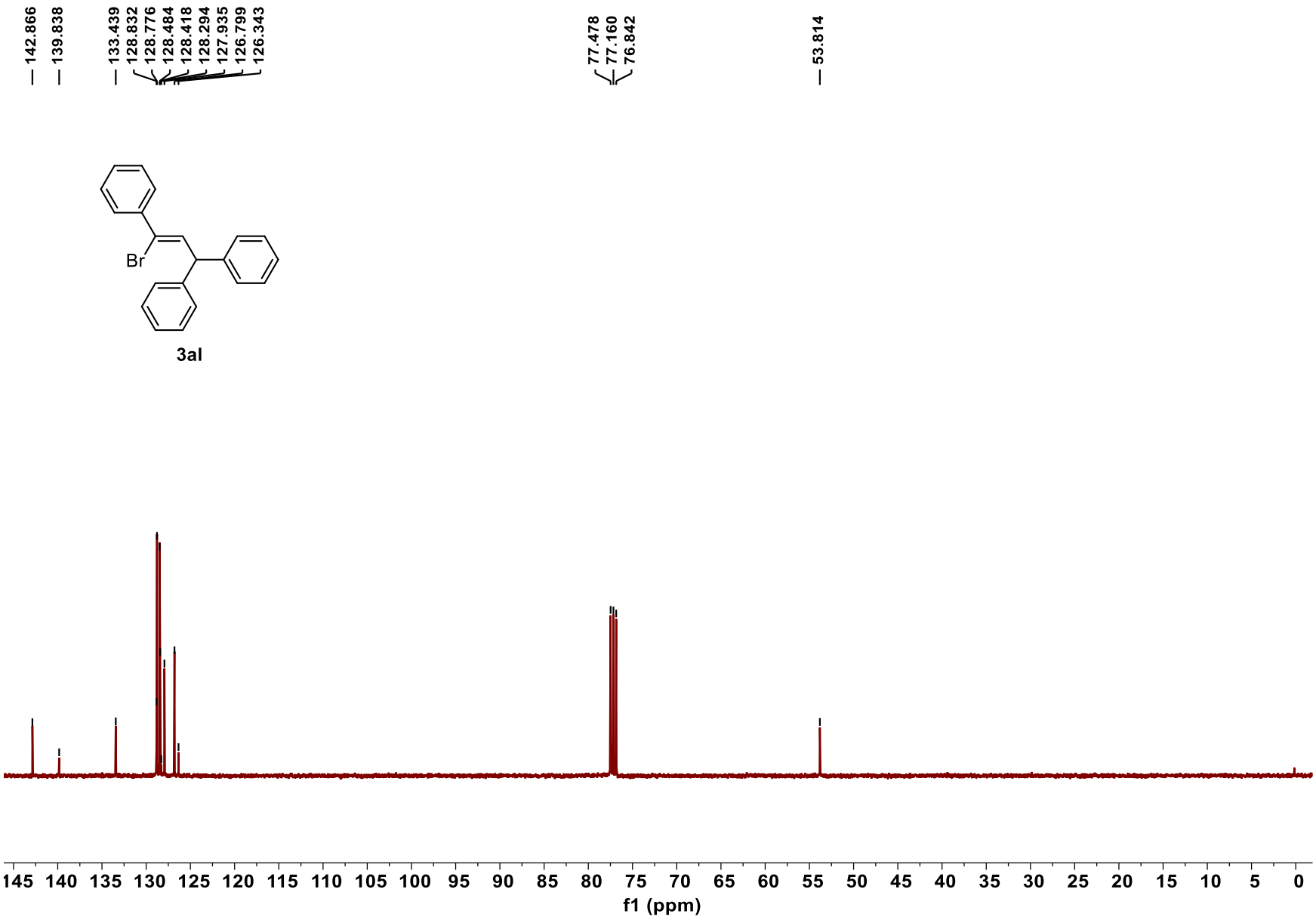

Br

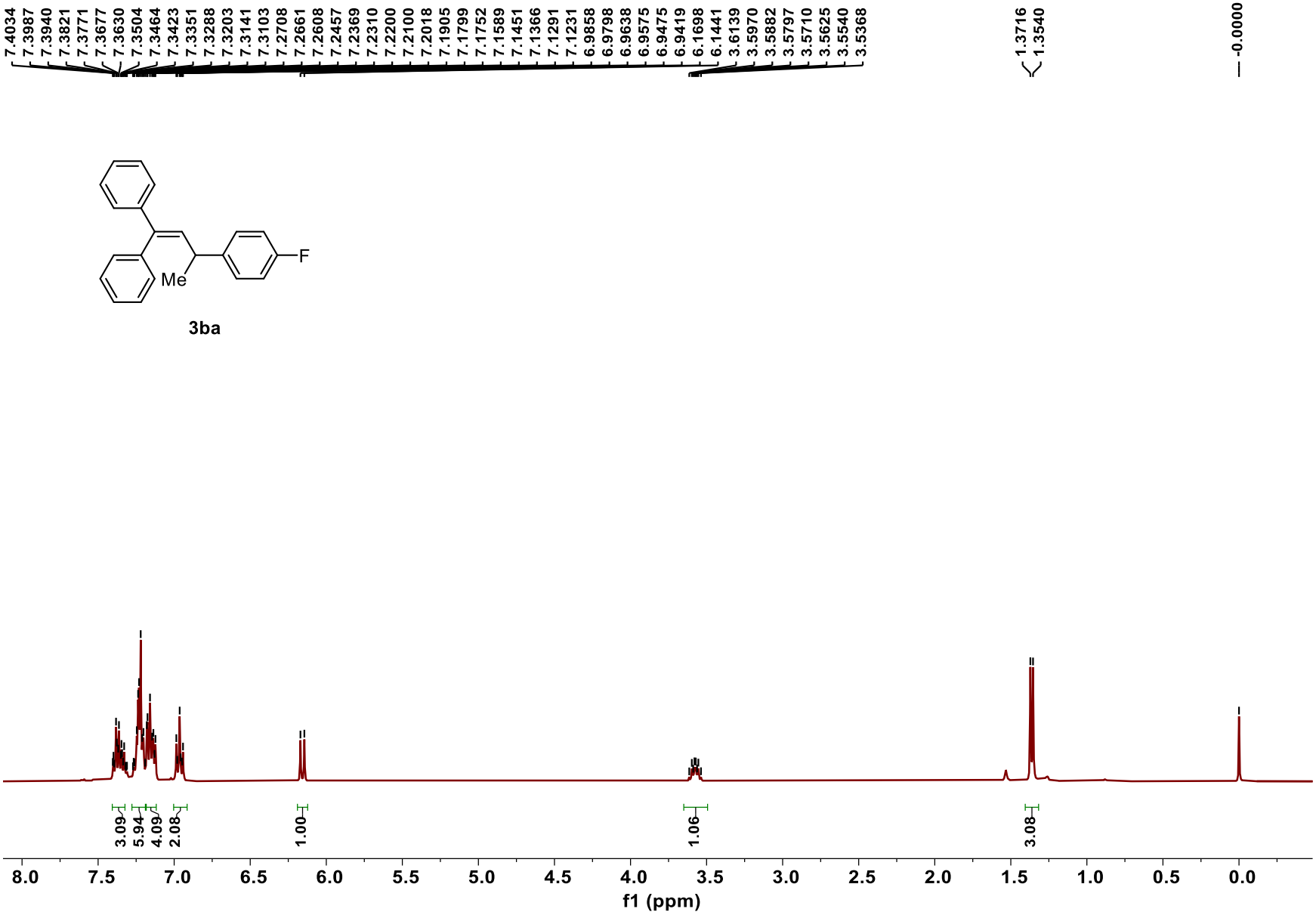
3ak


1.07
32.35
1.13
0.13
0.14
1.00
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)

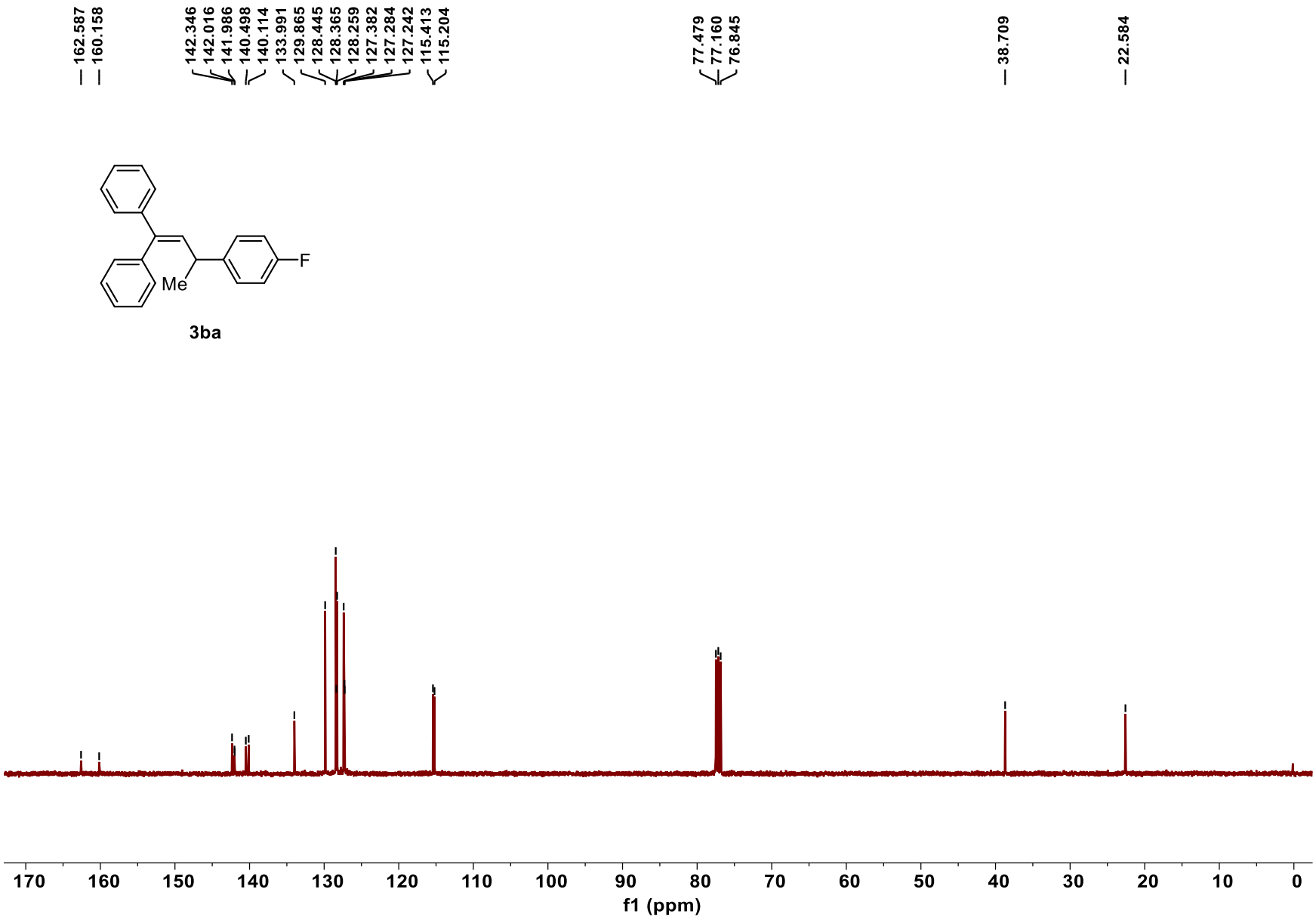

144.062
142.343
140.915
140.167
140.035
133.304
132.207
132.013
129.531
129.254
128.677
128.650
128.624
128.605
128.521
128.479
128.453
128.164
127.568
127.507
127.401
127.333
126.714
126.483
126.403
124.372
77.479
77.160
76.841
50.956
0.149
Br
3ak
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

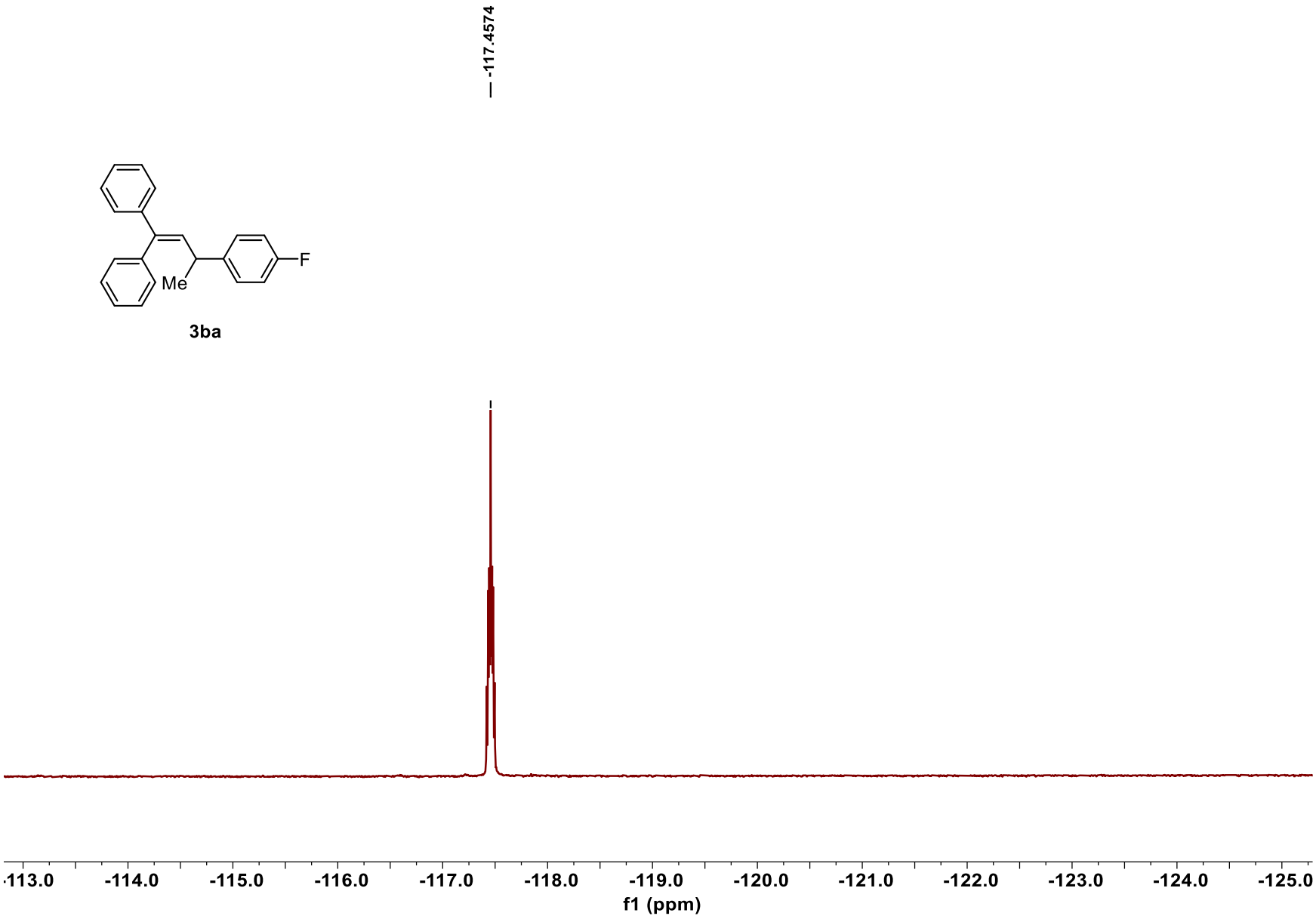

7.5801
7.5758
7.5707
7.5596
7.5562
7.3513
7.3393
7.3353
7.3293
7.3209
7.3109
7.3075
7.3028
7.2920
7.2690
7.2512
7.2445
7.2370
7.2311
7.2226
7.2193
6.6845
6.6610
5.3745
5.3510
0.0000
Br
3al
1.94
7.06
7.64
1.00
0.98
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

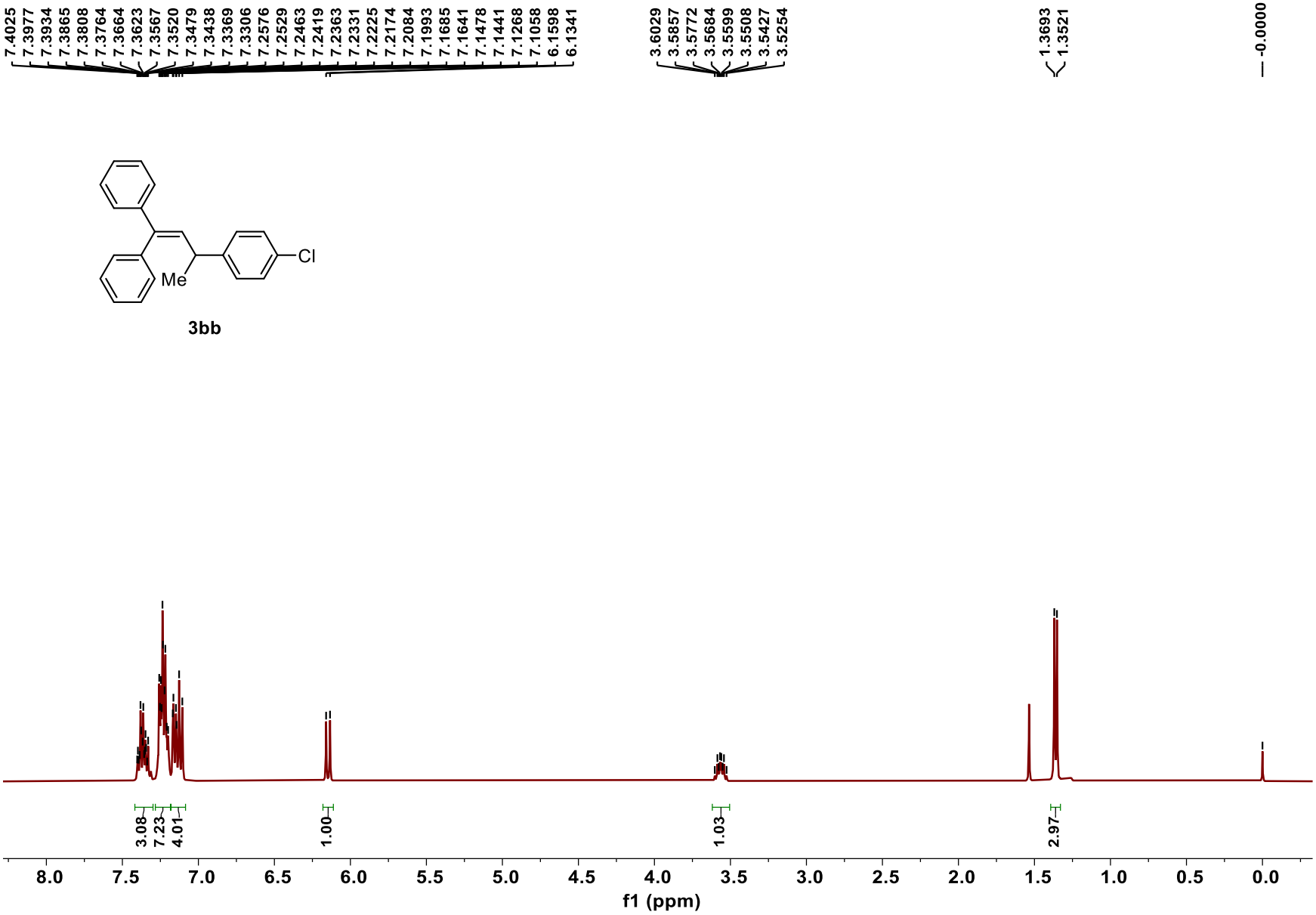

142.866
139.838
133.439
128.832
128.776
128.484
128.418
128.294
127.935
126.799
126.343
77.478
77.160
76.842
53.814
Br
3al
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

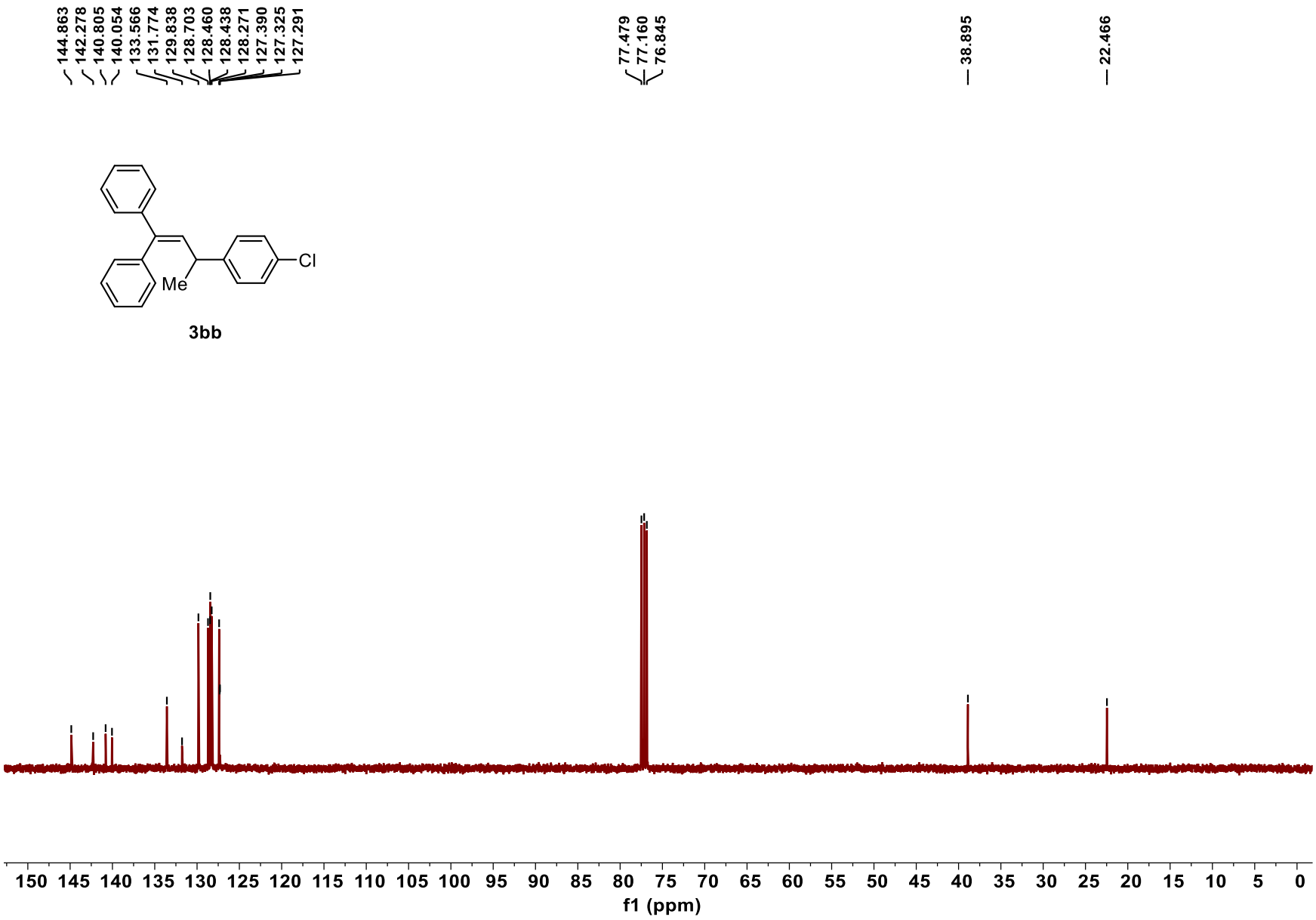

7.4034
7.3987
7.3940
7.3821
7.3771
7.3677
7.3630
7.3504
7.3464
7.3423
7.3351
7.3288
7.3203
7.3141
7.3103
7.2708
7.2661
7.2608
7.2457
7.2369
7.2310
7.2200
7.2100
7.2018
7.1905
7.1799
7.1752
7.1589
7.1451
7.1366
7.1291
7.1231
6.9858
6.9798
6.9638
6.9575
6.9475
6.9419
6.1698
6.1441
3.6139
3.5970
3.5882
3.5797
3.5710
3.5625
3.5540
3.5368
1.3716
1.3540
-0.0000
F
Me
3.09
5.94
4.09
2.08
1.00
1.06
3.08
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)

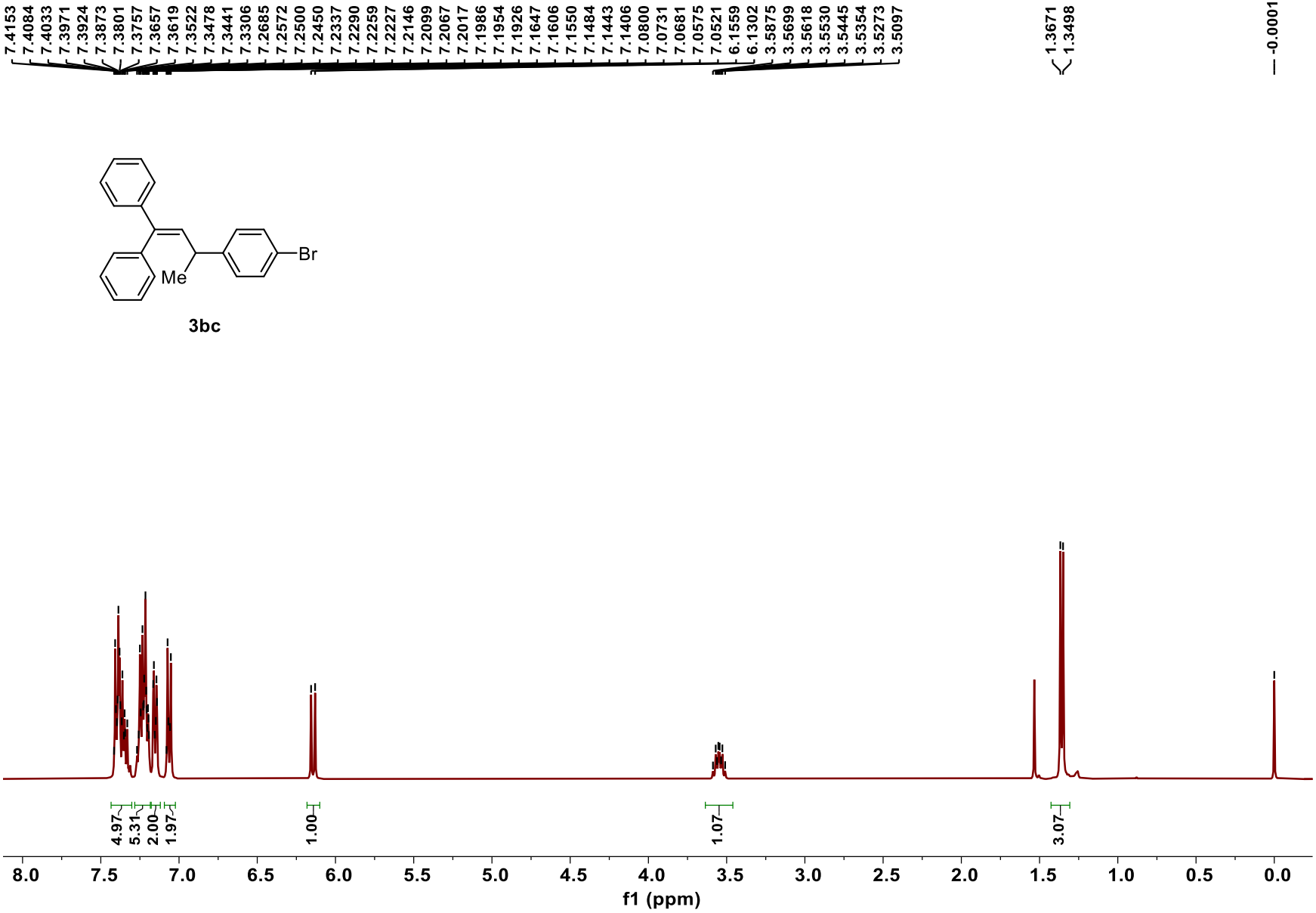
3ba

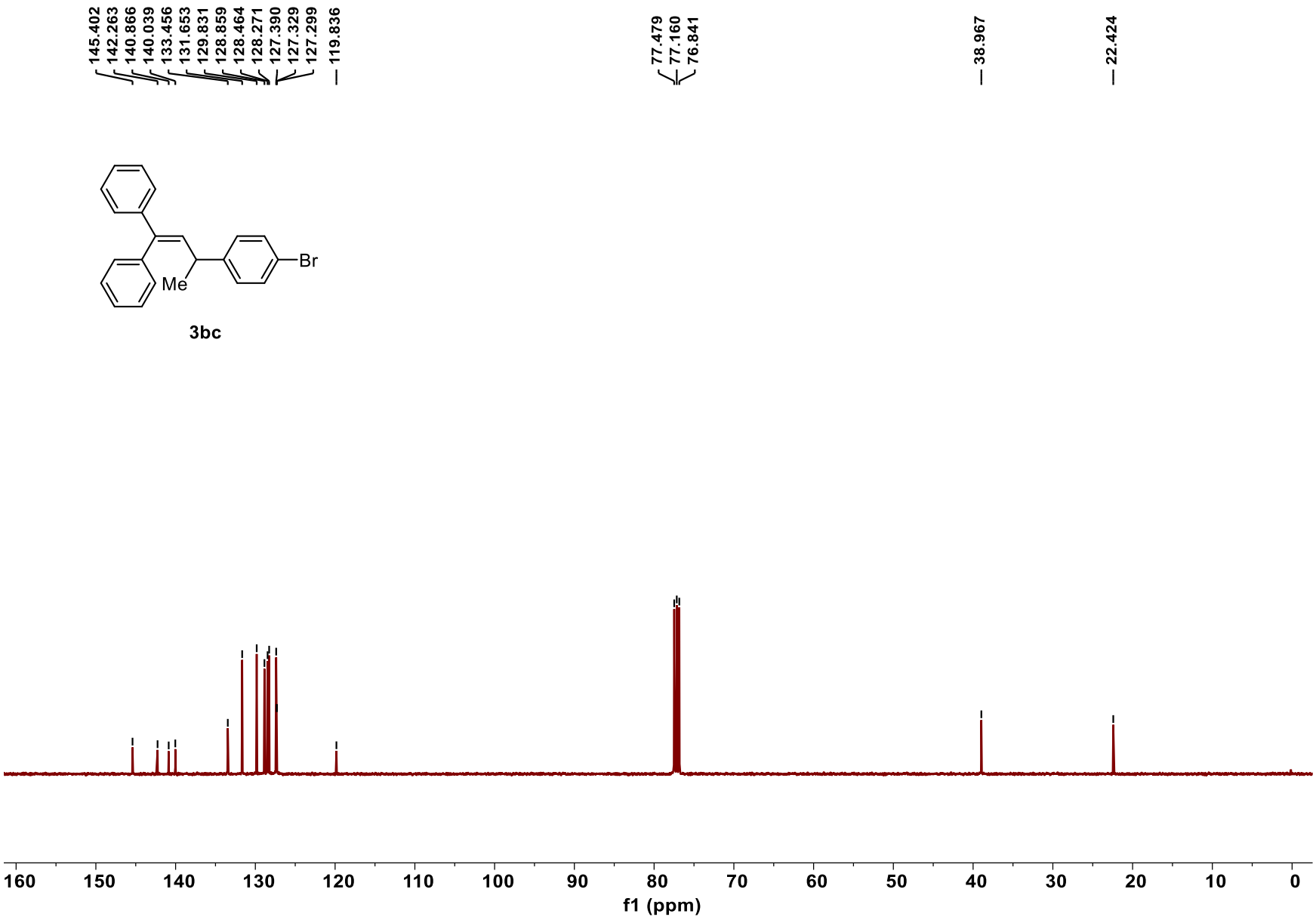

162.587
160.158
142.346
142.016
141.986
140.498
140.114
133.991
129.865
128.445
128.365
128.259
127.382
127.284
127.242
115.413
115.204
77.479
77.160
76.845
38.709
22.584
F
Me
3ba
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

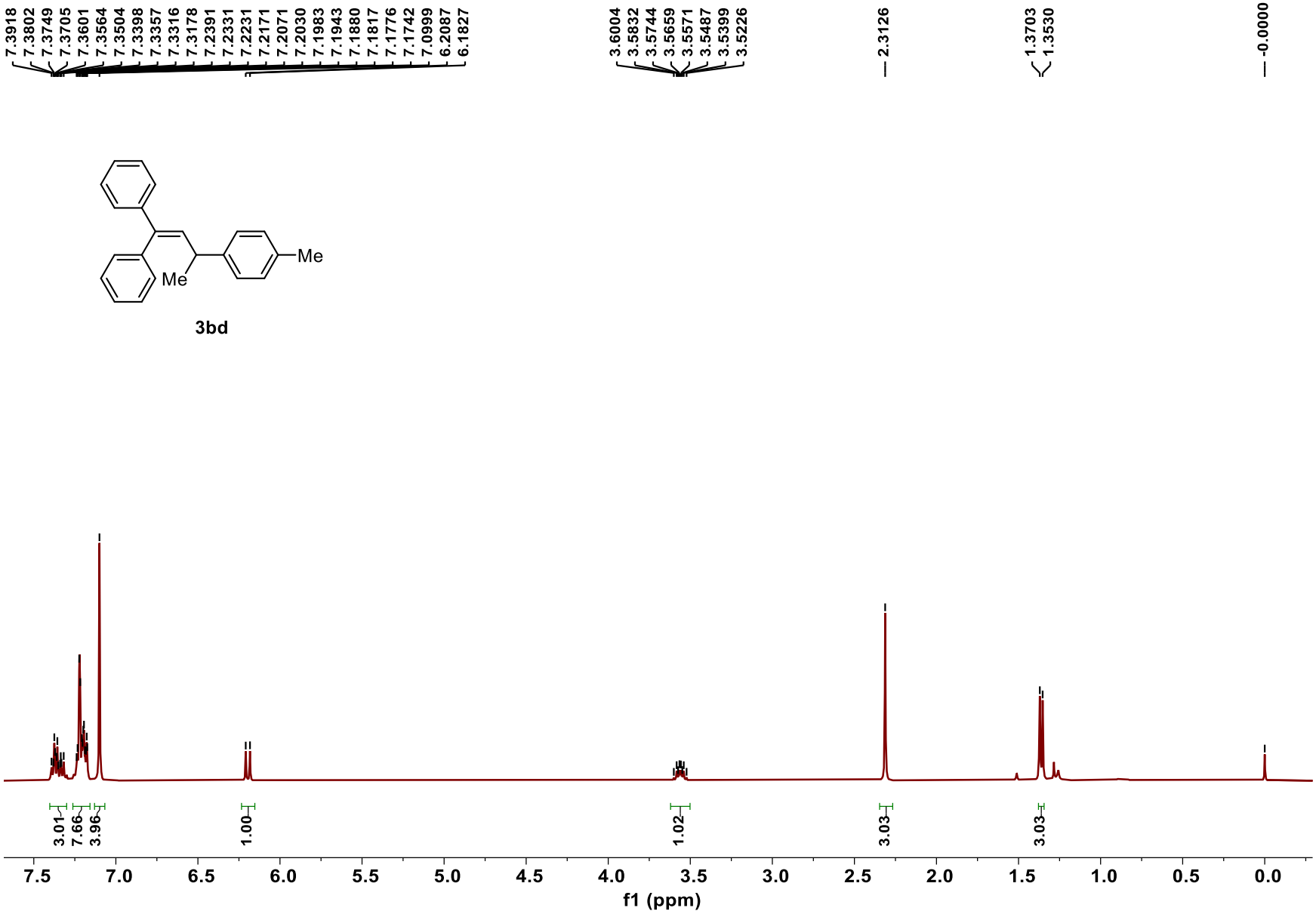

-117.4574
F
Me
3ba
-113.0
-114.0
-115.0
-116.0
-117.0
-118.0
-119.0
-120.0
-121.0
-122.0
-123.0
-124.0
-125.0
f1 (ppm)

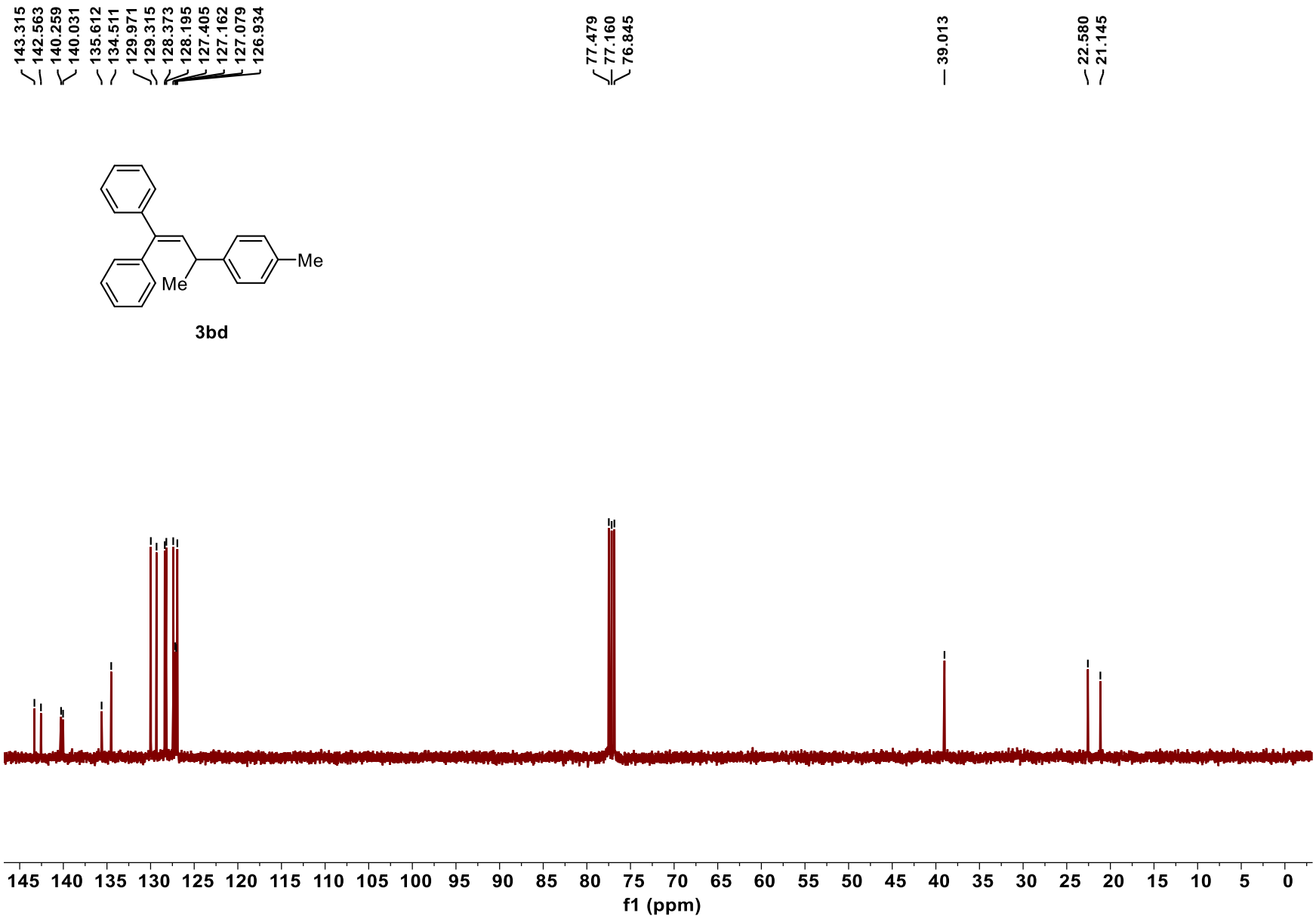

7.4025
7.3977
7.3934
7.3865
7.3808
7.3764
7.3664
7.3623
7.3567
7.3520
7.3479
7.3438
7.3369
7.3306
7.2576
7.2529
7.2463
7.2419
7.2363
7.2331
7.2225
7.2174
7.2084
7.1993
7.1685
7.1641
7.1478
7.1441
7.1268
7.1058
6.1598
6.1341
3.6029
3.5857
3.5772
3.5684
3.5599
3.5508
3.5427
3.5254
1.3693
1.3521
-0.0000
Cl
Me
3bb
3.08
7.23
4.01
1.00
1.03
2.97
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)

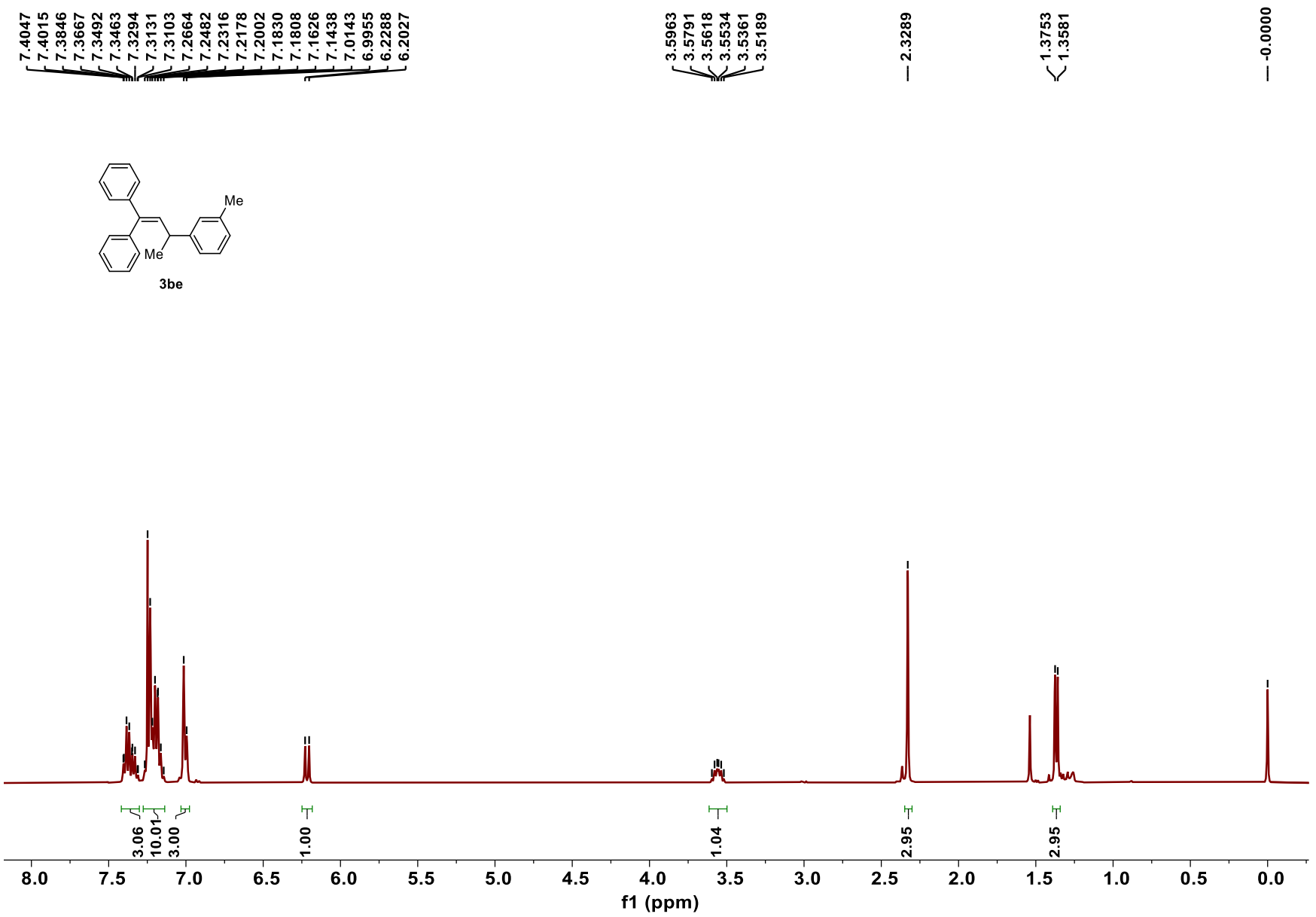

144.863
142.278
140.805
140.054
133.566
131.774
129.838
128.703
128.460
128.438
128.271
127.390
127.325
127.291
77.479
77.160
76.845
38.895
22.466
Cl
Me
3bb
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)


7.4153
7.4084
7.4033
7.3971
7.3924
7.3873
7.3801
7.3757
7.3657
7.3619
7.3522
7.3478
7.3441
7.3306
7.2685
7.2572
7.2500
7.2450
7.2337
7.2290
7.2259
7.2227
7.2146
7.2099
7.2067
7.2017
7.1986
7.1954
7.1926
7.1647
7.1606
7.1550
7.1484
7.1443
7.1406
7.0800
7.0731
7.0681
7.0575
7.0521
6.1559
6.1302
3.5875
3.5699
3.5618
3.5530
3.5445
3.5354
3.5273
3.5097
1.3671
1.3498
-0.0001
Br
Me
3bc
4.97
5.31
2.00
1.97
1.00
1.07
3.07
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)

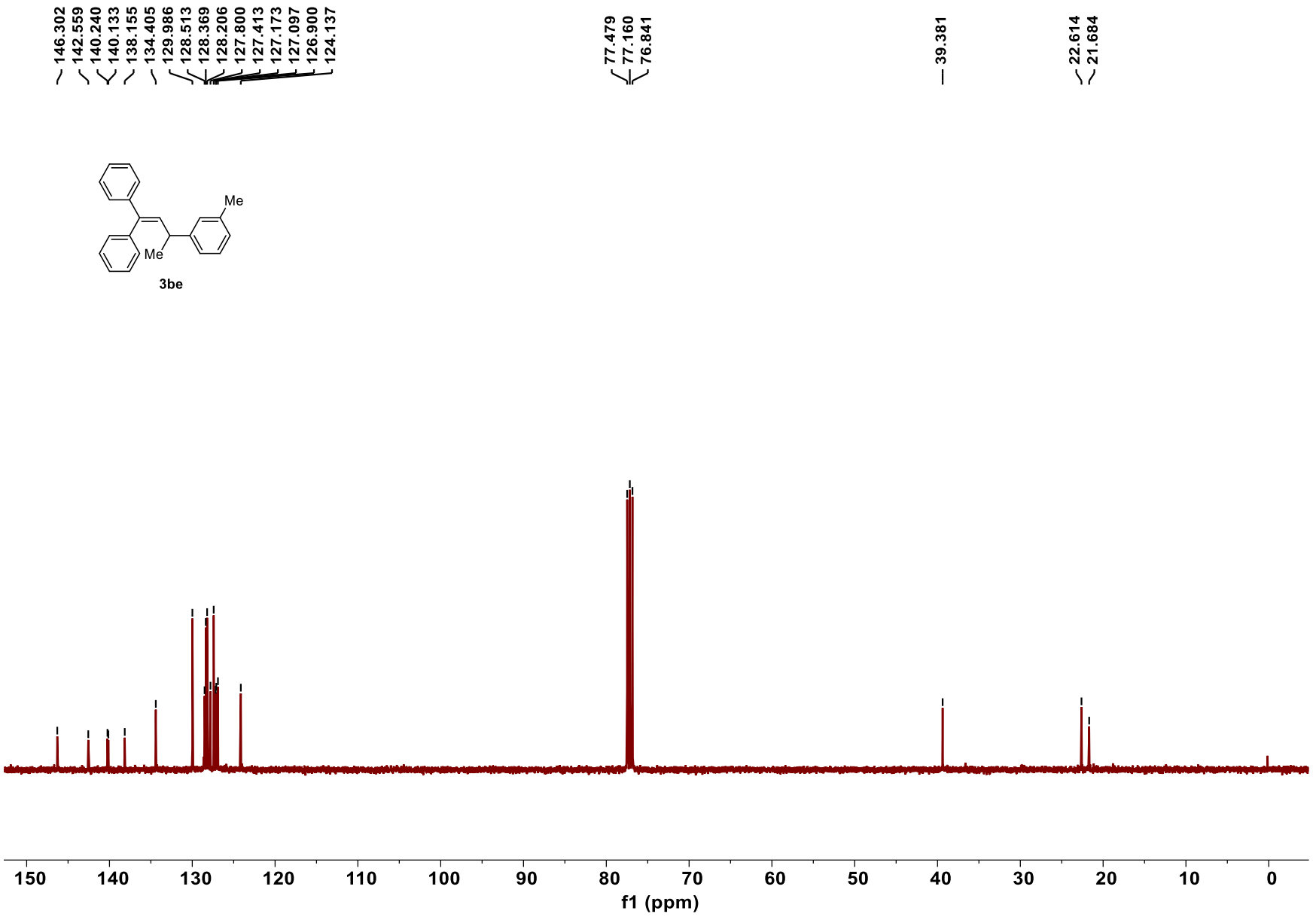

145.402
142.263
140.866
140.039
133.456
131.653
129.831
128.859
128.464
128.271
127.390
127.329
127.299
119.836
77.479
77.160
76.841
38.967
22.424
Br
Me
3bc
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

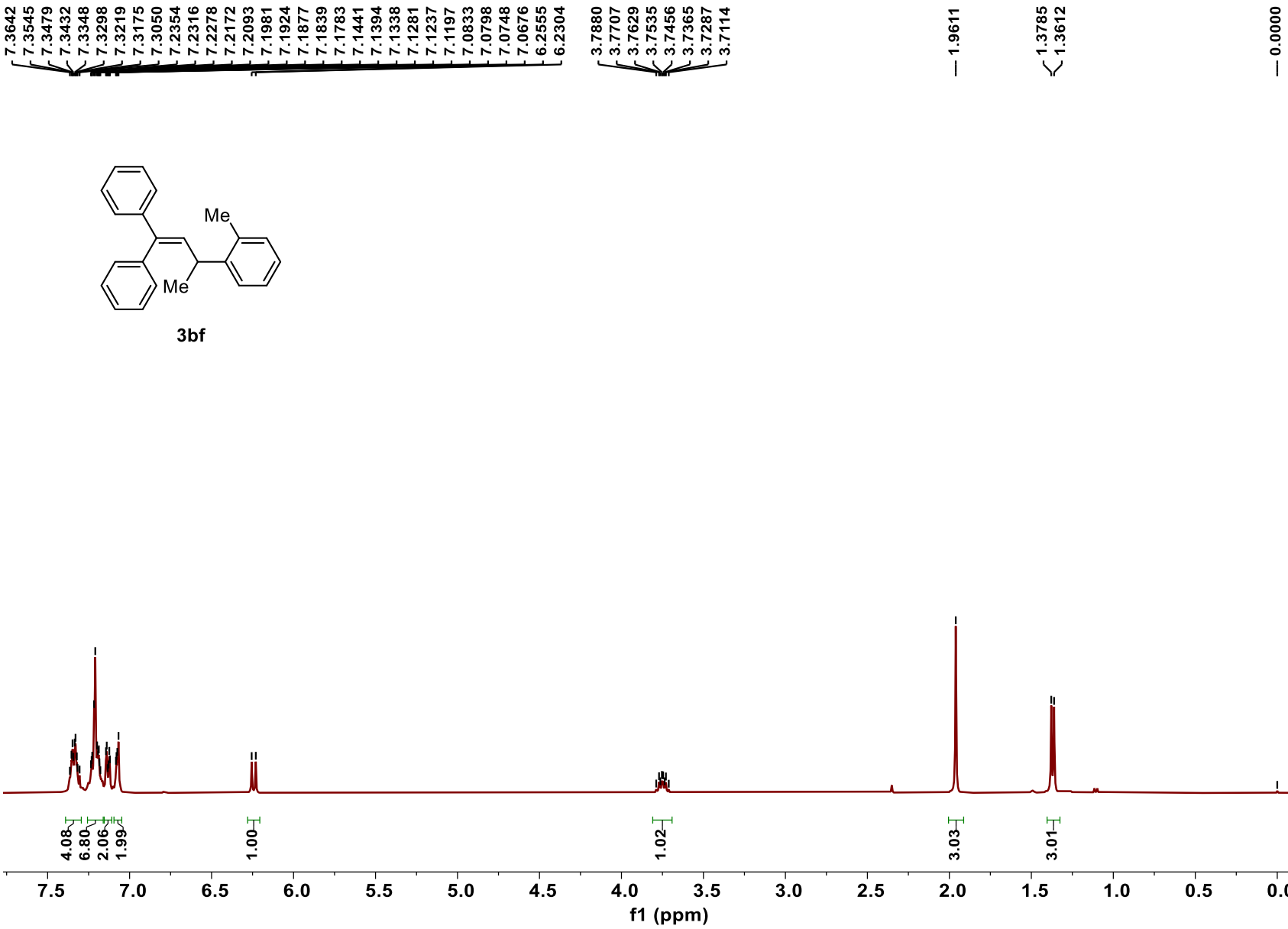

7.3918
7.3802
7.3749
7.3705
7.3601
7.3564
7.3504
7.3398
7.3357
7.3316
7.3178
7.2391
7.2331
7.2231
7.2171
7.2071
7.2030
7.1983
7.1943
7.1880
7.1817
7.1776
7.1742
7.0999
6.2087
6.1827
3.6004
3.5832
3.5744
3.5659
3.5571
3.5487
3.5399
3.5226
2.3126
1.3703
1.3530
-0.0000
Me
Me
3bd
3.01
7.66
3.96
1.00
1.02
3.03
3.03
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)

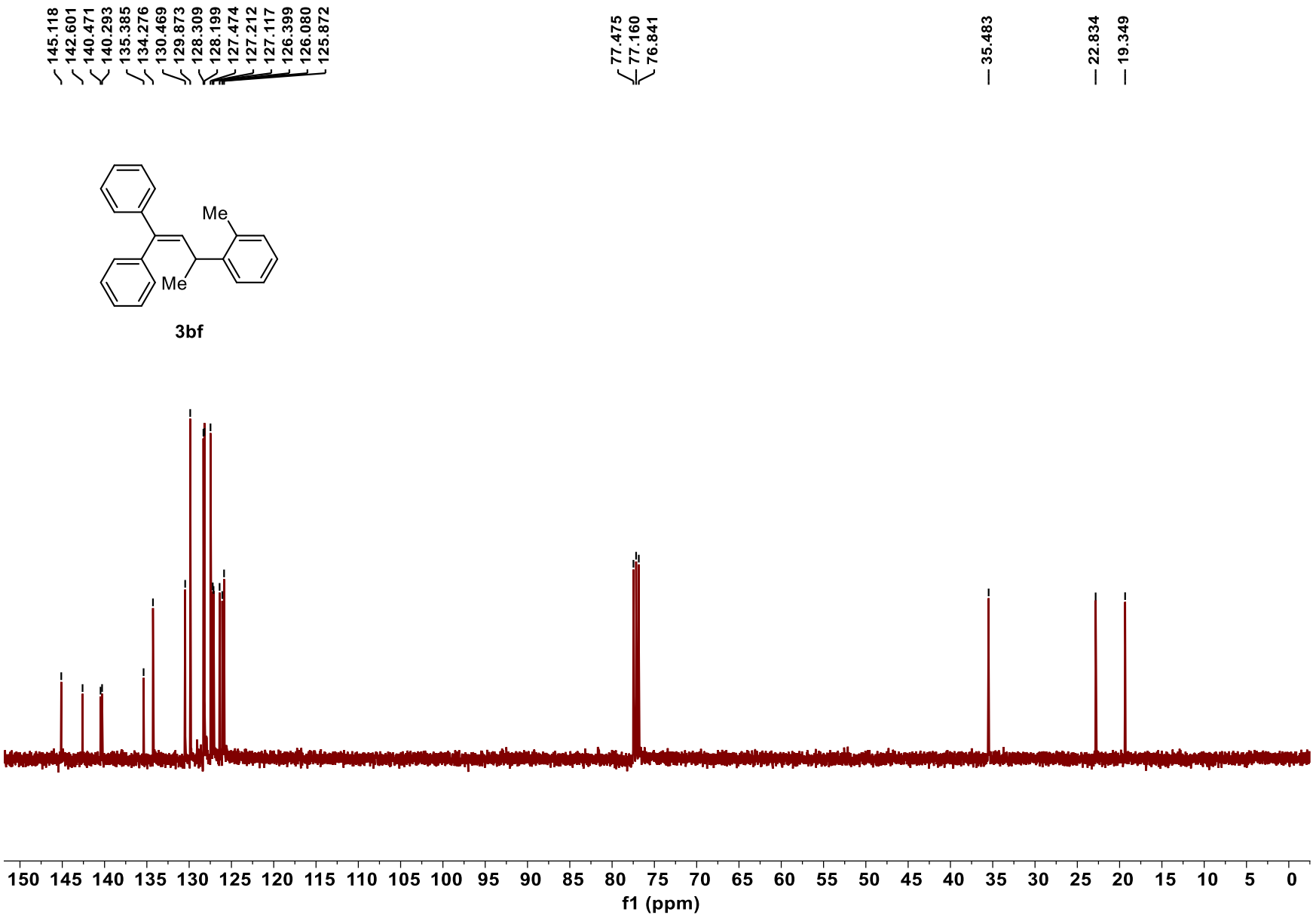

143.315
142.563
140.259
140.031
135.612
134.511
129.971
129.315
128.373
128.195
127.405
127.162
127.079
126.934
77.479
77.160
76.845
39.013
22.580
21.145
Me
Me
3bd
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

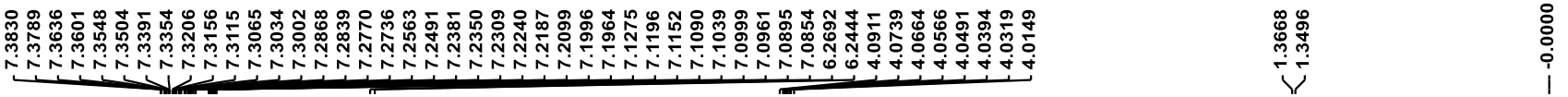

7.4047
7.4015
7.3846
7.3667
7.3492
7.3463
7.3294
7.3131
7.3103
7.2664
7.2482
7.2316
7.2178
7.2002
7.1830
7.1808
7.1626
7.1438
7.0143
6.9955
6.2288
6.2027
3.5963
3.5791
3.5618
3.5534
3.5361
3.5189
2.3289
1.3753
1.3581
-0.0000
Me
Me
3.06
10.01
3.00
1.00
1.04
2.95
2.95
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

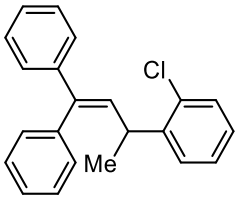
3be


146.302
142.559
140.240
140.133
138.155
134.405
129.986
128.513
128.369
128.206
127.800
127.413
127.173
127.097
126.900
124.137
77.479
77.160
76.841
39.381
22.614
21.684
Me
Me
3be
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

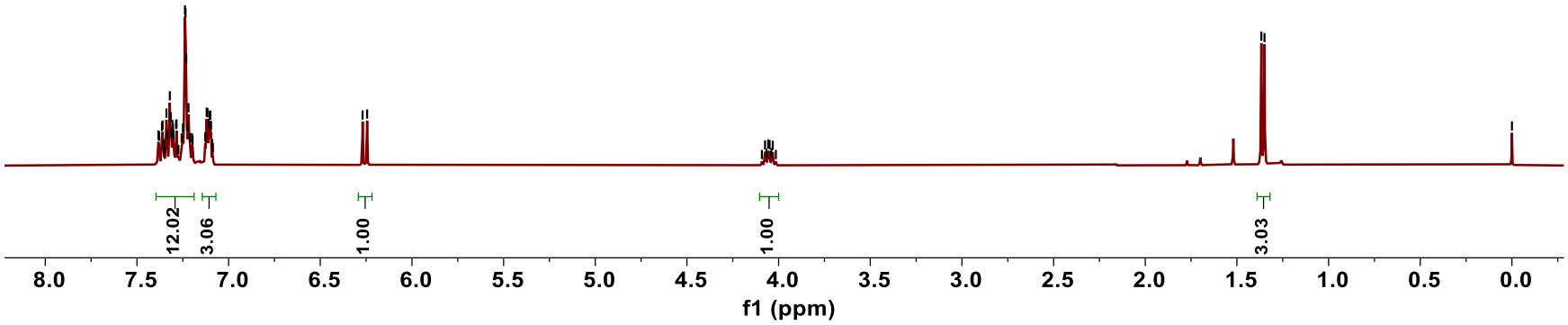

7.3642
7.3545
7.3479
7.3432
7.3348
7.3298
7.3219
7.3175
7.3050
7.2354
7.2316
7.2278
7.2172
7.2093
7.1981
7.1924
7.1877
7.1839
7.1783
7.1441
7.1394
7.1338
7.1281
7.1237
7.1197
7.0833
7.0798
7.0748
7.0676
6.2555
6.2304
3.7880
3.7707
3.7629
3.7535
3.7456
3.7365
3.7287
3.7114
1.9611
1.3785
1.3612
0.0000
Me
Me
3bf
4.08
6.80
2.06
1.99
1.00
1.02
3.03
3.01
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)

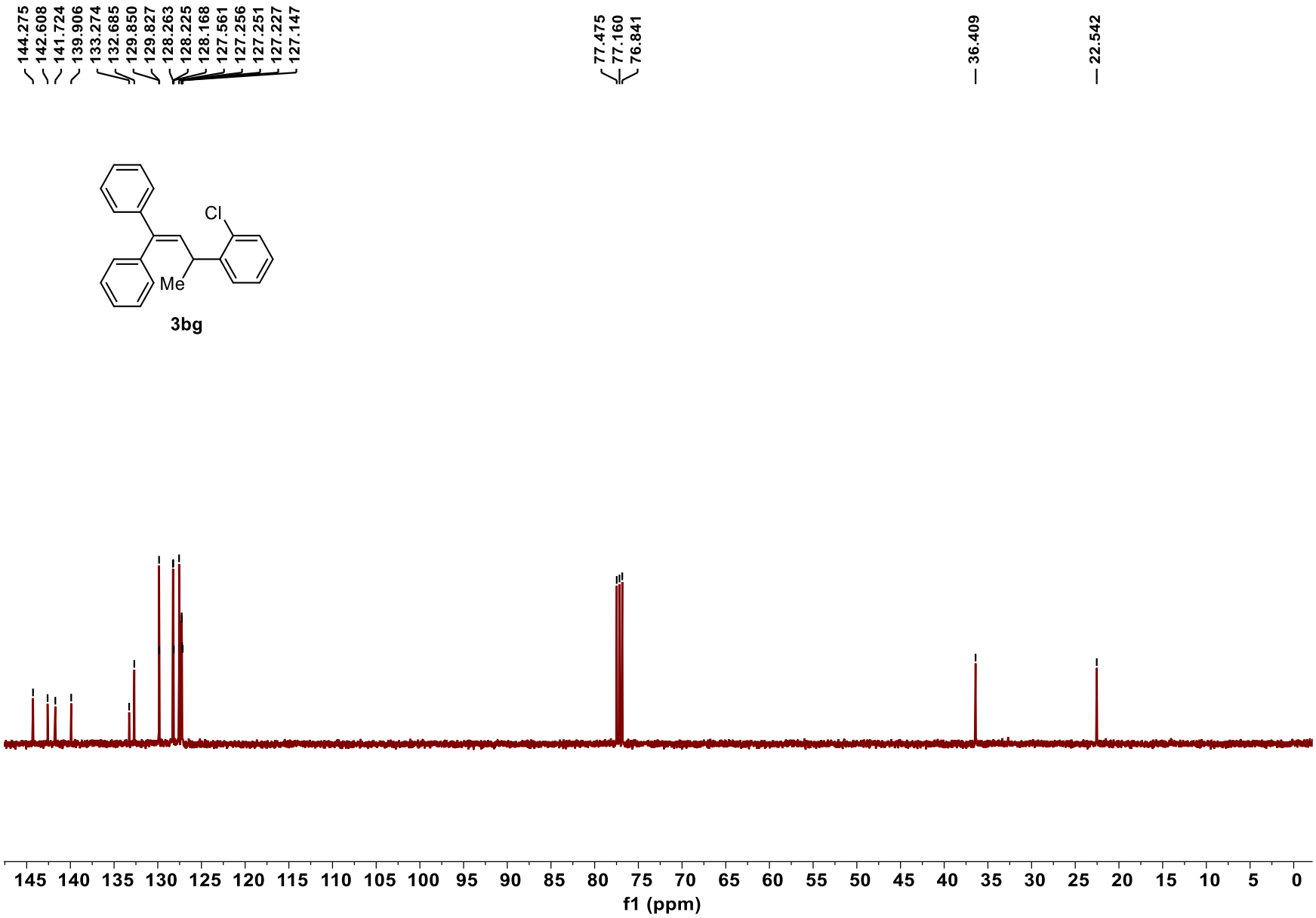

145.118
142.601
140.471
140.293
135.385
134.276
130.469
129.873
128.309
128.199
127.474
127.212
127.117
126.399
126.080
125.872
77.475
77.160
76.841
35.483
22.834
19.349
Me
Me
3bf
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

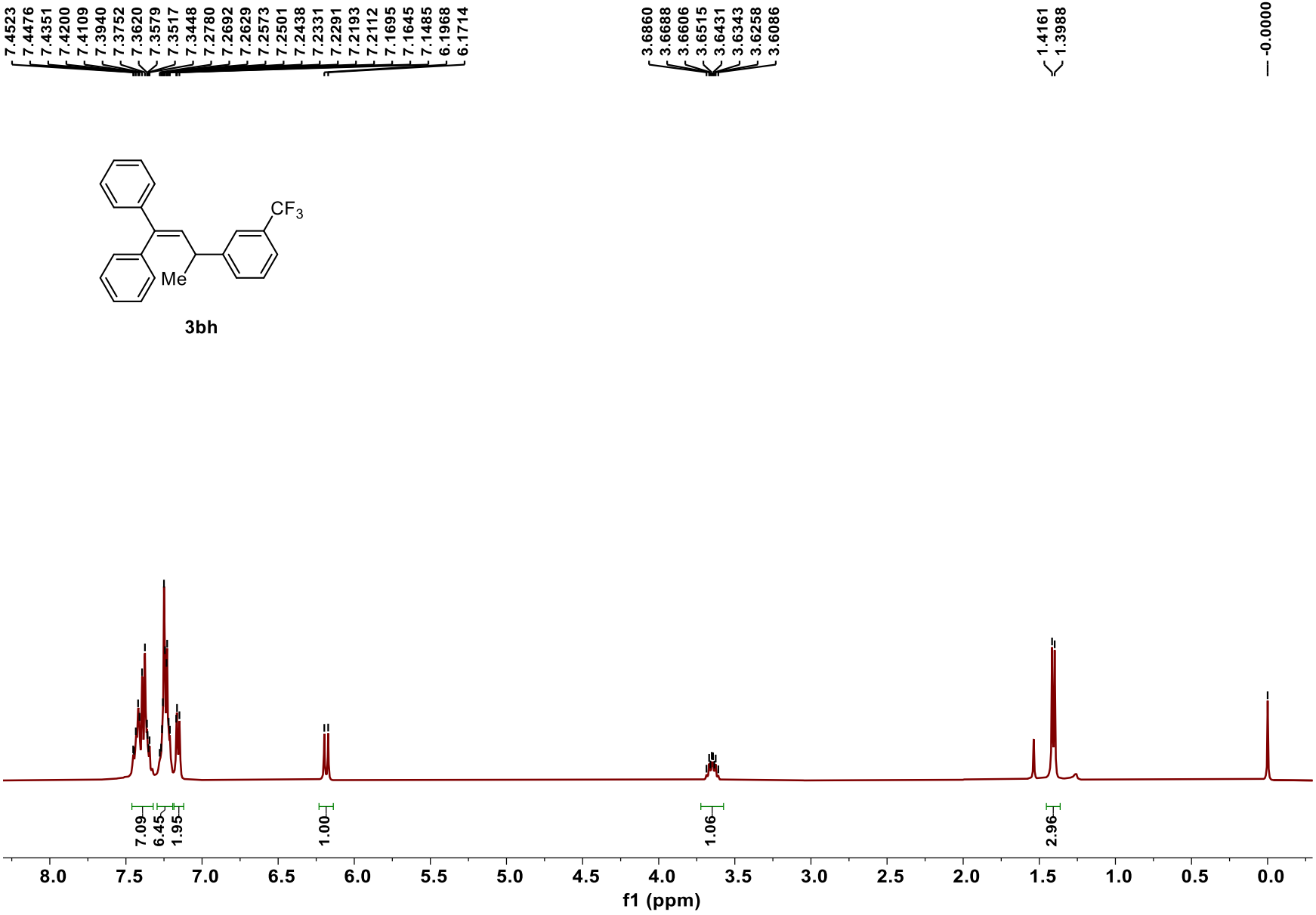

7.3830
7.3789
7.3636
7.3601
7.3548
7.3504
7.3391
7.3354
7.3206
7.3156
7.3115
7.3065
7.3034
7.3002
7.2868
7.2839
7.2770
7.2736
7.2563
7.2491
7.2381
7.2350
7.2309
7.2240
7.2187
7.2099
7.1996
7.1964
7.1275
7.1196
7.1152
7.1090
7.1039
7.0999
7.0961
7.0895
7.0854
6.2692
6.2444
4.0911
4.0739
4.0664
4.0566
4.0491
4.0394
4.0319
4.0149
1.3668
1.3496
-0.0000

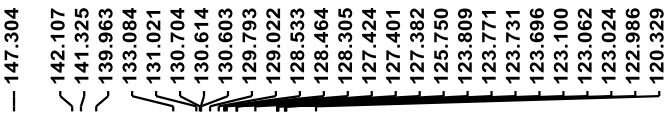

Cl
Me

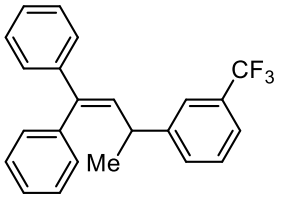
3bg


12.02
3.06
1.00
1.00
3.03
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)

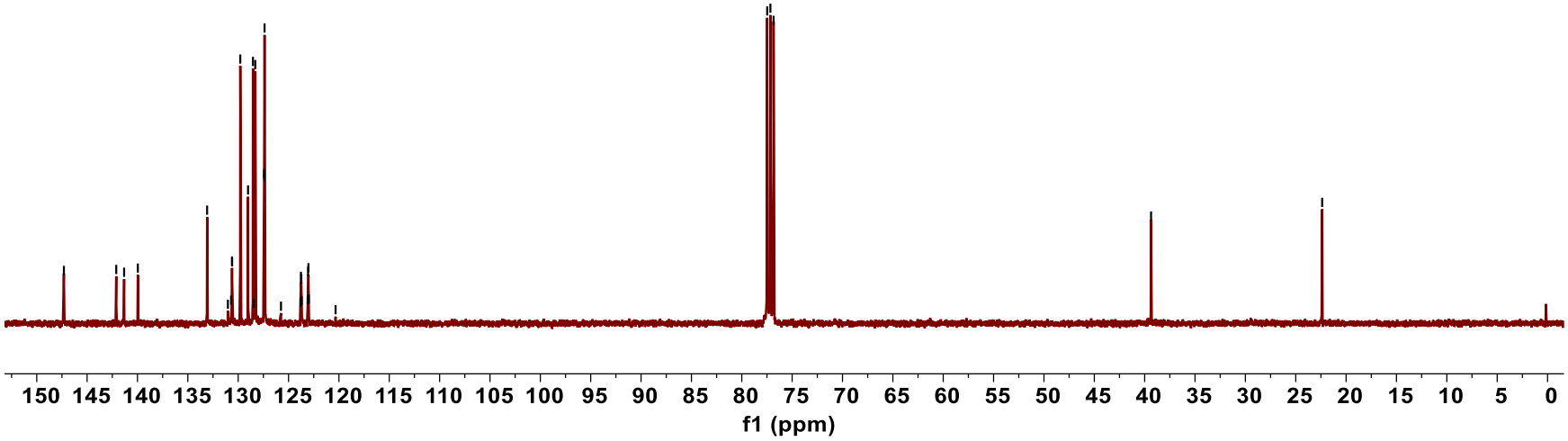

144.275
142.608
141.724
139.906
133.274
132.685
129.850
129.827
128.263
128.225
128.168
127.561
127.256
127.251
127.227
127.147
77.475
77.160
76.841
36.409
22.542
Cl
Me
3bg
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)


7.4523
7.4476
7.4351
7.4200
7.4109
7.3940
7.3752
7.3620
7.3579
7.3517
7.3448
7.2780
7.2692
7.2629
7.2573
7.2501
7.2438
7.2331
7.2291
7.2193
7.2112
7.1695
7.1645
7.1485
6.1968
6.1714
3.6860
3.6688
3.6606
3.6515
3.6431
3.6343
3.6258
3.6086
1.4161
1.3988
-0.0000
CF3
Me
3bh
7.09
6.45
1.95
1.00
1.06
2.96
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)


147.304
142.107
141.325
139.963
133.084
131.021
130.704
130.614
130.603
129.793
129.022
128.533
128.464
128.305
127.424
127.401
127.382
125.750
123.809
123.771
123.731
123.696
123.100
123.062
123.024
122.986
120.329
77.479
77.160
76.841
39.366
22.378

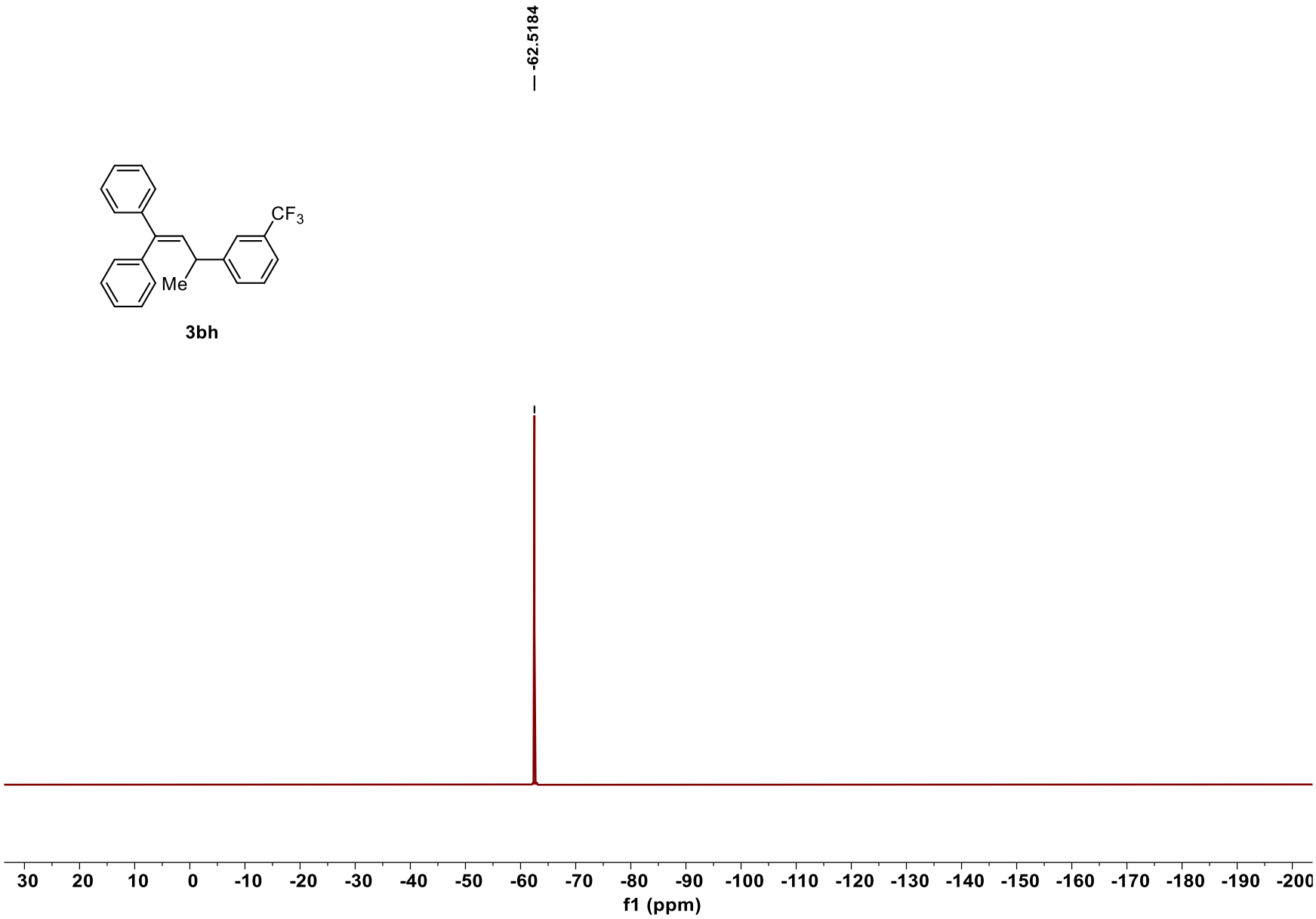

CF3
Me

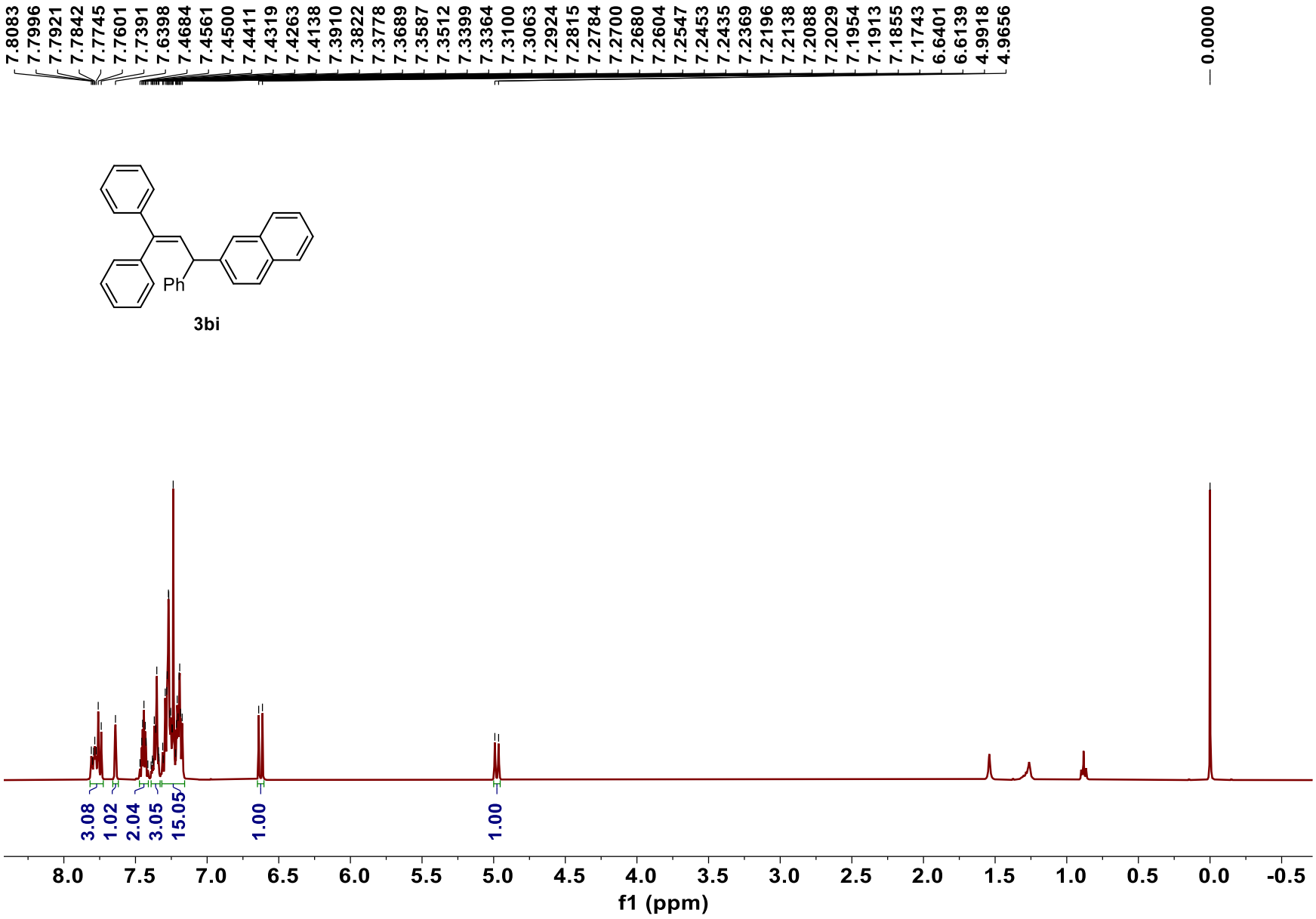
3bh

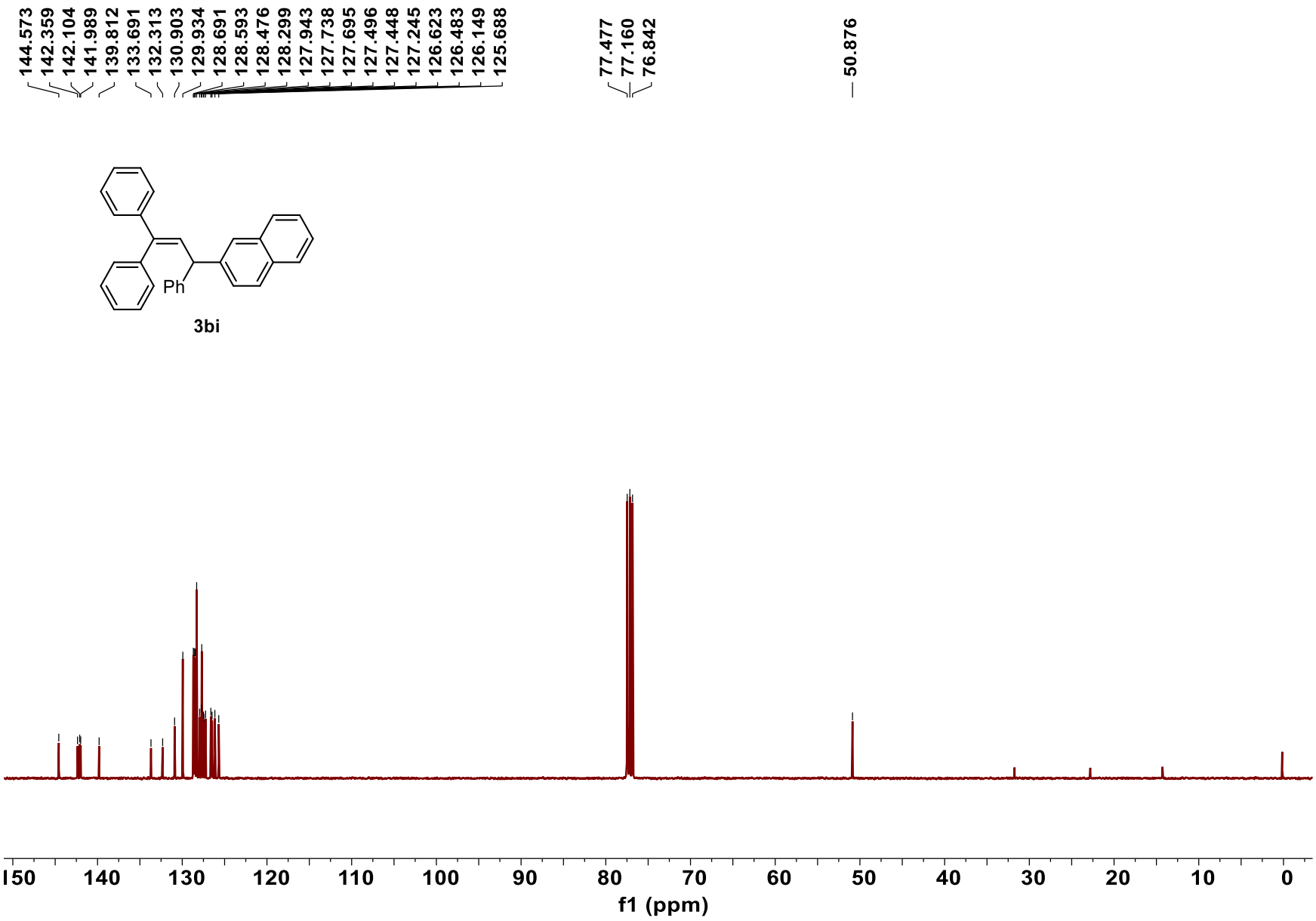

150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

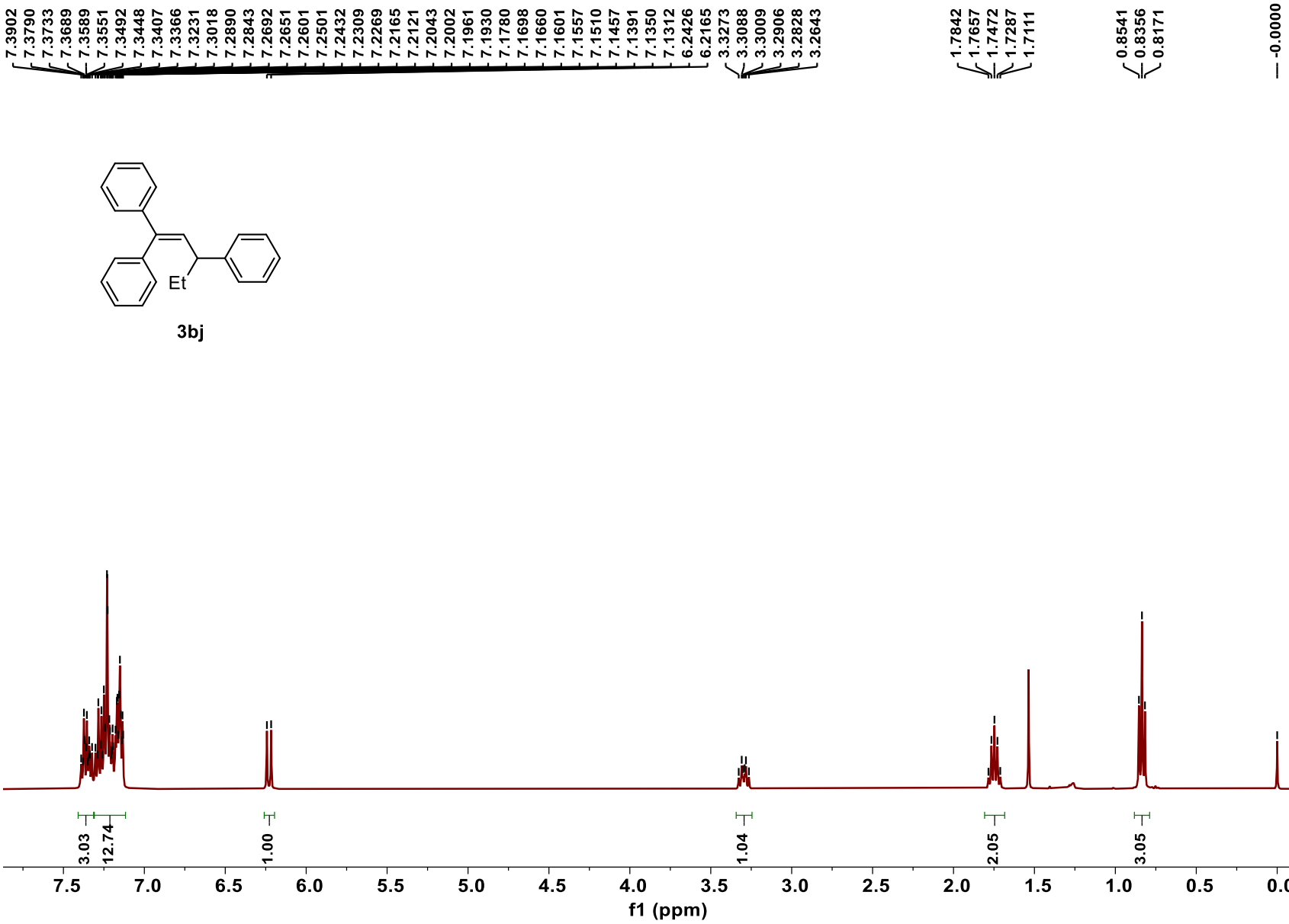
CF3
Me

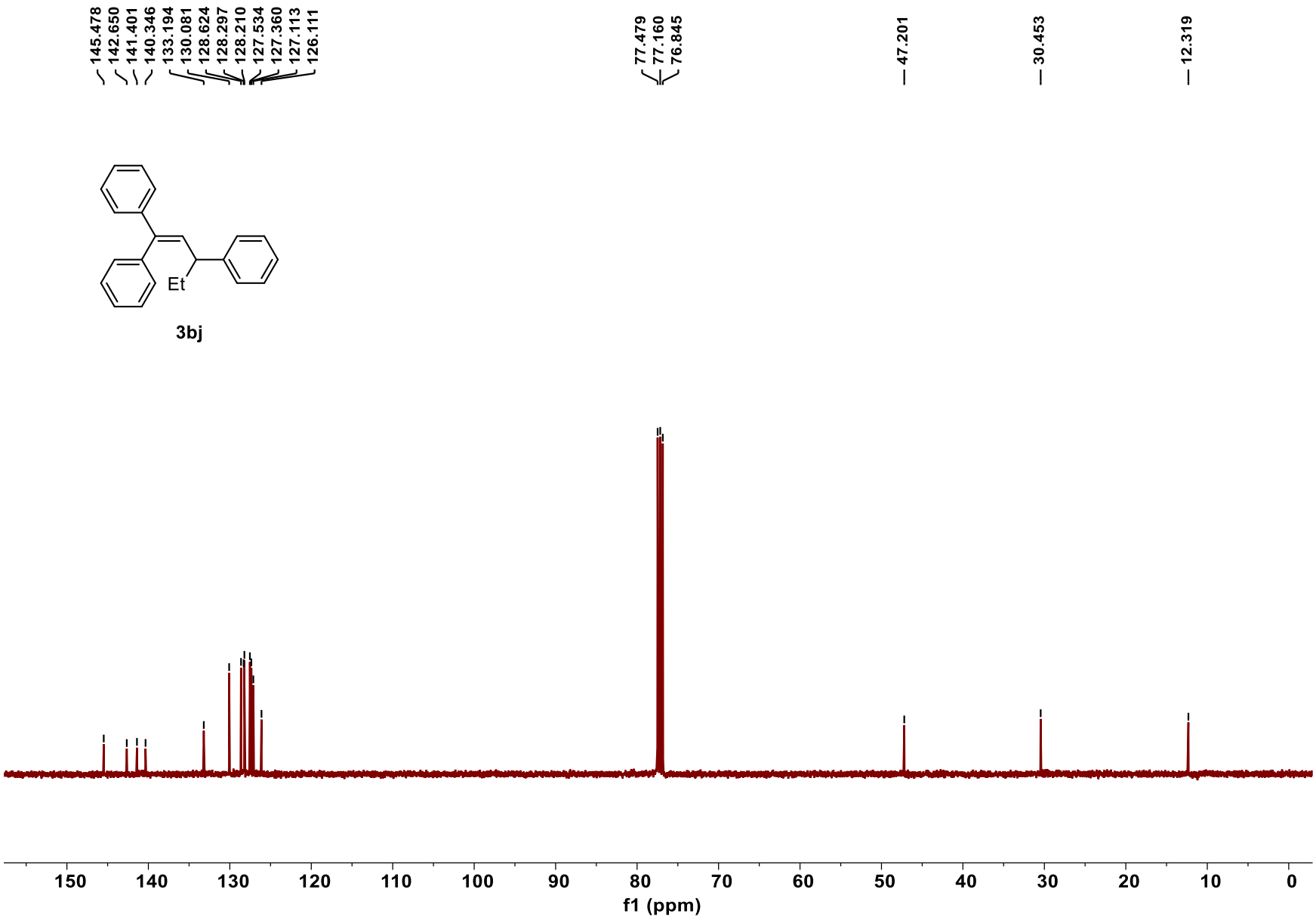
3bh

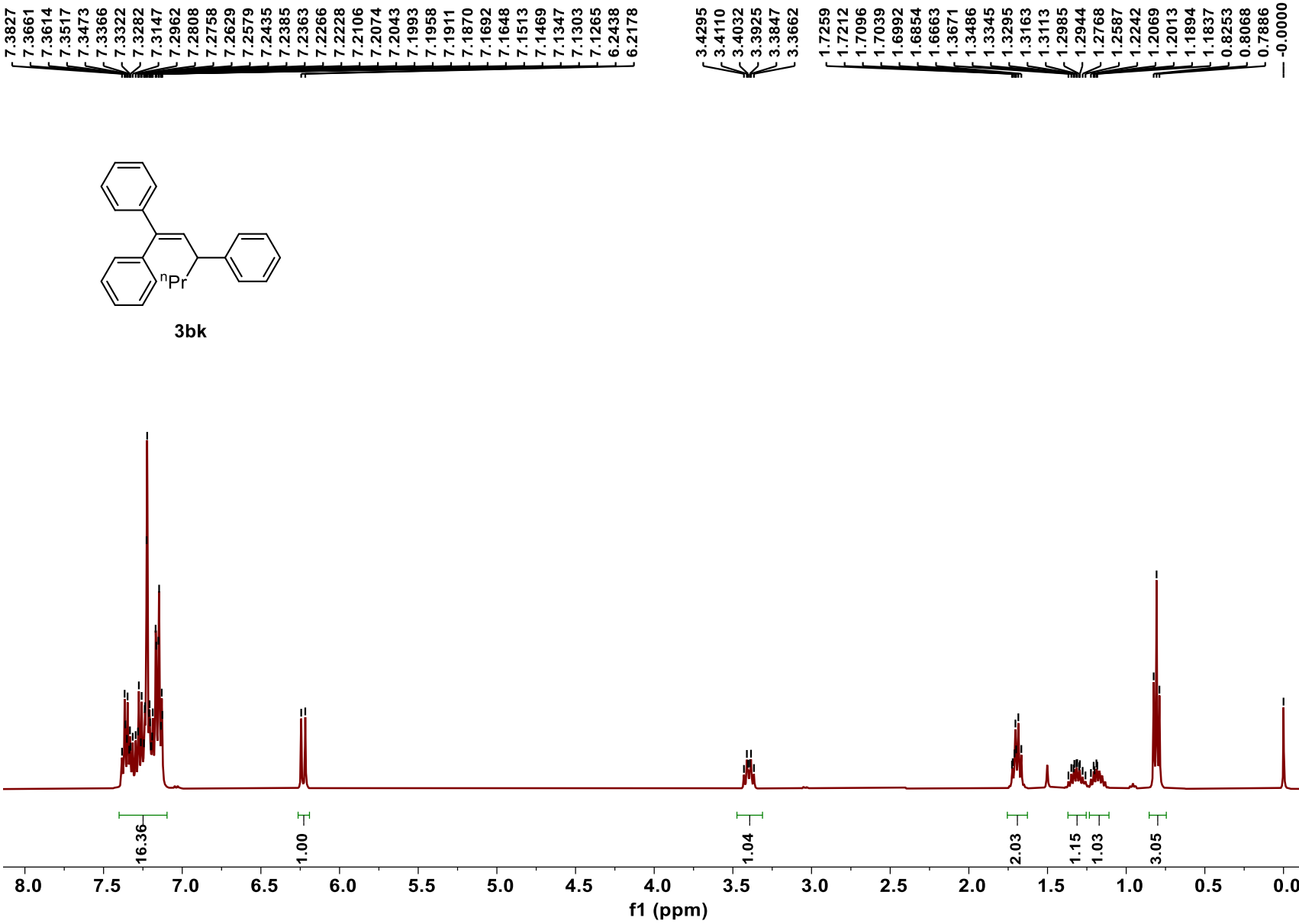

-62.5184
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

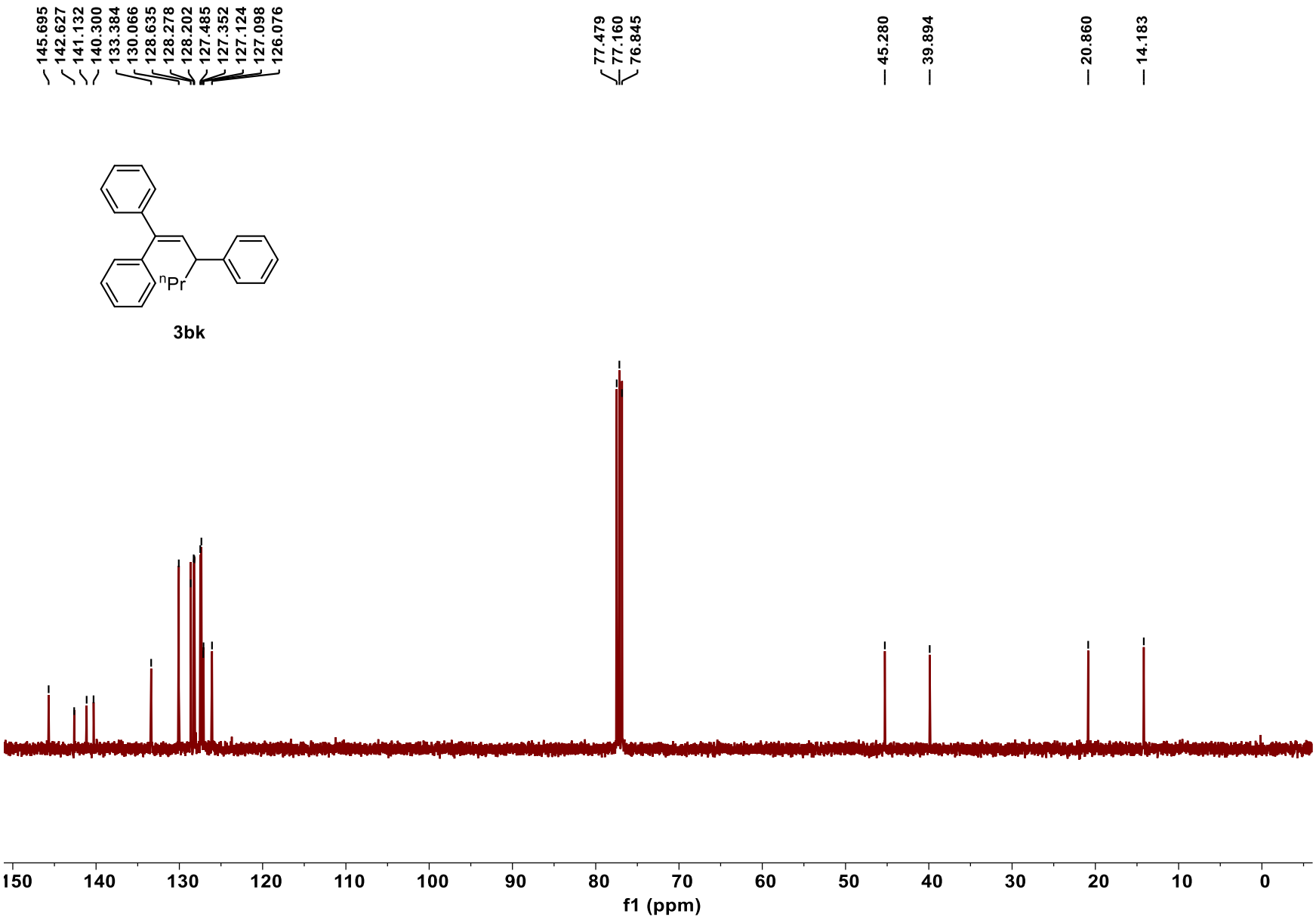

7.8083
7.7996
7.7921
7.7842
7.7745
7.7601
7.7391
7.6398
7.4684
7.4561
7.4500
7.4411
7.4319
7.4263
7.4138
7.3910
7.3822
7.3778
7.3689
7.3587
7.3512
7.3399
7.3364
7.3100
7.3063
7.2924
7.2815
7.2784
7.2700
7.2680
7.2604
7.2547
7.2453
7.2435
7.2369
7.2196
7.2138
7.2088
7.2029
7.1954
7.1913
7.1855
7.1743
6.6401
6.6139
4.9918
4.9656
0.0000
Ph
3bi
3.08
1.02
2.04
3.05
15.05
1.00
1.00
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
-0.5
f1 (ppm)

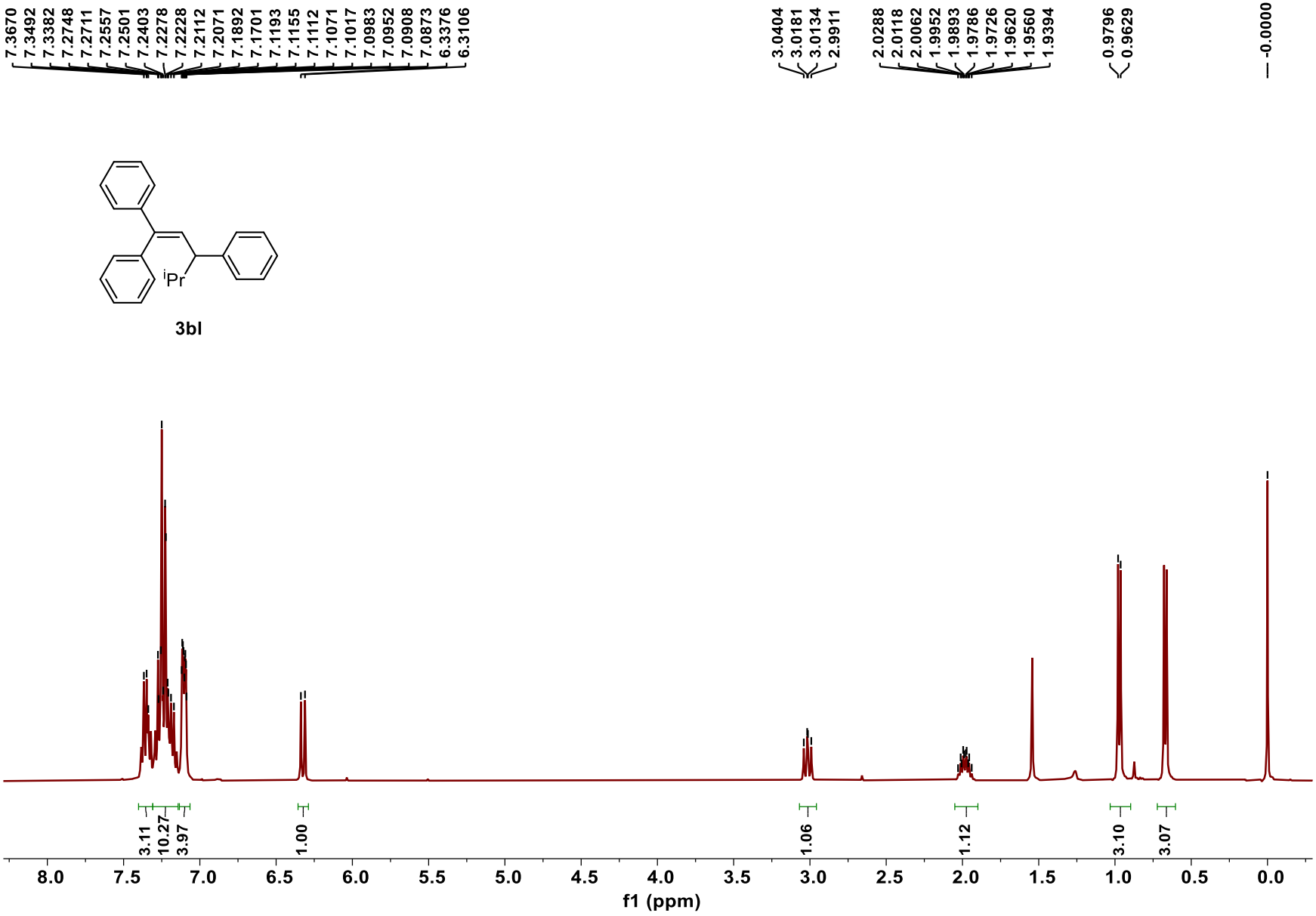

144.573
142.359
142.104
141.989
139.812
133.691
132.313
130.903
129.934
128.691
128.593
128.476
128.299
127.943
127.738
127.695
127.496
127.448
127.245
126.623
126.483
126.149
125.688
77.477
77.160
76.842
50.876
Ph
3bi
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

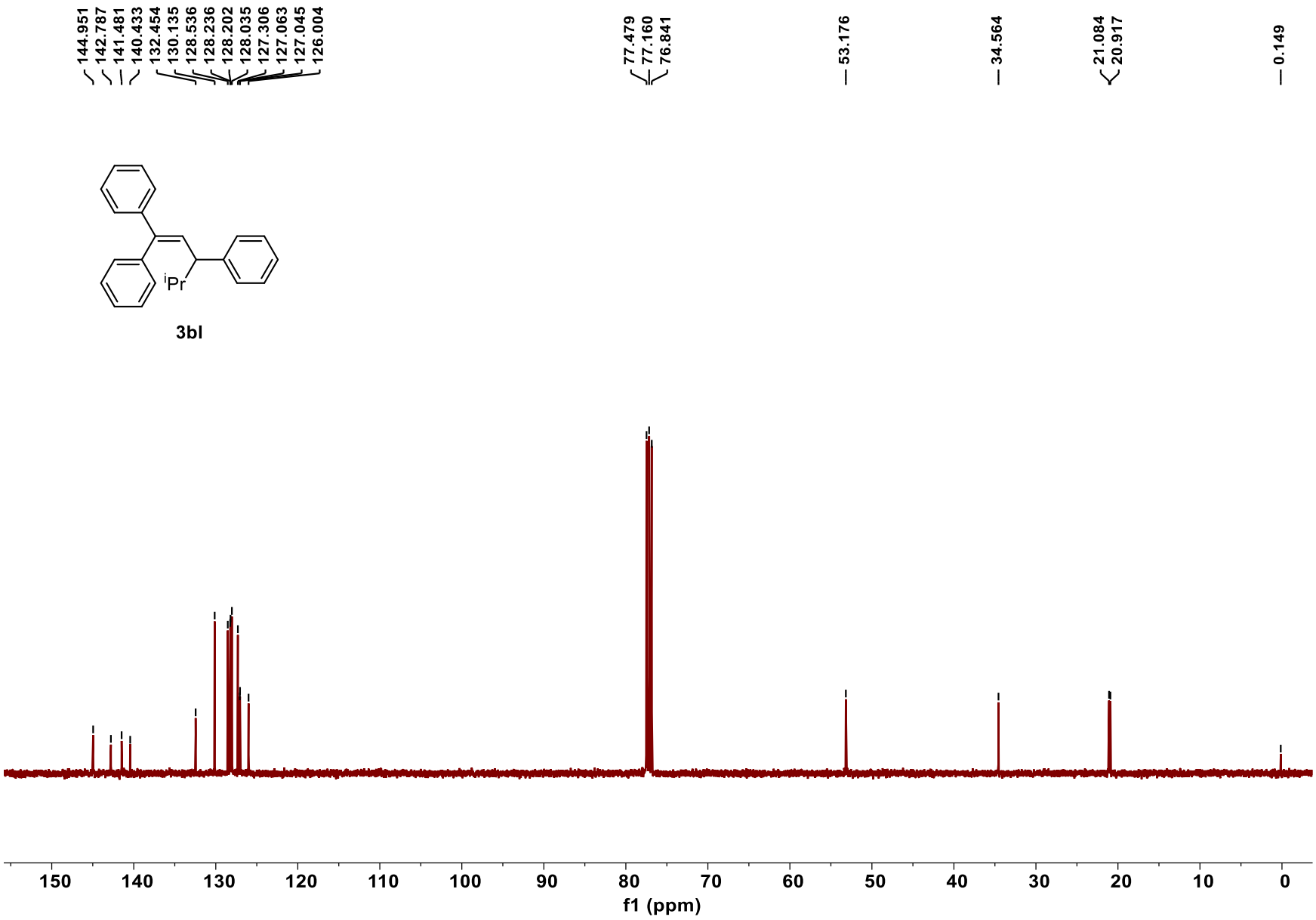

7.3902
7.3790
7.3733
7.3689
7.3589
7.3551
7.3492
7.3448
7.3407
7.3366
7.3231
7.3018
7.2890
7.2843
7.2692
7.2651
7.2601
7.2501
7.2432
7.2309
7.2269
7.2165
7.2121
7.2043
7.2002
7.1961
7.1930
7.1780
7.1698
7.1660
7.1601
7.1557
7.1510
7.1457
7.1391
7.1350
7.1312
6.2426
6.2165
3.3273
3.3088
3.3009
3.2906
3.2828
3.2643
1.7842
1.7657
1.7472
1.7287
1.7111
0.8541
0.8356
0.8171
-0.0000
Et
3bj
3.03
12.74
1.00
1.04
2.05
3.05
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)

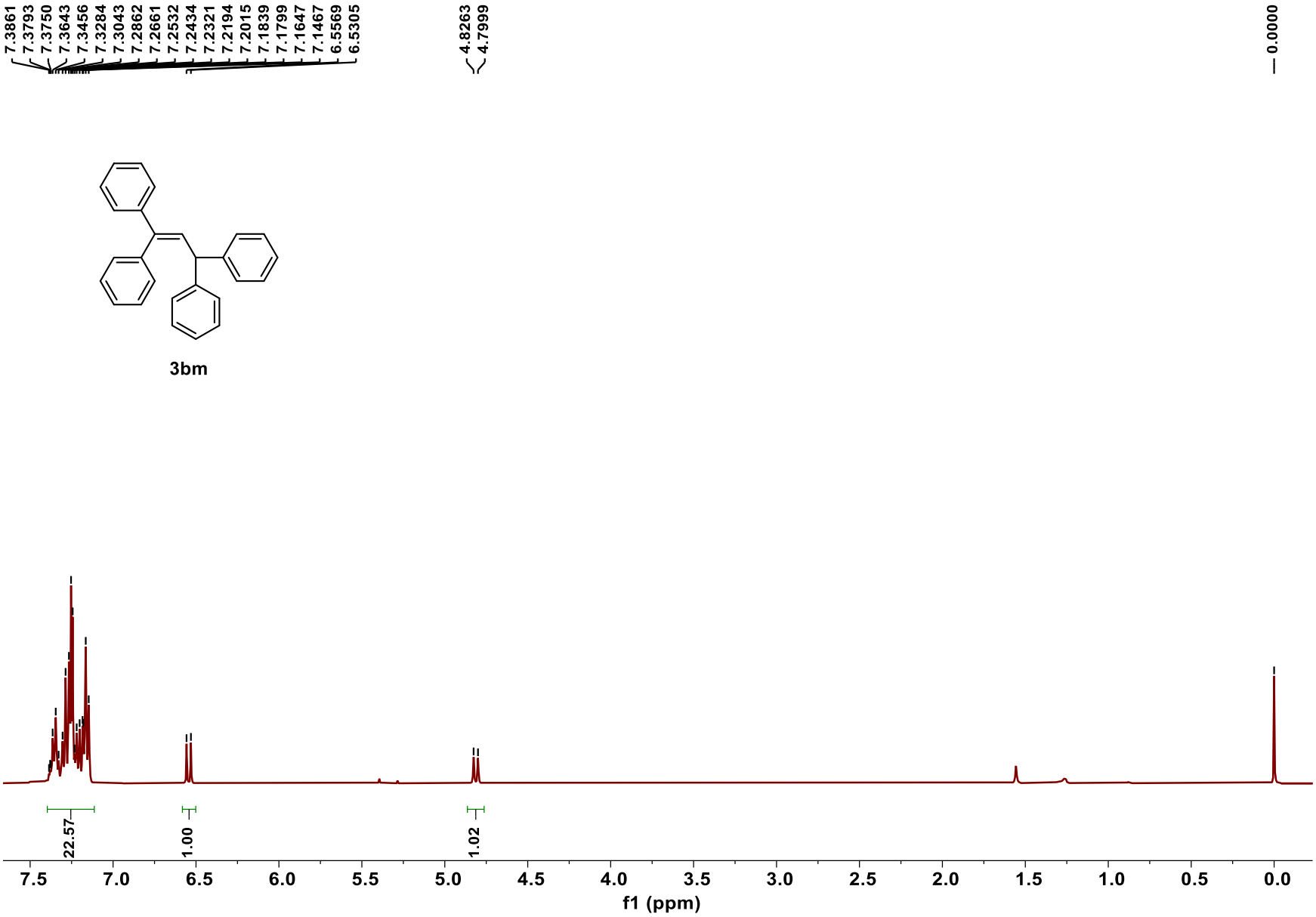

145.478
142.650
141.401
140.346
133.194
130.081
128.624
128.297
128.210
127.534
127.360
127.113
126.111
77.479
77.160
76.845
47.201
30.453
12.319
Et
3bj
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

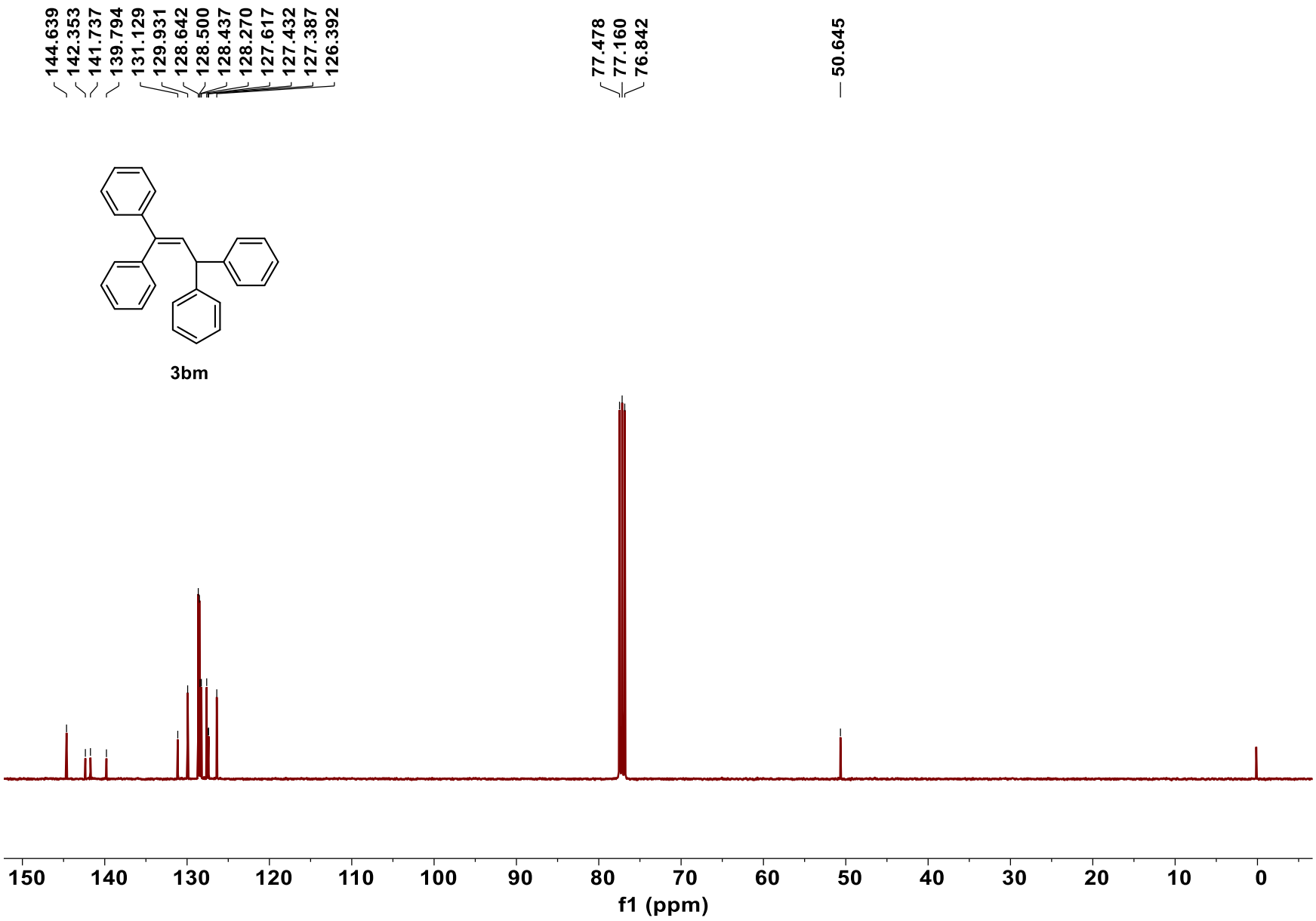

7.3827
7.3661
7.3614
7.3517
7.3473
7.3366
7.3322
7.3282
7.3147
7.2962
7.2808
7.2758
7.2629
7.2579
7.2435
7.2385
7.2363
7.2266
7.2228
7.2106
7.2074
7.2043
7.1993
7.1958
7.1911
7.1870
7.1692
7.1648
7.1513
7.1469
7.1347
7.1303
7.1265
6.2438
6.2178
3.4295
3.4110
3.4032
3.3925
3.3847
3.3662
1.7259
1.7212
1.7096
1.7039
1.6992
1.6854
1.6663
1.3671
1.3486
1.3345
1.3295
1.3163
1.3113
1.2985
1.2944
1.2768
1.2587
1.2242
1.2069
1.2013
1.1894
1.1837
0.8253
0.8068
0.7886
-0.0000
nPr
3bk
16.36
1.00
1.04
2.03
1.15
1.03
3.05
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)

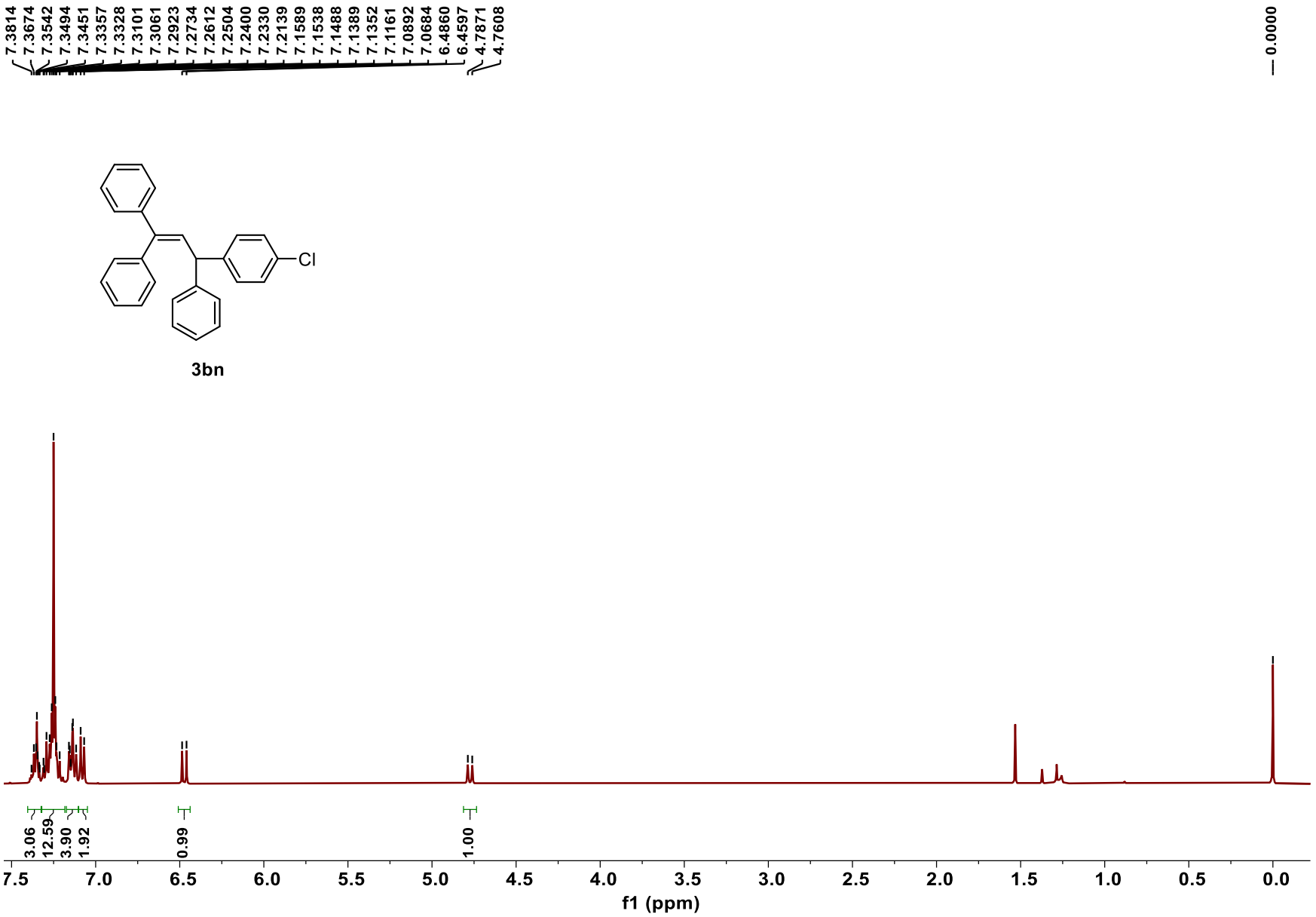

145.695
142.627
141.132
140.300
133.384
130.066
128.635
128.278
128.202
127.485
127.352
127.124
127.098
126.076
77.479
77.160
76.845
45.280
39.894
20.860
14.183
nPr
3bk
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

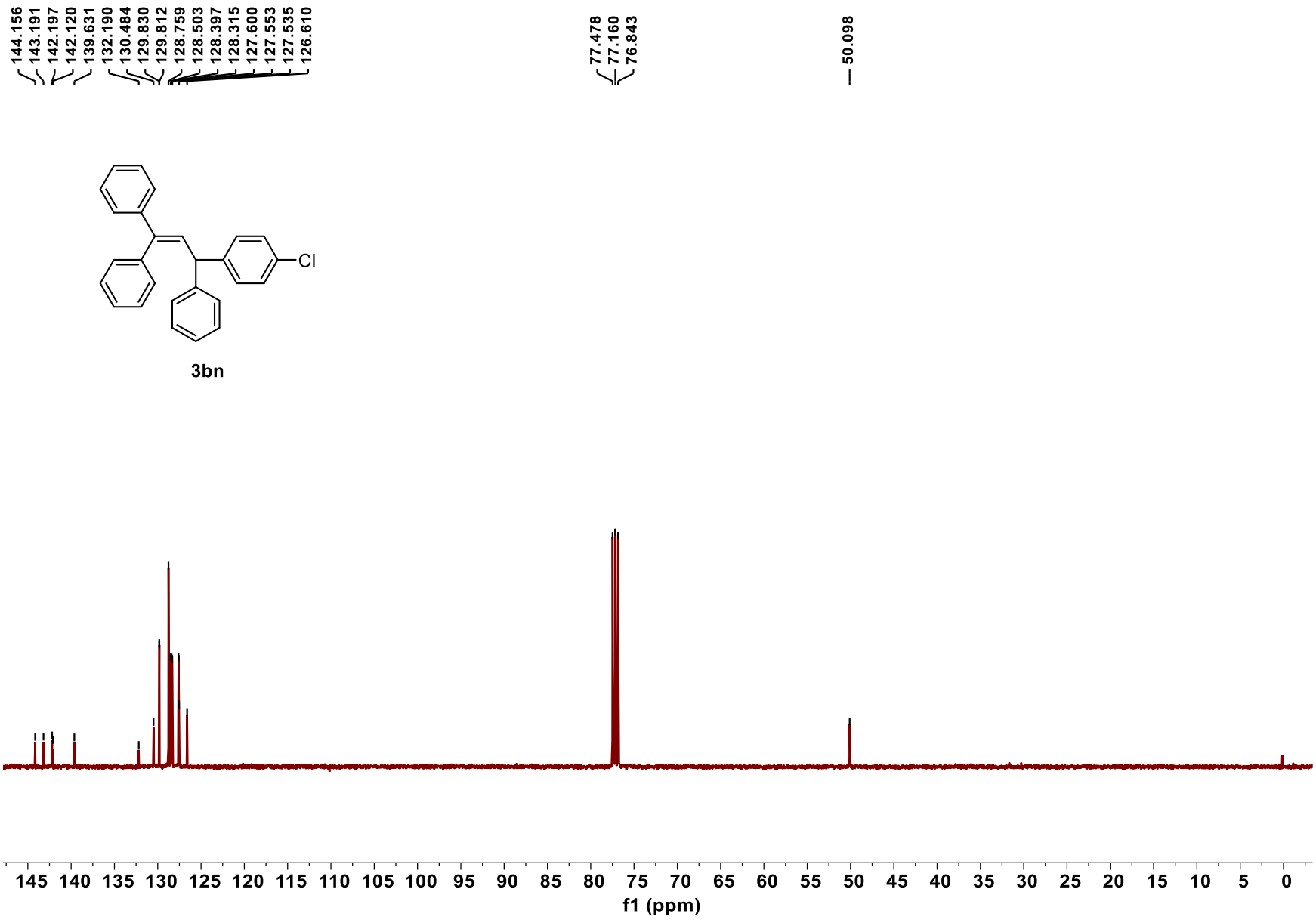

7.3670
7.3492
7.3382
7.2748
7.2711
7.2557
7.2501
7.2403
7.2278
7.2228
7.2112
7.2071
7.1892
7.1701
7.1193
7.1155
7.1112
7.1071
7.1017
7.0983
7.0952
7.0908
7.0873
6.3376
6.3106
3.0404
3.0181
3.0134
2.9911
2.0288
2.0118
2.0062
1.9952
1.9893
1.9786
1.9726
1.9620
1.9560
1.9394
0.9796
0.9629
-0.0000
iPr
3bl
3.11
10.27
3.97
1.00
1.06
1.12
3.10
3.07
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)

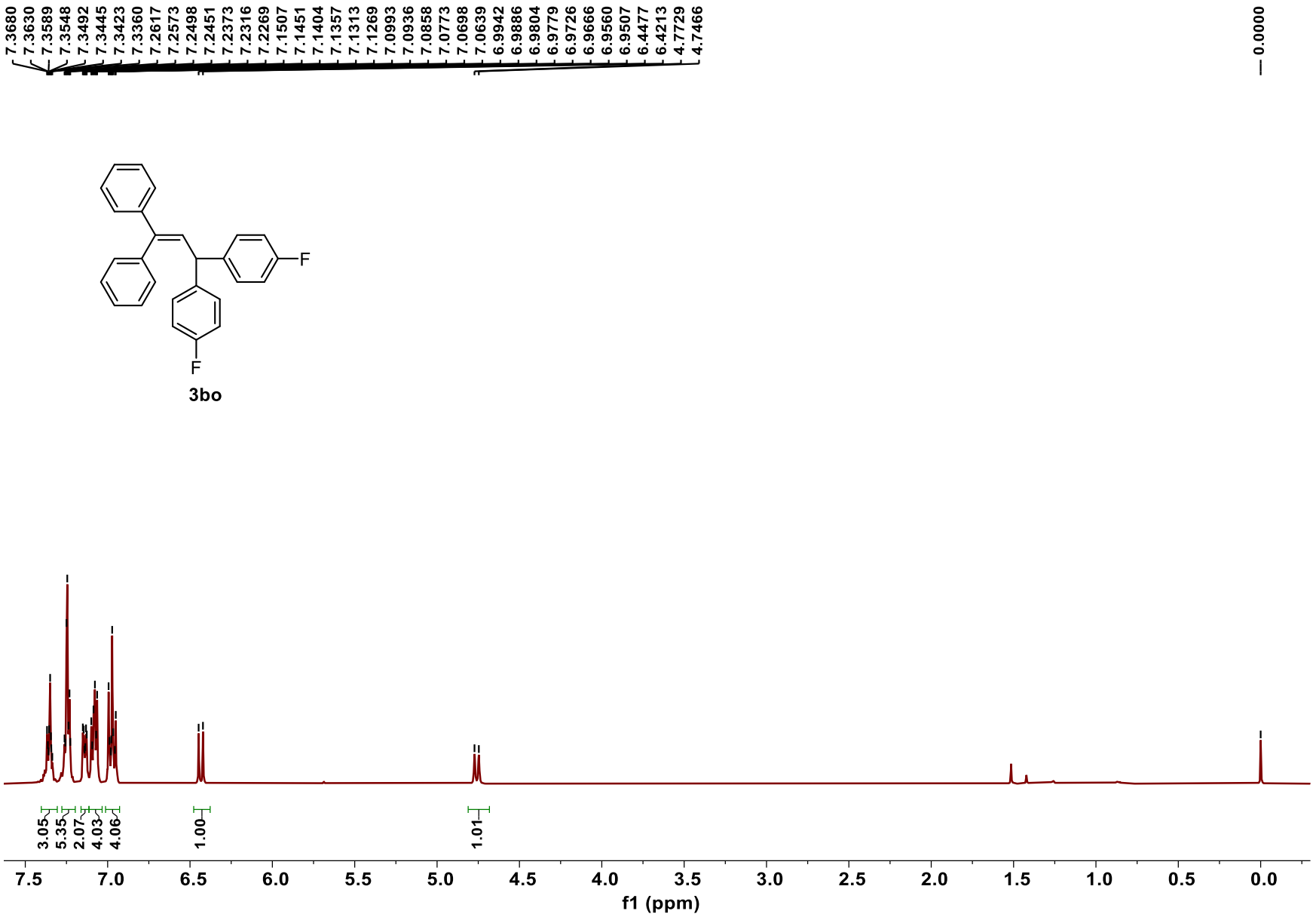

144.951
142.787
141.481
140.433
132.454
130.135
128.536
128.236
128.202
128.035
127.306
127.063
127.045
126.004
77.479
77.160
76.841
53.176
34.564
21.084
20.917
0.149
iPr
3bl
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

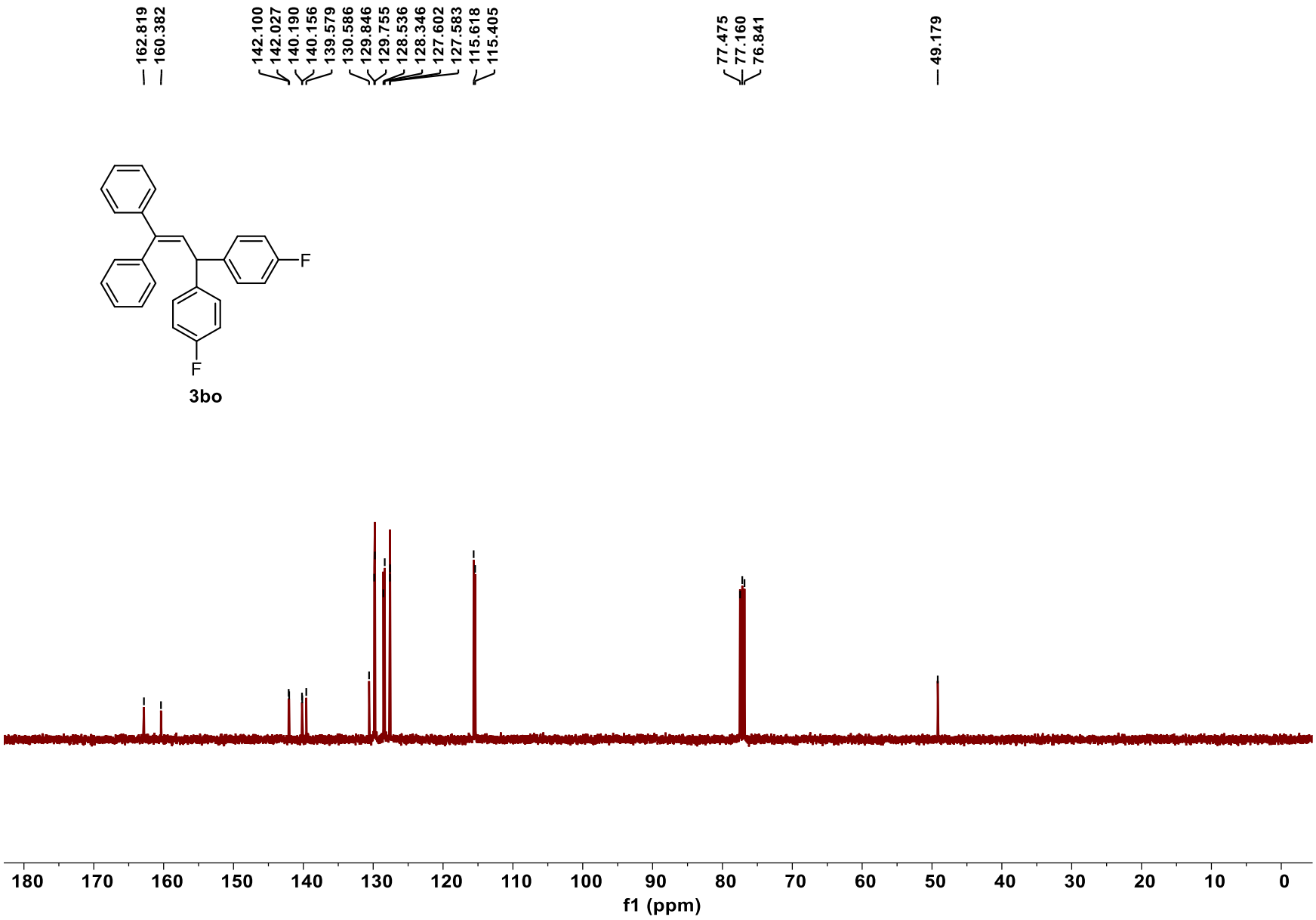

7.3861
7.3793
7.3750
7.3643
7.3456
7.3284
7.3043
7.2862
7.2661
7.2532
7.2434
7.2321
7.2194
7.2015
7.1839
7.1799
7.1647
7.1467
6.5569
6.5305
4.8263
4.7999
0.0000
3bm
22.57
1.00
1.02
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


144.639
142.353
141.737
139.794
131.129
129.931
128.642
128.500
128.437
128.270
127.617
127.432
127.387
126.392
77.478
77.160
76.842
50.645
3bm
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.3814
7.3674
7.3542
7.3494
7.3451
7.3357
7.3328
7.3101
7.3061
7.2923
7.2734
7.2612
7.2504
7.2400
7.2330
7.2139
7.1589
7.1538
7.1488
7.1389
7.1352
7.1161
7.0892
7.0684
6.4860
6.4597
4.7871
4.7608
0.0000
Cl
3bn
3.06
12.59
3.90
1.92
0.99
1.00
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)

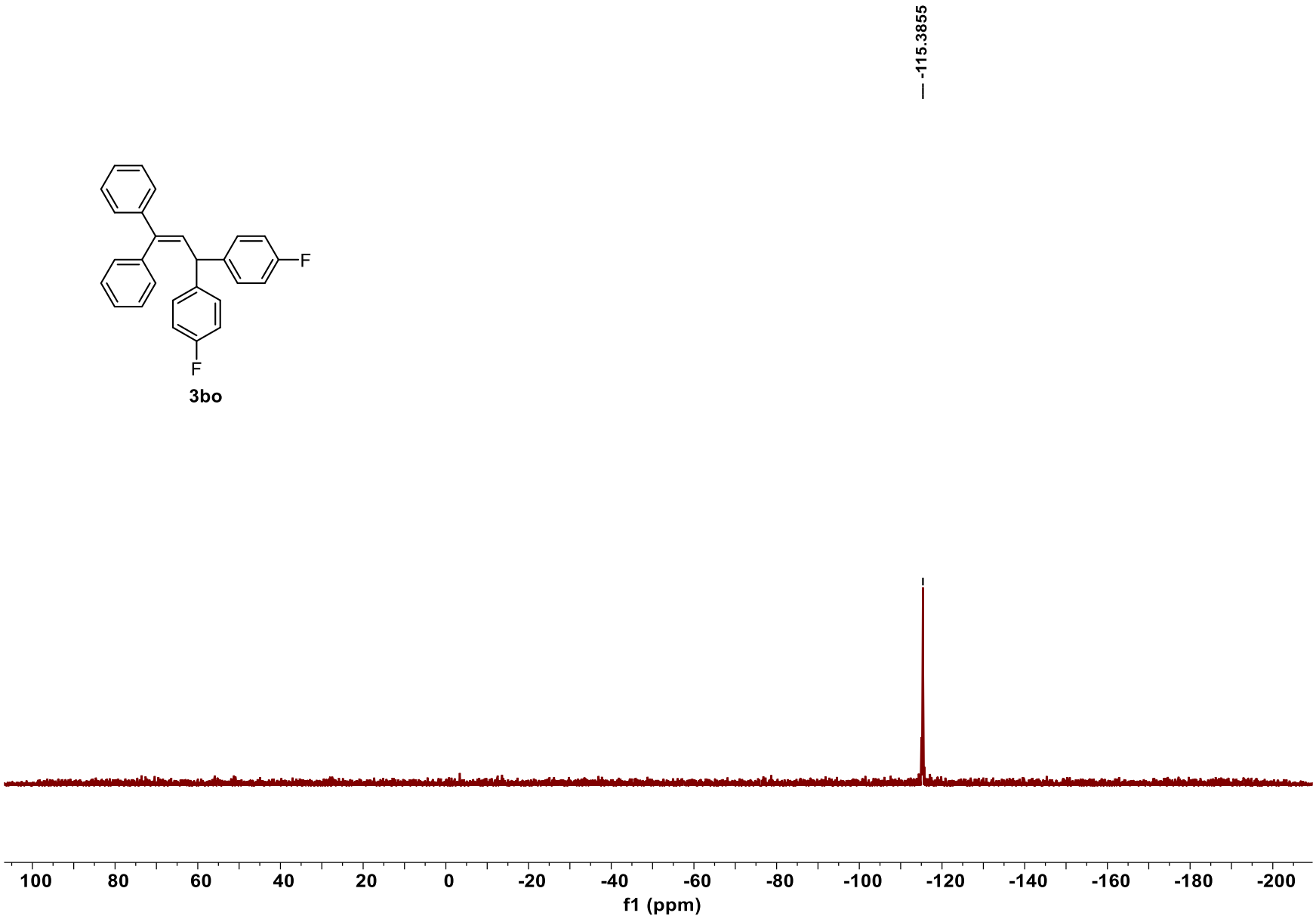

144.156
143.191
142.197
142.120
139.631
132.190
130.484
129.830
129.812
128.759
128.503
128.397
128.315
127.600
127.553
127.535
126.610
77.478
77.160
76.843
50.098
Cl
3bn
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

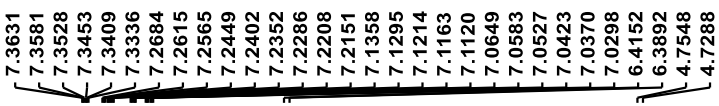

7.3680
7.3630
7.3589
7.3548
7.3492
7.3445
7.3423
7.3360
7.2617
7.2573
7.2498
7.2451
7.2373
7.2316
7.2269
7.1507
7.1451
7.1404
7.1357
7.1313
7.1269
7.0993
7.0936
7.0858
7.0773
7.0698
7.0639
6.9942
6.9886
6.9804
6.9779
6.9726
6.9666
6.9560
6.9507
6.4477
6.4213
4.7729
4.7466
0.0000
F
F
3bo
3.05
5.35
2.07
4.03
4.06
1.00
1.01
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)


162.819
160.382
142.100
142.027
140.190
140.156
139.579
130.586
129.846
129.755
128.536
128.346
127.602
127.583
115.618
115.405
77.475
77.160
76.841
49.179
F
F
3bo
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

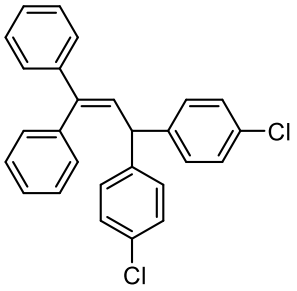
F
F

3bo

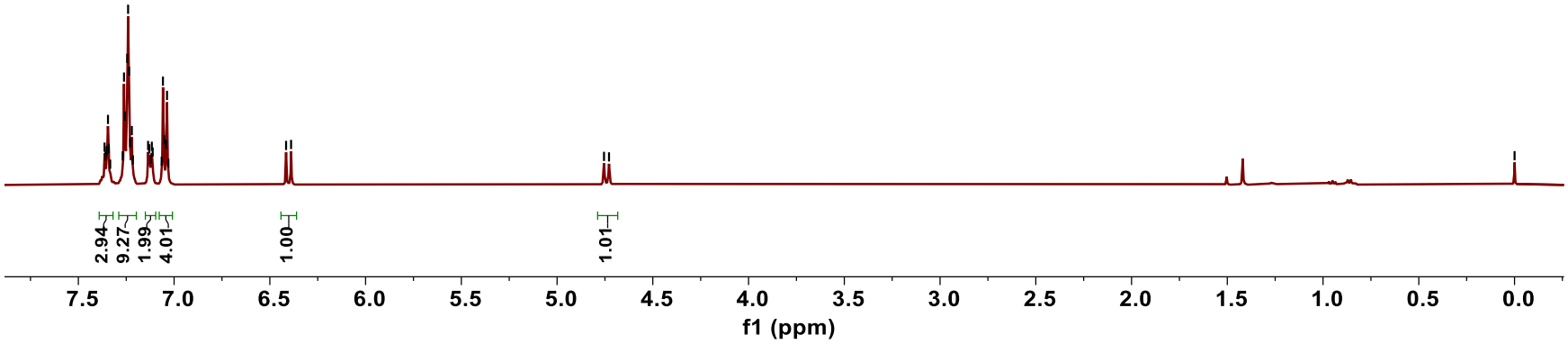

-115.3855
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
f1 (ppm)

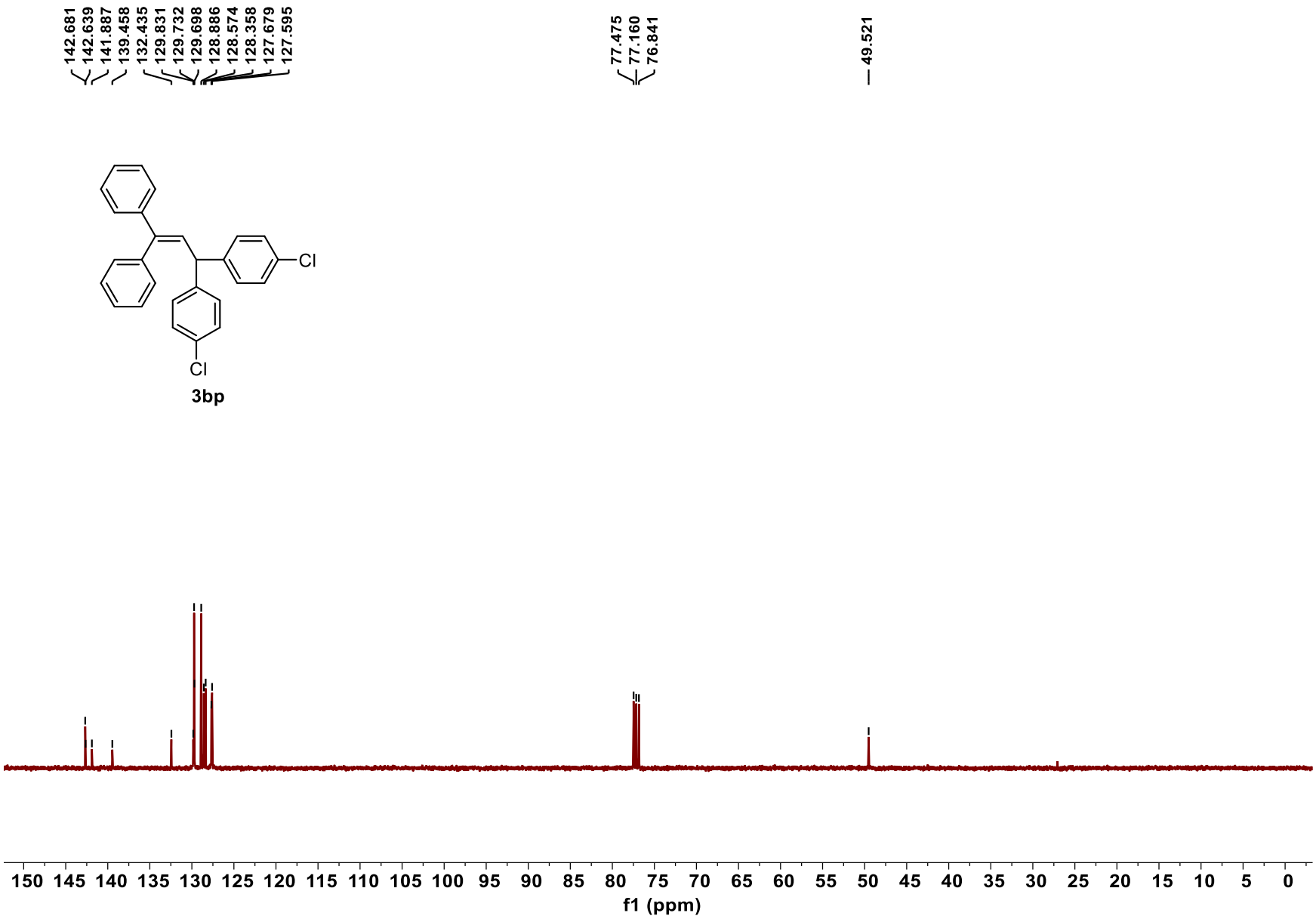

7.3631
7.3581
7.3528
7.3453
7.3409
7.3336
7.2684
7.2615
7.2565
7.2449
7.2402
7.2352
7.2286
7.2208
7.2151
7.1358
7.1295
7.1214
7.1163
7.1120
7.0649
7.0583
7.0527
7.0423
7.0370
7.0298
6.4152
6.3892
4.7548
4.7288

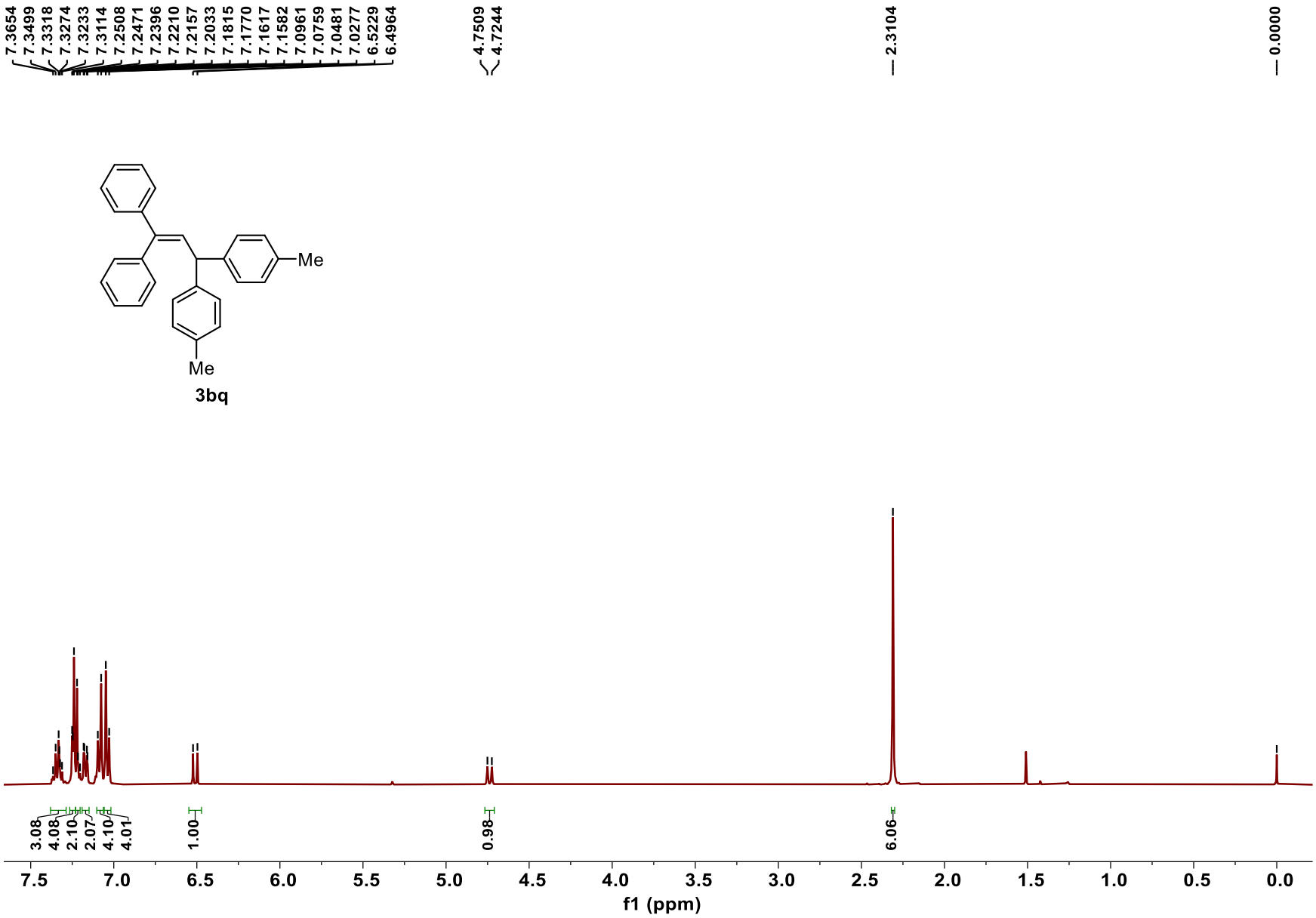
-0.0002

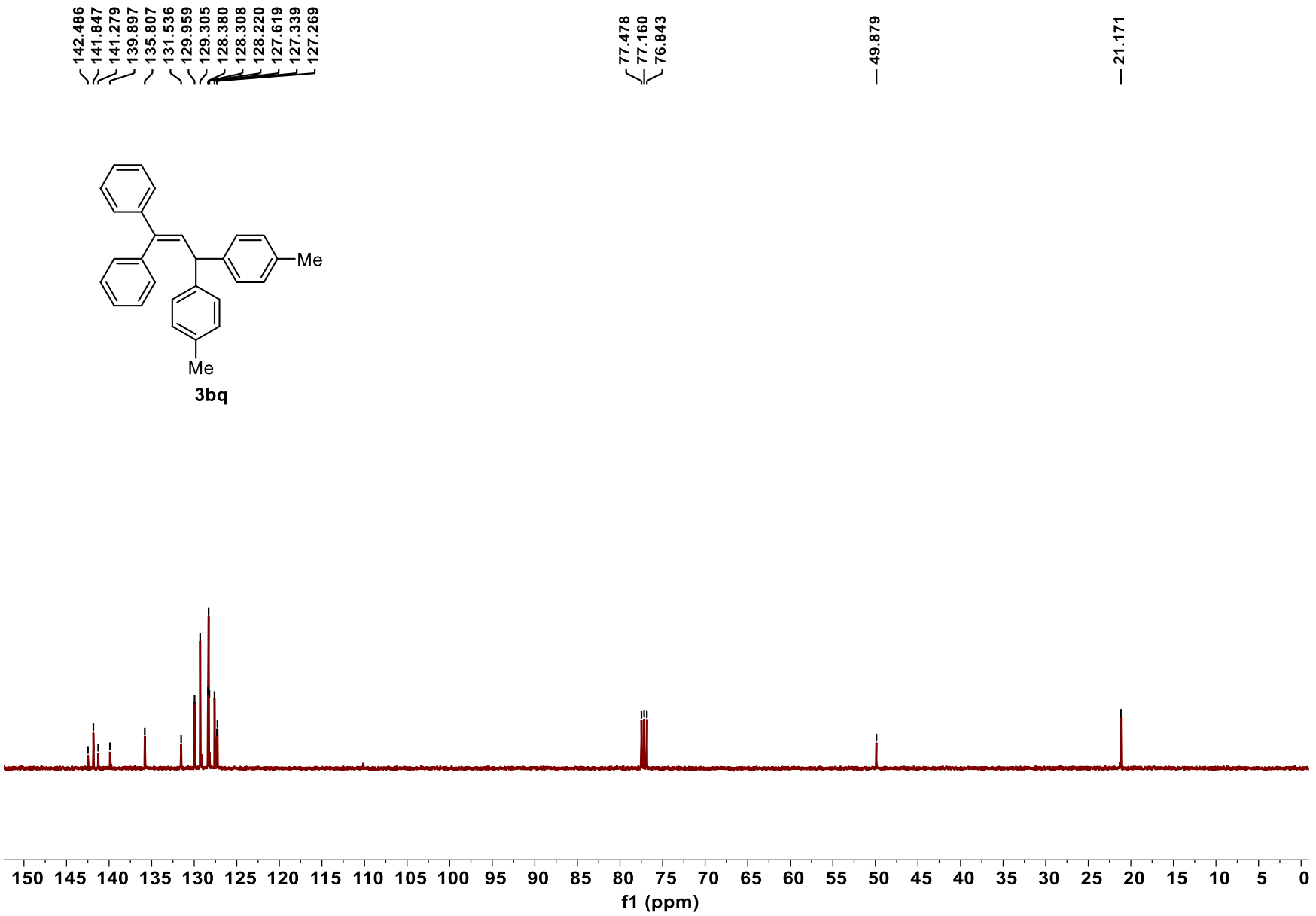

Cl
Cl

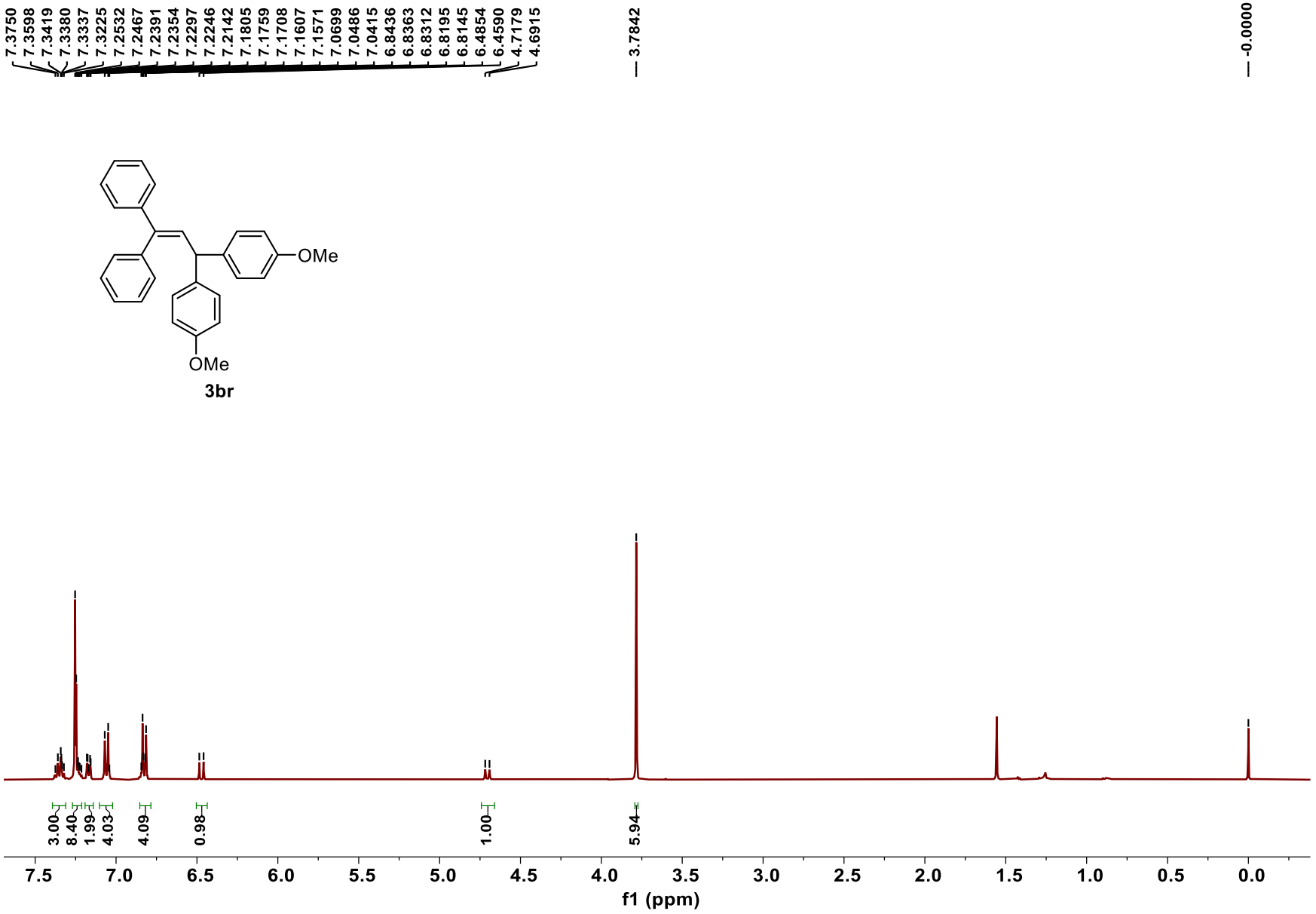
3bp

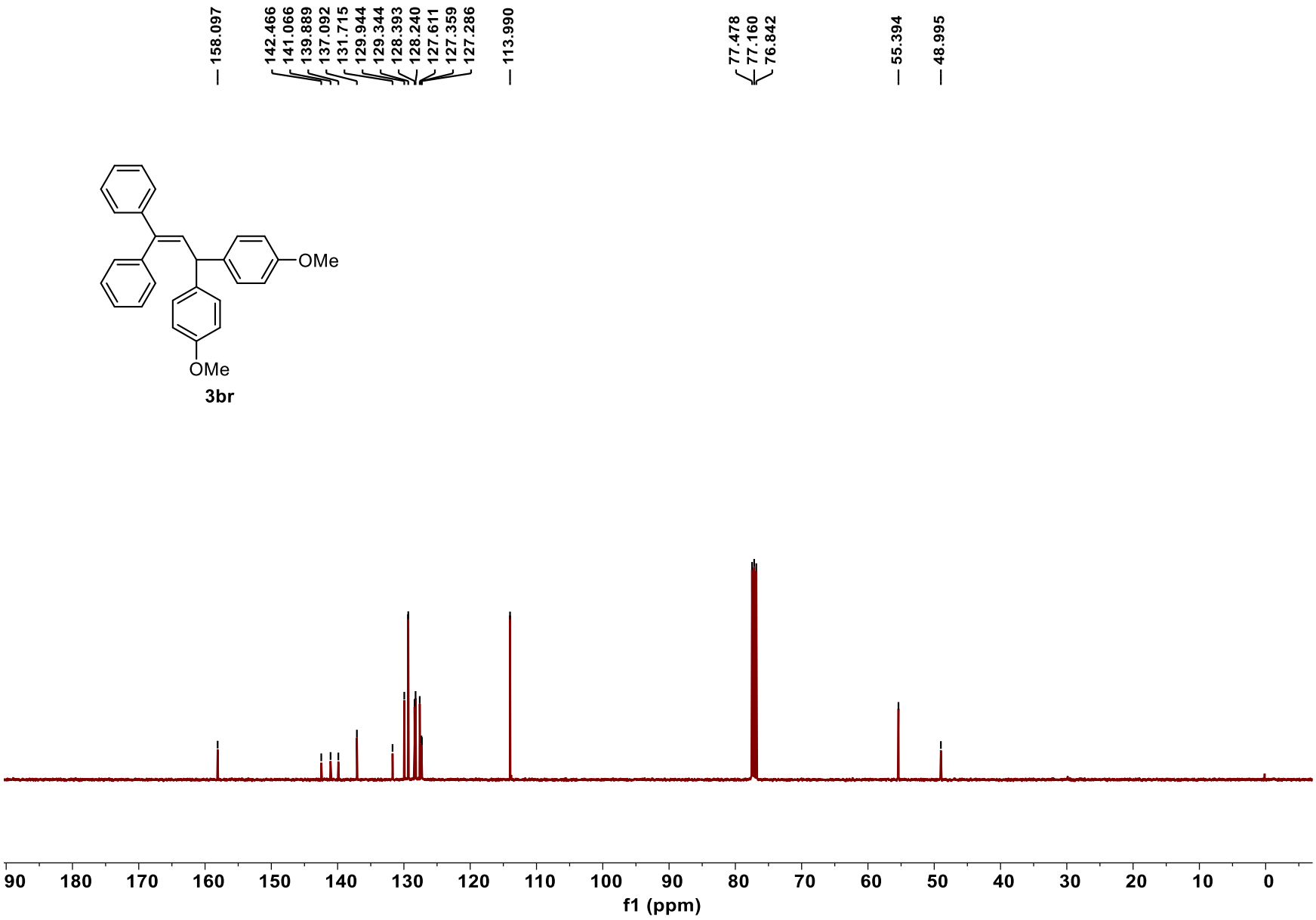

2.94
9.27
1.99
4.01
1.00
1.01
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

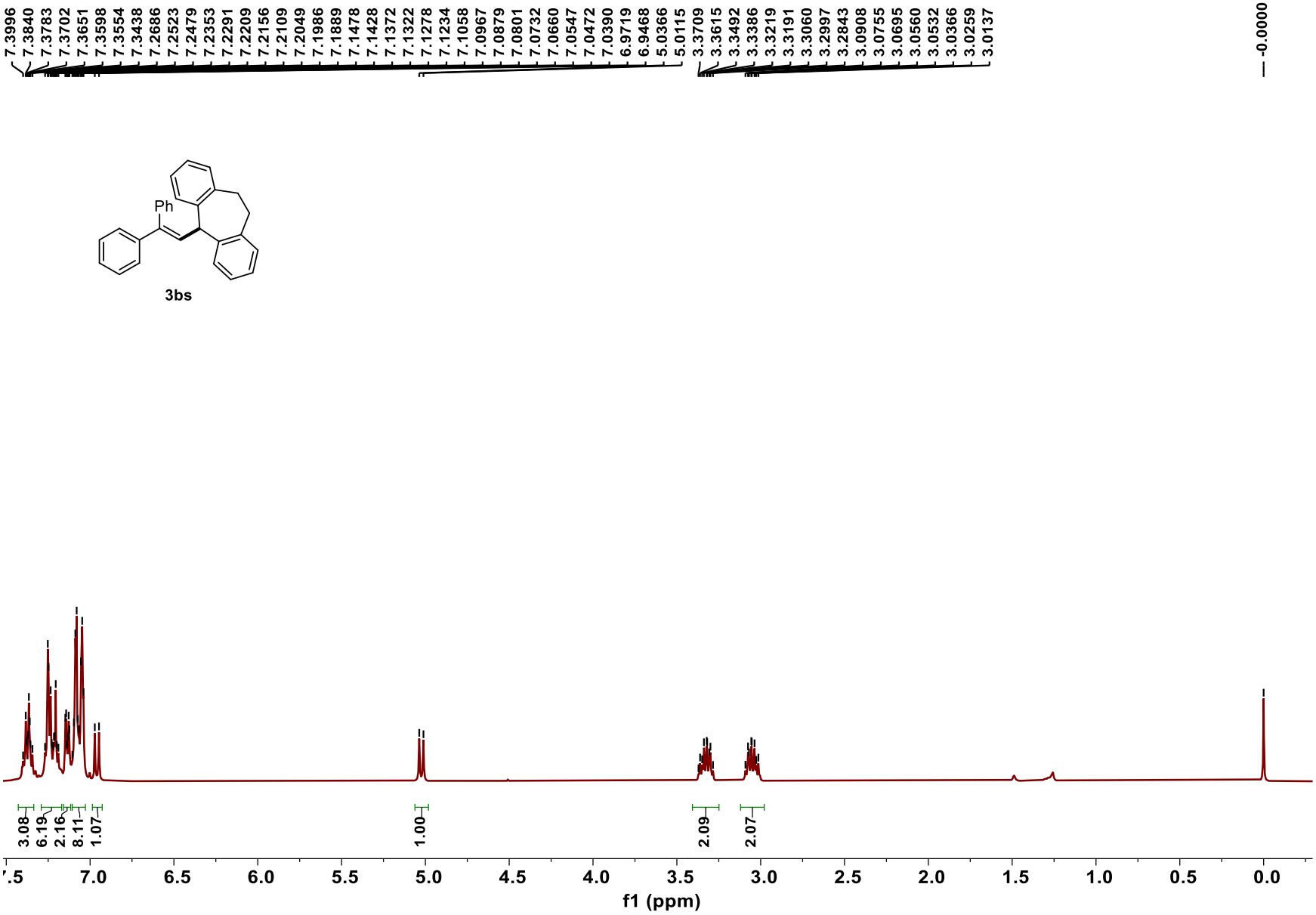

142.681
142.639
141.887
139.458
132.435
129.831
129.732
129.698
128.886
128.574
128.358
127.679
127.595
77.475
77.160
76.841
49.521
Cl
Cl
3bp
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

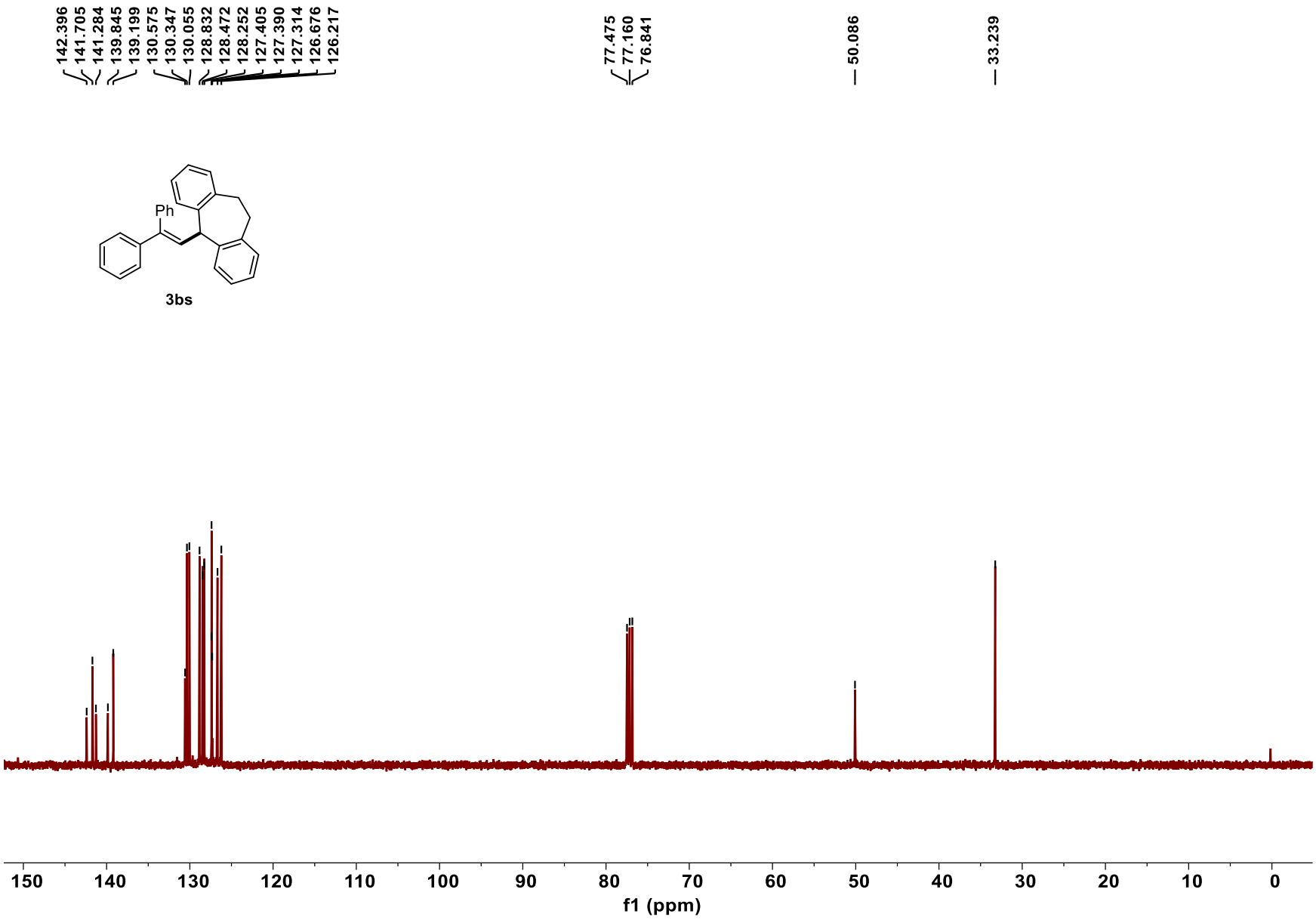

7.3654
7.3499
7.3318
7.3274
7.3233
7.3114
7.2508
7.2471
7.2396
7.2210
7.2157
7.2033
7.1815
7.1770
7.1617
7.1582
7.0961
7.0759
7.0481
7.0277
6.5229
6.4964
4.7509
4.7244
2.3104
0.0000
Me
Me
3bq
3.08
4.08
2.10
2.07
4.10
4.01
1.00
0.98
6.06
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

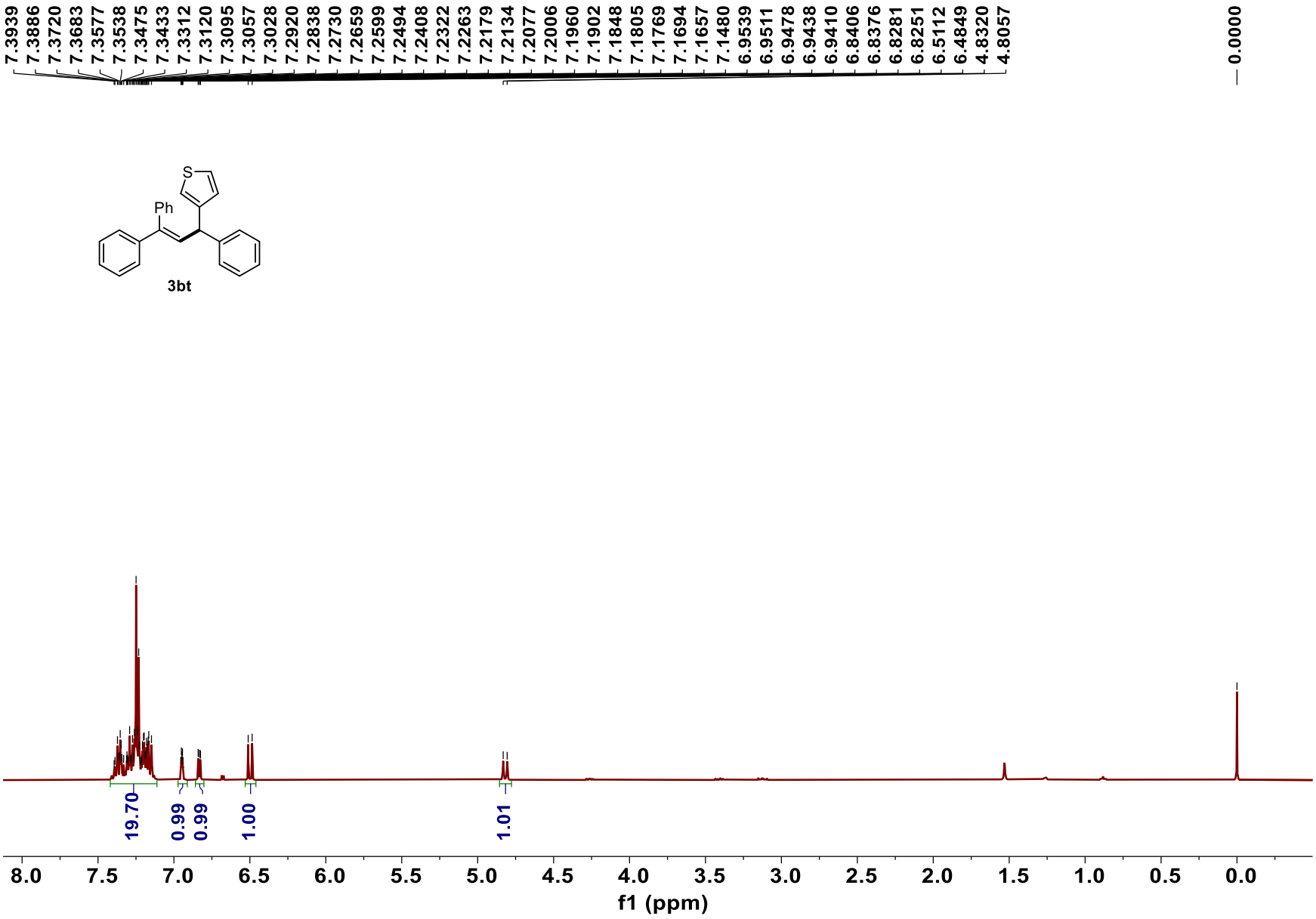

142.486
141.847
141.279
139.897
135.807
131.536
129.959
129.305
128.380
128.308
128.220
127.619
127.339
127.269
77.478
77.160
76.843
49.879
21.171
Me
Me
3bq
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

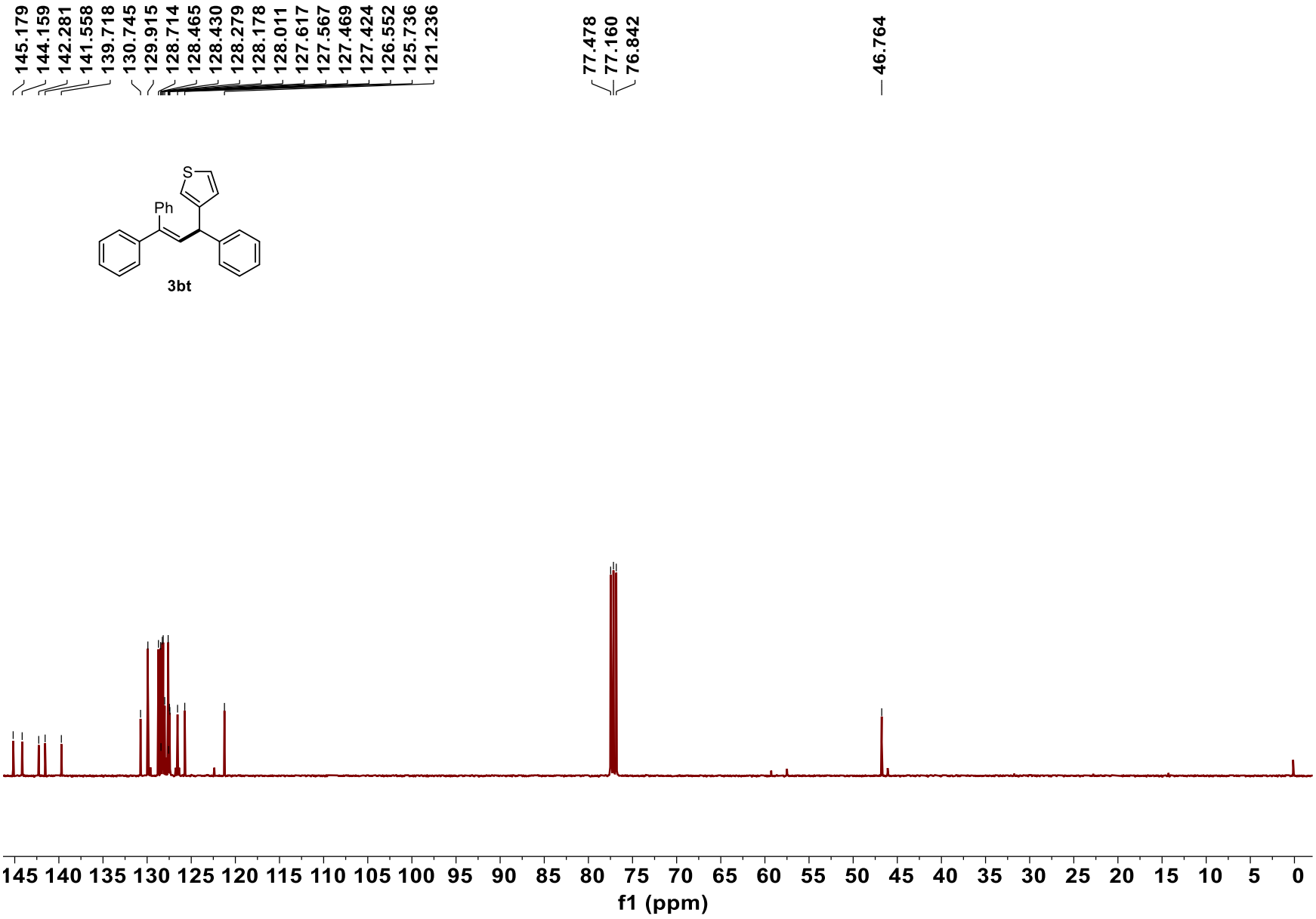

7.3750
7.3598
7.3419
7.3380
7.3337
7.3225
7.2532
7.2467
7.2391
7.2354
7.2297
7.2246
7.2142
7.1805
7.1759
7.1708
7.1607
7.1571
7.0699
7.0486
7.0415
6.8436
6.8363
6.8312
6.8195
6.8145
6.4854
6.4590
4.7179
4.6915
3.7842
-0.0000
OMe
OMe
3br
3.00
8.40
1.99
4.03
4.09
0.98
1.00
5.94
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

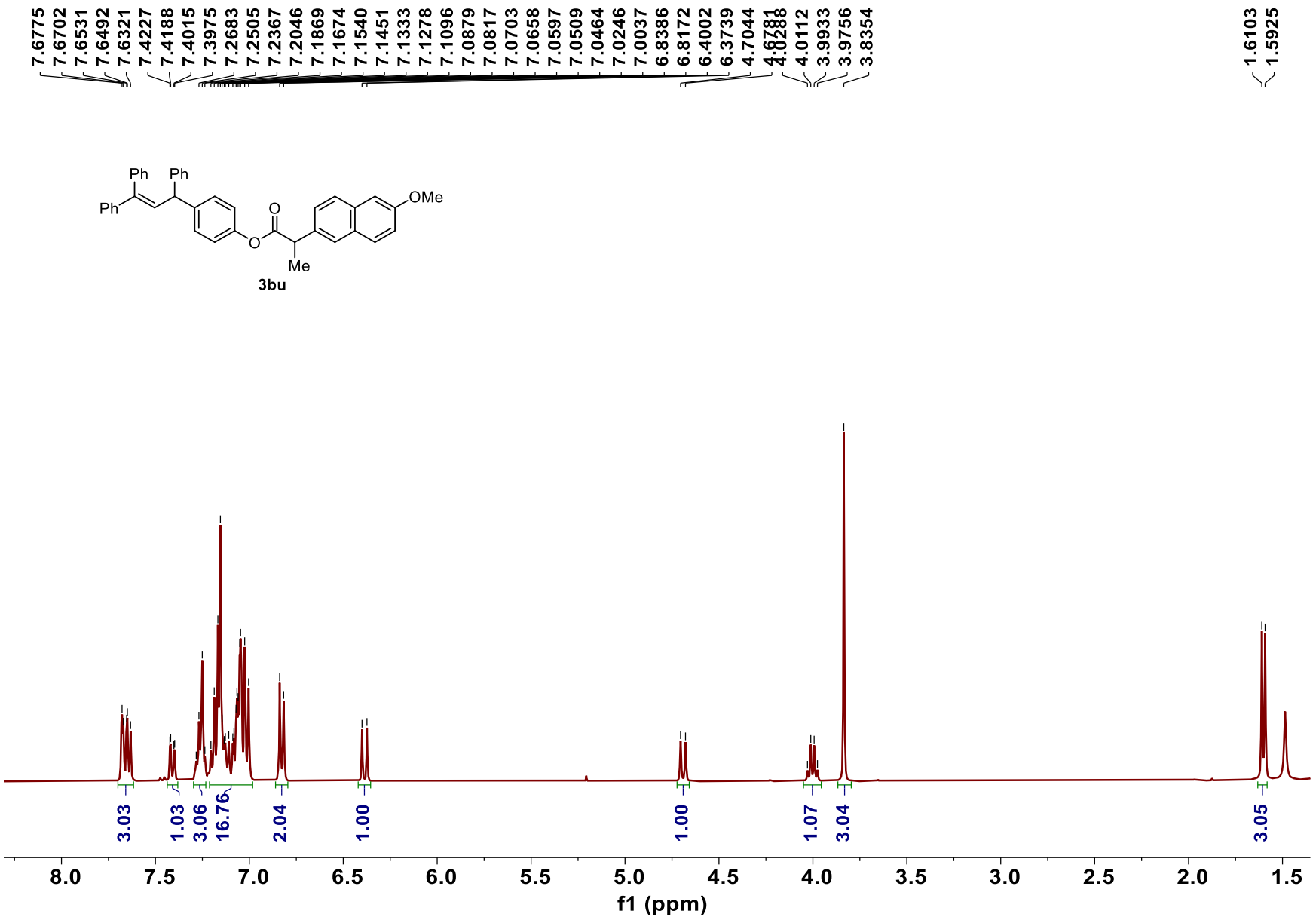

158.097
142.466
141.066
139.889
137.092
131.715
129.944
129.344
128.393
128.240
127.611
127.359
127.286
113.990
77.478
77.160
76.842
55.394
48.995
OMe
OMe
3br
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

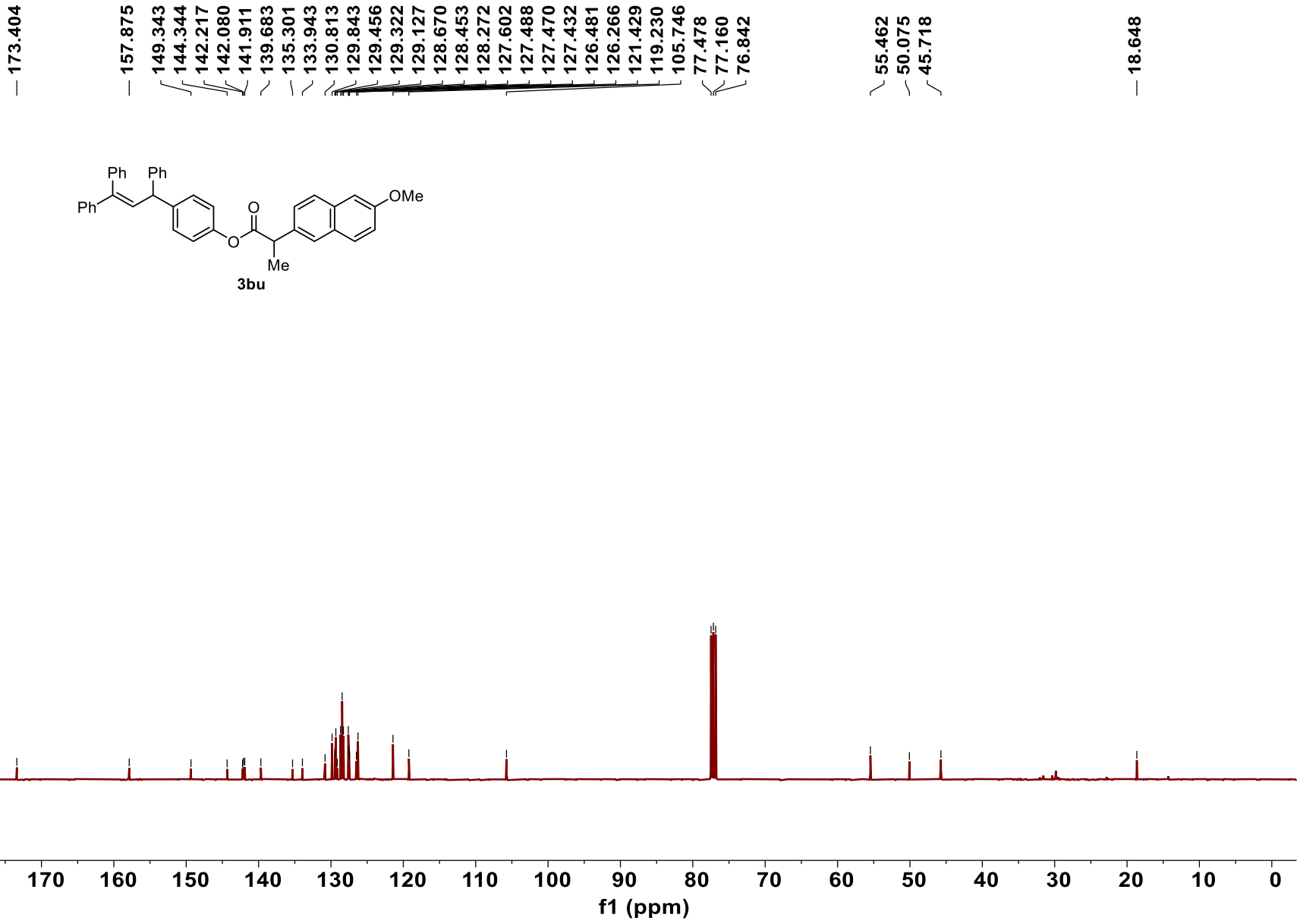

7.3996
7.3840
7.3783
7.3702
7.3651
7.3598
7.3554
7.3438
7.2686
7.2523
7.2479
7.2353
7.2291
7.2209
7.2156
7.2109
7.2049
7.1986
7.1889
7.1478
7.1428
7.1372
7.1322
7.1278
7.1234
7.1058
7.0967
7.0879
7.0801
7.0732
7.0660
7.0547
7.0472
7.0390
6.9719
6.9468
5.0366
5.0115
3.3709
3.3615
3.3492
3.3386
3.3219
3.3191
3.3060
3.2997
3.2843
3.0908
3.0755
3.0695
3.0560
3.0532
3.0366
3.0259
3.0137
-0.0000
Ph
3bs
3.08
6.19
2.16
8.11
1.07
1.00
2.09
2.07
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)

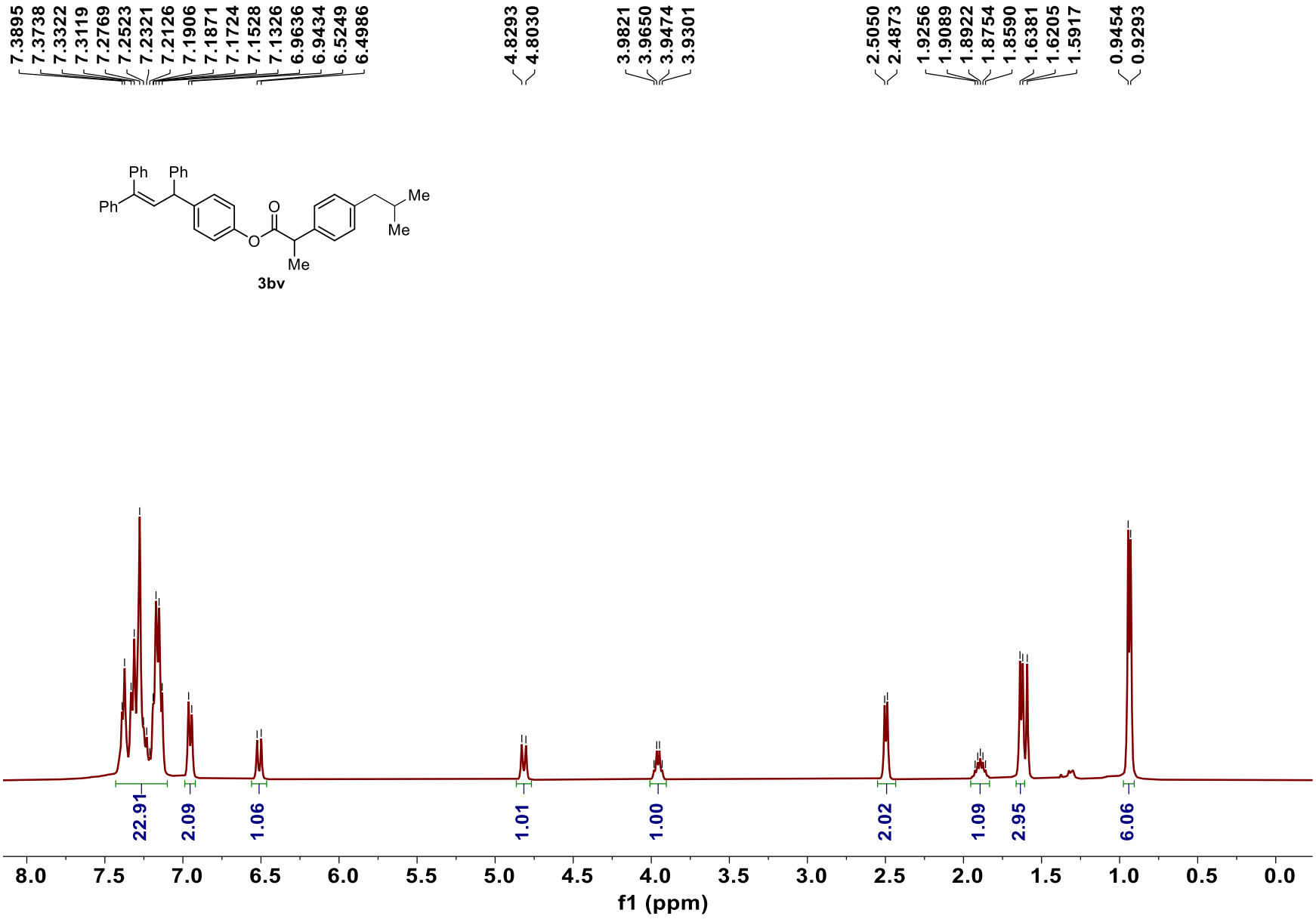

142.396
141.705
141.284
139.845
139.199
130.575
130.347
130.055
128.832
128.472
128.252
127.405
127.390
127.314
126.676
126.217
77.475
77.160
76.841
50.086
33.239
Ph
3bs
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

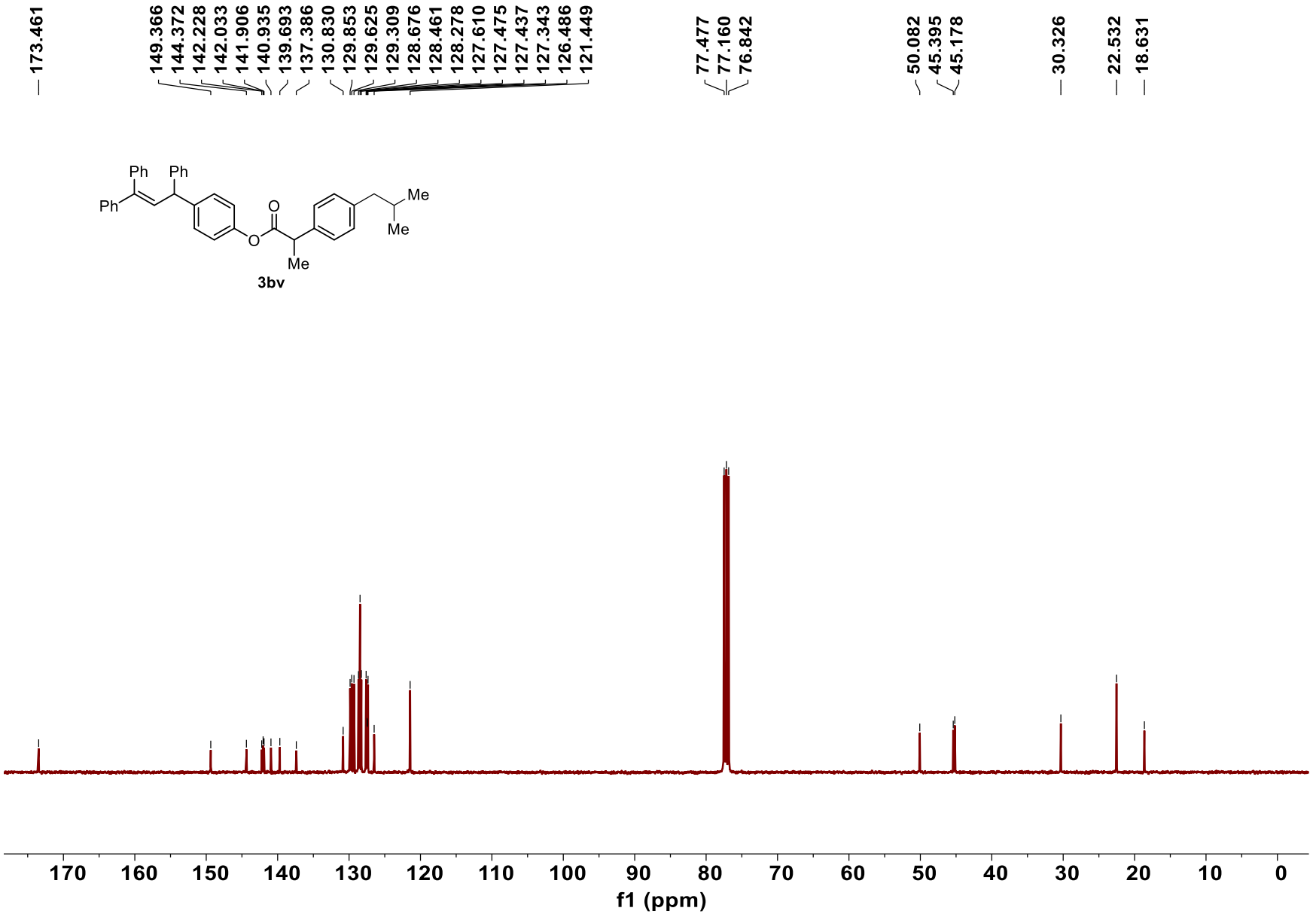

7.3939
7.3886
7.3720
7.3683
7.3577
7.3538
7.3475
7.3433
7.3312
7.3120
7.3095
7.3057
7.3028
7.2920
7.2838
7.2730
7.2659
7.2599
7.2494
7.2408
7.2322
7.2263
7.2179
7.2134
7.2077
7.2006
7.1960
7.1902
7.1848
7.1805
7.1769
7.1694
7.1657
7.1480
6.9539
6.9511
6.9478
6.9438
6.9410
6.8406
6.8376
6.8281
6.8251
6.5112
6.4849
4.8320
4.8057
0.0000
S
Ph
3bt
19.70
0.99
0.99
1.00
1.01
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)

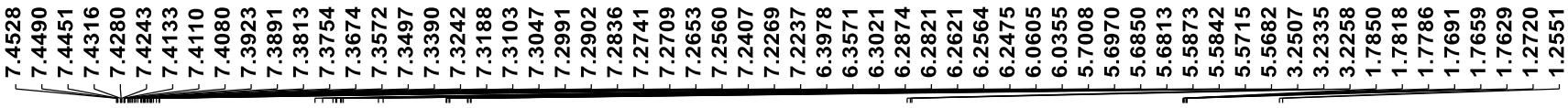

145.179
144.159
142.281
141.558
139.718
130.745
129.915
128.714
128.465
128.430
128.279
128.178
128.011
127.617
127.567
127.469
127.424
126.552
125.736
121.236
77.478
77.160
76.842
46.764
S
Ph
3bt
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

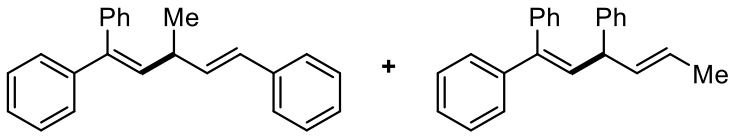

7.6775
7.6702
7.6531
7.6492
7.6321
7.4227
7.4188
7.4015
7.3975
7.2683
7.2505
7.2367
7.2046
7.1869
7.1674
7.1540
7.1451
7.1333
7.1278
7.1096
7.0879
7.0817
7.0703
7.0658
7.0597
7.0509
7.0464
7.0246
7.0037
6.8386
6.8172
6.4002
6.3739
4.7044
4.6781
4.0288
4.0112
3.9933
3.9756
3.8354
1.6103
1.5925
Ph
Ph
Ph
O
O
OMe
Me
3bu
3.03
1.03
3.06
16.76
2.04
1.00
1.00
1.07
3.04
3.05
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
f1 (ppm)


173.404
157.875
149.343
144.344
142.217
142.080
141.911
139.683
135.301
133.943
130.813
129.843
129.456
129.322
129.127
128.670
128.453
128.272
127.602
127.488
127.470
127.432
126.481
126.266
121.429
119.230
105.746
77.478
77.160
76.842
55.462
50.075
45.718
18.648
Ph
Ph
Ph
O
O
OMe
Me
3bu
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

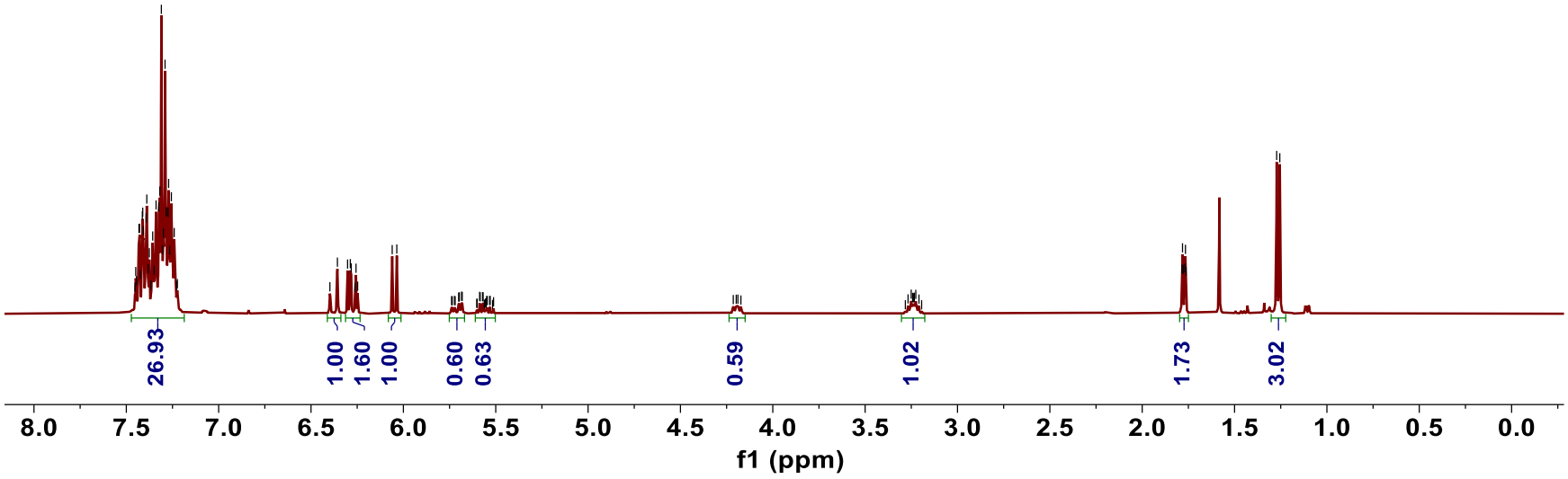

7.3895
7.3738
7.3322
7.3119
7.2769
7.2523
7.2321
7.2126
7.1906
7.1871
7.1724
7.1528
7.1326
6.9636
6.9434
6.5249
6.4986
4.8293
4.8030
3.9821
3.9650
3.9474
3.9301
2.5050
2.4873
1.9256
1.9089
1.8922
1.8754
1.8590
1.6381
1.6205
1.5917
0.9454
0.9293
Ph
Ph
Ph
O
O
Me
Me
Me
3bv
22.91
2.09
1.06
1.01
1.00
2.02
1.09
2.95
6.06
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)

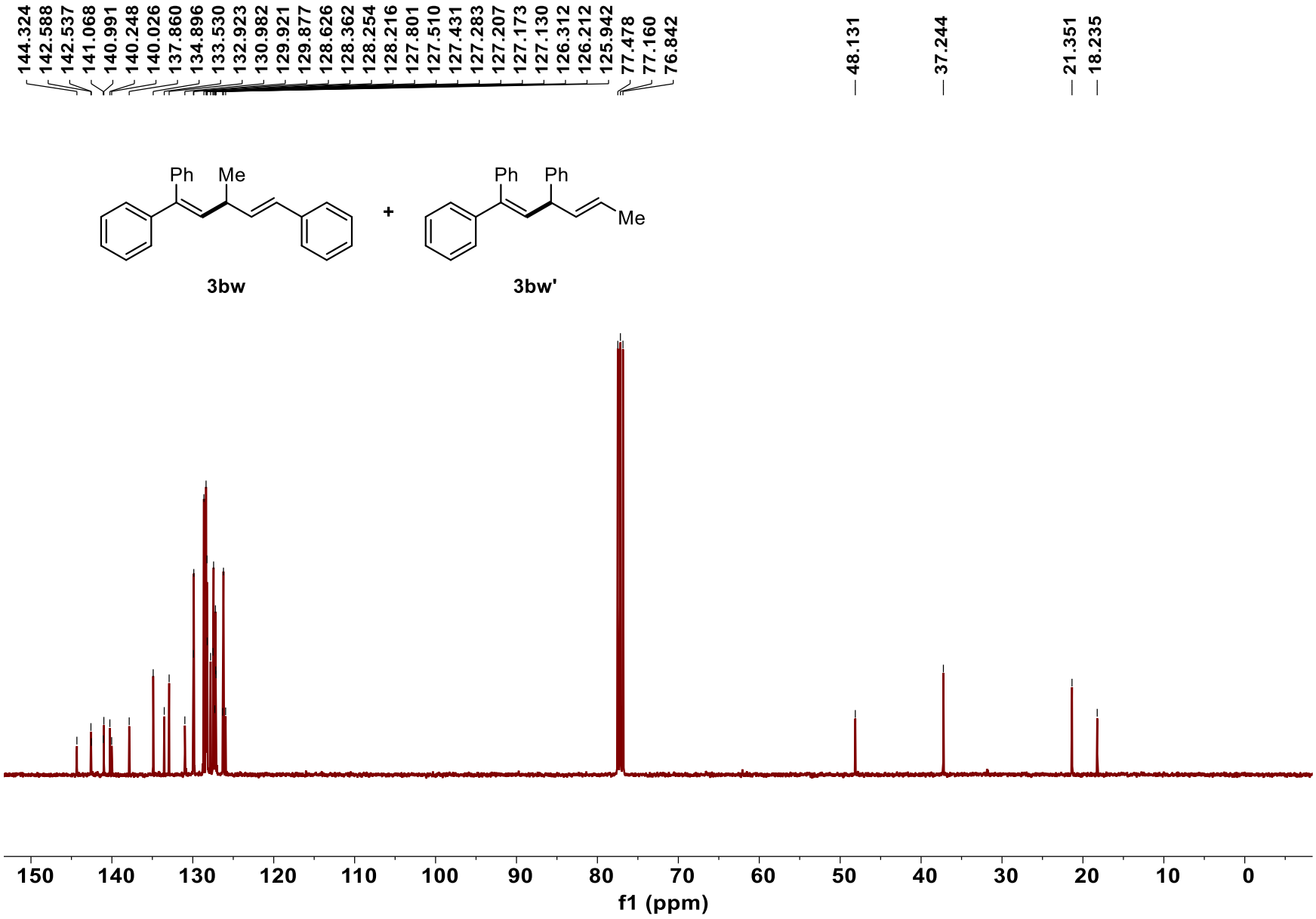

173.461
149.366
144.372
142.228
142.033
141.906
140.935
139.693
137.386
130.830
129.853
129.625
129.309
128.676
128.461
128.278
127.610
127.475
127.437
127.343
126.486
121.449
77.477
77.160
76.842
50.082
45.395
45.178
30.326
22.532
18.631
Ph
Ph
Ph
O
O
Me
Me
Me
3bv
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

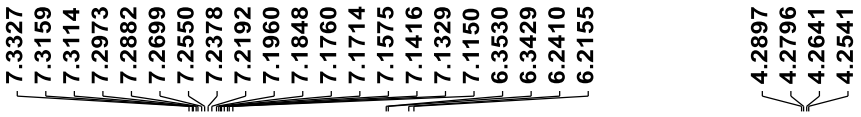

7.4528
7.4490
7.4451
7.4316
7.4280
7.4243
7.4133
7.4110
7.4080
7.3923
7.3891
7.3813
7.3754
7.3674
7.3572
7.3497
7.3390
7.3242
7.3188
7.3103
7.3047
7.2991
7.2902
7.2836
7.2741
7.2709
7.2653
7.2560
7.2407
7.2269
7.2237
6.3978
6.3571
6.3021
6.2874
6.2821
6.2621
6.2564
6.2475
6.0605
6.0355
5.7008
5.6970
5.6850
5.6813
5.5873
5.5842
5.5715
5.5682
3.2507
3.2335
3.2258
1.7850
1.7818
1.7786
1.7691
1.7659
1.7629
1.2720
1.2551

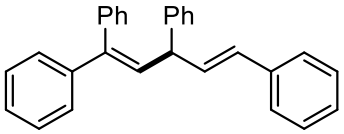

Ph
Me
+
Ph
Ph
Me

3bw **3bw'**

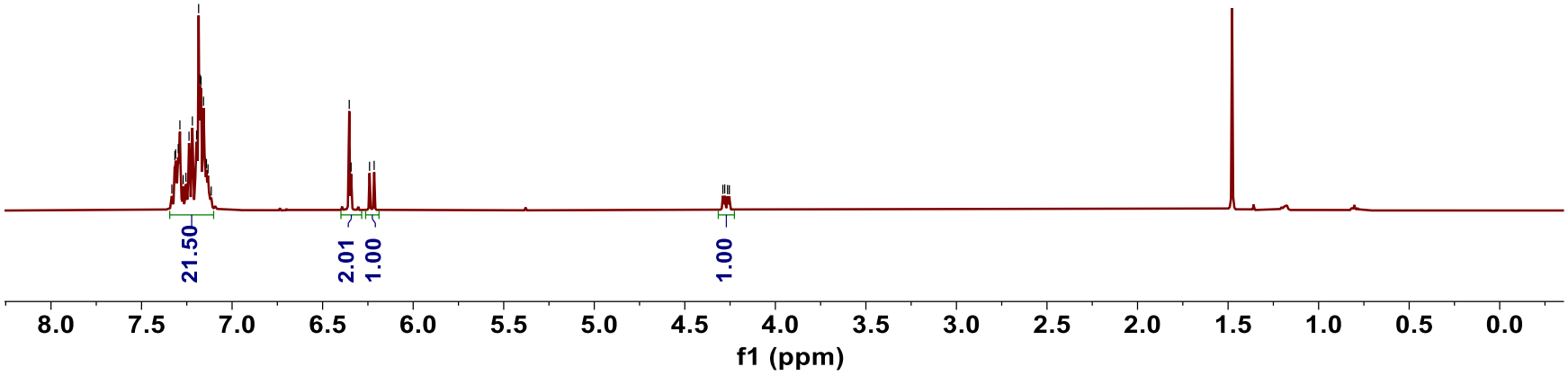

26.93
1.00
1.60
1.00
0.60
0.63
0.59
1.02
1.73
3.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

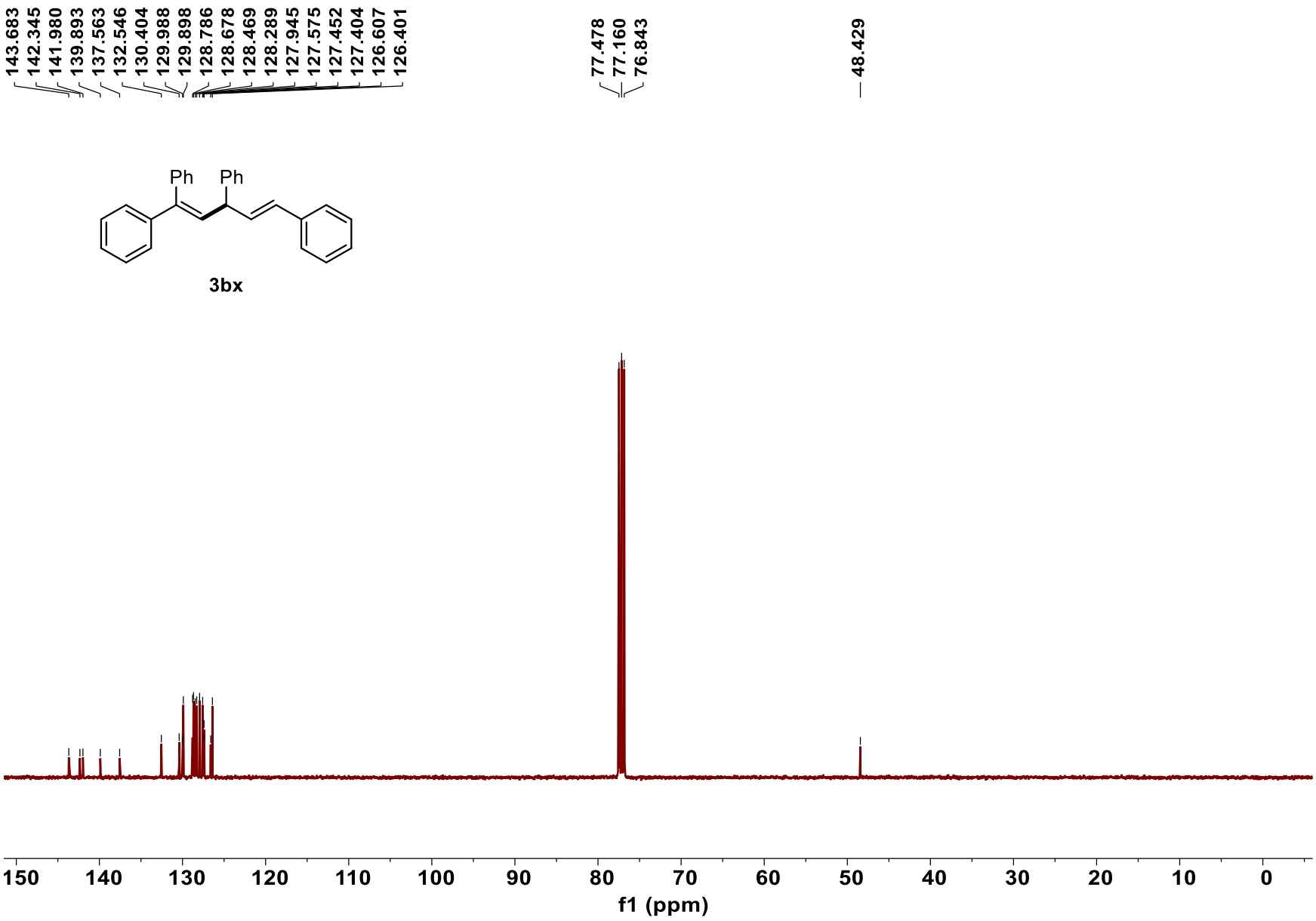

144.324
142.588
142.537
141.068
140.991
140.248
140.026
137.860
134.896
133.530
132.923
130.982
129.921
129.877
128.626
128.362
128.254
128.216
127.801
127.510
127.431
127.283
127.207
127.173
127.130
126.312
126.212
125.942
77.478
77.160
76.842
48.131
37.244
21.351
18.235
Ph
Me
+
Ph
Ph
Me
3bw
3bw'
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

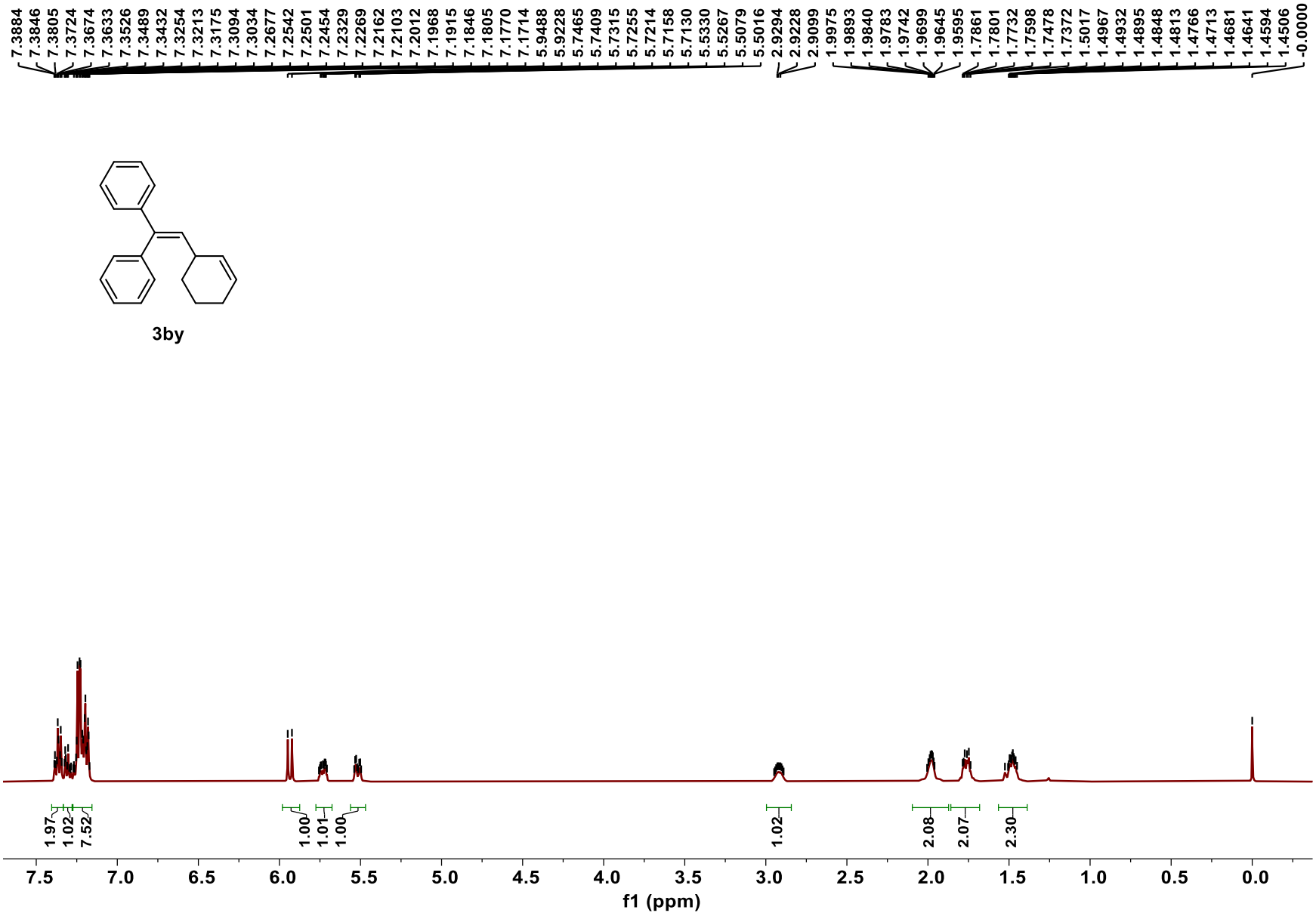

7.3327
7.3159
7.3114
7.2973
7.2882
7.2699
7.2550
7.2378
7.2192
7.1960
7.1848
7.1760
7.1714
7.1575
7.1416
7.1329
7.1150
6.3530
6.3429
6.2410
6.2155
4.2897
4.2796
4.2641
4.2541

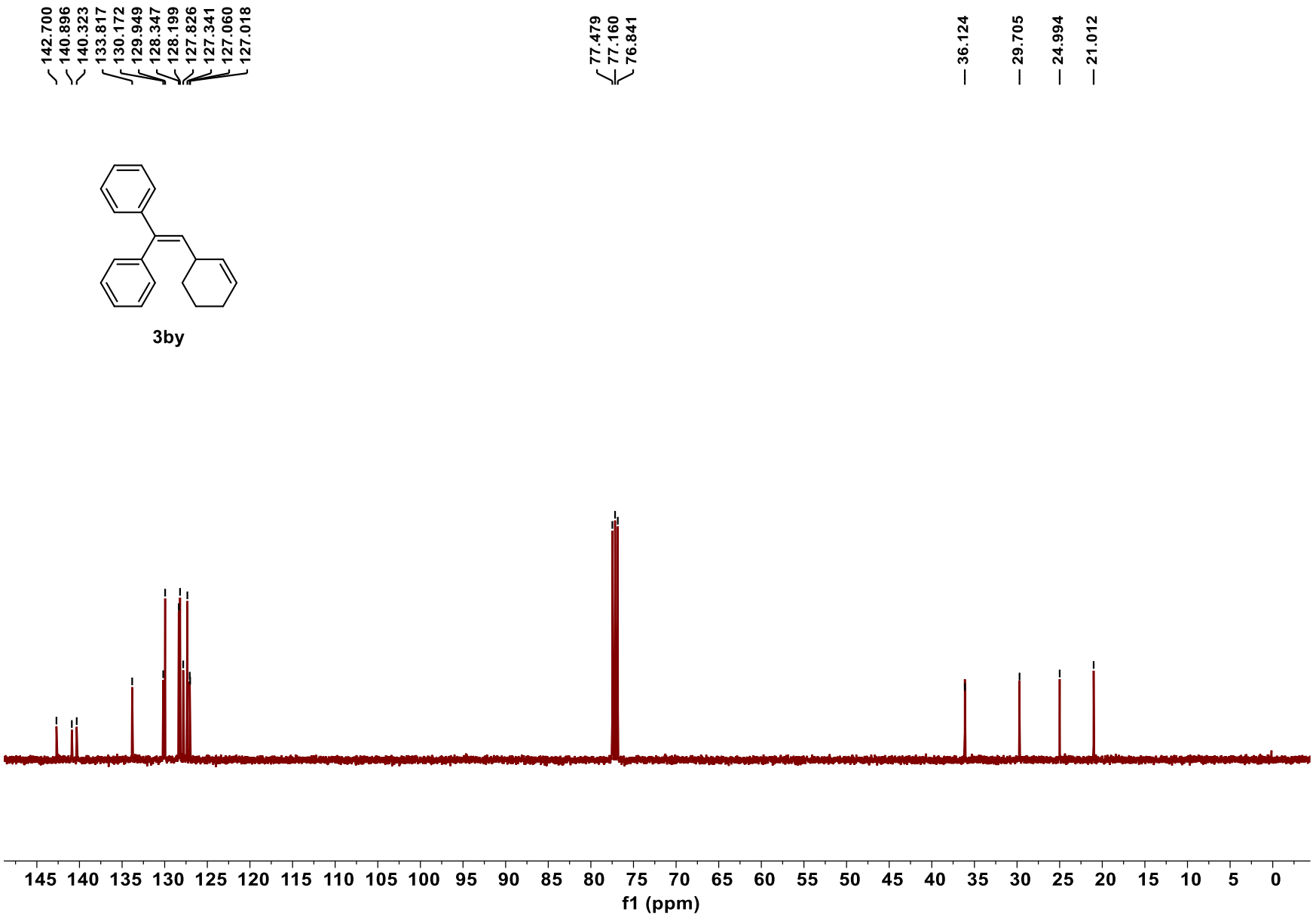

Ph
Ph

3bx


21.50
2.01
1.00
1.00
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)


143.683
142.345
141.980
139.893
137.563
132.546
130.404
129.988
129.898
128.786
128.678
128.469
128.289
127.945
127.575
127.452
127.404
126.607
126.401
77.478
77.160
76.843
48.429
Ph
Ph
3bx
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.3884
7.3846
7.3805
7.3724
7.3674
7.3633
7.3526
7.3489
7.3432
7.3254
7.3213
7.3175
7.3094
7.3034
7.2677
7.2542
7.2501
7.2454
7.2329
7.2269
7.2162
7.2103
7.2012
7.1968
7.1915
7.1846
7.1805
7.1770
7.1714
5.9488
5.9228
5.7465
5.7409
5.7315
5.7255
5.7214
5.7158
5.7130
5.5330
5.5267
5.5079
5.5016
2.9294
2.9228
2.9099
1.9975
1.9893
1.9840
1.9783
1.9742
1.9699
1.9645
1.9595
1.7861
1.7801
1.7732
1.7598
1.7478
1.7372
1.5017
1.4967
1.4932
1.4895
1.4848
1.4813
1.4766
1.4713
1.4681
1.4641
1.4594
1.4506
-0.0000
3by
1.97
1.02
7.52
1.00
1.01
1.00
1.02
2.08
2.07
2.30
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)


142.700
140.896
140.323
133.817
130.172
129.949
128.347
128.199
127.826
127.341
127.060
127.018
77.479
77.160
76.841
36.124
29.705
24.994
21.012
3by
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)

S7. References

1. G. Zhang, R.-X. Bai, C.-H. Li, C.-G. Feng, G.-Q. Lin, *Tetrahedron* **2019**, *75*, 1658–1662.
2. W.-T. Wei, F. Teng, Y. Li, R.-J. Song, J.-H. Li, *Org. Lett.* **2019**, *21*, 6285–6288.
3. K. Tanaka, M. Kishimoto, Y. Hoshino, K. Honda, *Tetrahedron Lett.* **2018**, *59*, 1841–1845.
4. T.-J. Hu, G. Zhang, Y.-H. Chen, C.-G. Feng, G.-Q. Lin, *J. Am. Chem. Soc.* **2016**, *138*, 2897–2900.
5. S. Liu, H. W. Gibson, *Tetrahedron Letters* **1994**, *35*, 8533–8536.
6. C. C. Tyrol, N. S. Yone, C. F. Gallin, J. A. Byers, *Chem. Comm.* **2020**, *56*, 14661–14664.
7. S. Auricchio, C. Magnani, A. M. Truscello, *Eur. J. Org. Chem.* **2002**, 2411–2416.