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SUPPORTING INFORMATION

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Title: HBF_4-Catalysed Nucleophilic Substitutions of Propargylic Alcohols
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1. General Considerations

All reactions were carried out in air using technical solvents without any particular precautions to exclude moisture or oxygen and all chemicals were used as supplied unless otherwise indicated. Tetrahydrofuran was dried by passing through the columns of molecular sieves in a purification system. Column chromatography and TLC were performed on silica gel, using UV light and a phosphomolybdic acid dip to visualize the products. ^{1}H NMR was recorded at 400 MHz in $CDCl_3$ using residual protic solvent $CDCl_3$ (δ=7.26 ppm, s) as internal standard and ^{13}C NMR was recorded at 101 MHz in $CDCl_3$ using $CDCl_3$ (δ=77 ppm, t) as internal standard. Chemical shifts are quoted in ppm using the following abbreviations: s, singlet; d, doublet; t, triplet; q, quartet; qn, quintet; m, multiplet; br, broad or a combination of these. Mass spectra (MS) were recorded on a Micromass Autospec Premier, Micromass LCT Premier or a VG Platform II spectrometer or on a Waters LCT Premier (ES-ToF)/Acquity i-Class spectrometer using EI or ES-ToF techniques respectively, at the Mass Spectroscopy Service of Imperial College London.

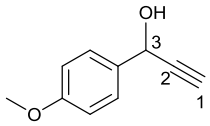
2. Preparation of Propargylic Alcohols (1)

A) General procedure for monosubstituted propargylic alcohols:

Ethynylmagnesium bromide (0.5 M in THF, 1.2 equiv) was added dropwise to a stirred solution of the corresponding aldehyde (1 equiv) in dry THF (1 M) at 0˚C under N_2 atmosphere. After 2 hours, the reaction was warmed to room temperature and stirred overnight. The resulting solution was quenched with saturated $NH_4Cl_{(aq)}$ and extracted with diethyl ether. The combined organic phases were washed with brine, dried over $MgSO_4$ and concentrated *in vacuo*. If required, the crude products were purified by flash column chromatography to give the pure propargylic alcohol **1**.

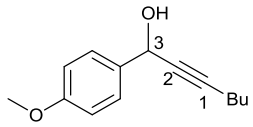
B) General procedure for disubstituted propargylic alcohols:

n-Butyllithium (2.5 M in hexane, 1.2 equiv) was added dropwise to a stirred solution of the corresponding alkyne (1 equiv) in dry THF (1 M) at -78 ˚C under N_2 atmosphere. After 30 min, the corresponding aldehyde (1 equiv) was added and the resulting solution was stirred for 5 min at 0˚C and 30 min at room temperature. The reaction mixture was then quenched with saturated $NH_4Cl_{(aq)}$ and extracted with diethyl ether. The combined organic phases were washed with brine, dried over $MgSO_4$ and concentrated *in vacuo*. If required, the crude products were purified by flash column chromatography to give the pure propargylic alcohol **1**.



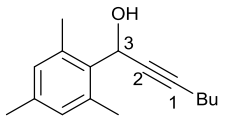
1-(4-Methoxyphenyl)prop-2-yn-1-ol (1a): Following the general procedure A, from anisaldehyde (1.5 mL, 12.4 mmol) and ethynylmagnesium bromide (31 mL, 14.8 mmol), a light brown oil was isolated (1.98 g, 12.19 mmol, 98%). Spectroscopic data for the title compound are in accordance with the literature.[1]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.48 (d, *J* = 8.9 Hz, 2H, H^{Ar}), 6.91 (d, *J* = 8.9 Hz, 2H, H^{Ar}), 5.42 (d, *J* = 2.1 Hz, 1H, CHC≡C), 3.81 (s, 3H, OMe), 2.65 (d, *J* = 2.1 Hz, 1H, C≡C–H), 2.35 (br s, 1H, OH). ^{13}C{^{1}H} NMR ($CDCl_3$, 101 MHz) δ: 159.7 (C, *C*OMe), 132.3 (CH, CH^{Ar}), 128.0 (CH, CH^{Ar}) 113.9 (C, C^{Ar}), 83.6 (C, C^2), 74.6 (CH, C^1), 63.9 (CH, C^3), 55.3 (CH_3, OMe).



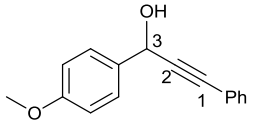
1-(4-Methoxyphenyl)hept-2-yn-1-ol (1b): Following the general procedure B, from anisaldehyde (1.36 g, 10 mmol) and hex-1-yne (1.5 mL, 13 mmol), and after purification by column chromatography (pentane/EtOAc, 90:10), a pale yellow oil was isolated (2.07 g, 95%). Spectroscopic data for the title compound are in accordance with the literature.[2]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.47 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 6.90 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 5.40 (br d, 1H, CHC≡C), 3.81 (s, 3H, OMe), 2.28 (td, *J* = 7.2; 2.0 Hz, 2H, C≡C–CH_2), 2.05 (br s, 1H, OH), 1.58–1.48 (m, 2H, C*H*$_2$CH$_2$CH$_3$), 1.48–1.38 (m, 2H, C*H*$_2$CH$_3$), 0.92 (t, *J* = 7.2 Hz, 3H, CH$_2$C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$, 101 MHz) δ: 159.5 (C, C^{Ar}), 133.7 (C, C^{Ar}), 128.1 (CH, CH^{Ar}), 113.9 (CH, CH^{Ar}), 87.4 (C, C^1), 80.1 (C, C^2), 64.4 (CH, C^3), 55.3 (CH_3, OMe), 30.7 (CH_2, C≡C*C*H$_2$), 22.0 (CH_2, *C*H$_2$CH$_2$CH$_3$), 18.5 (CH_2, *C*H$_2$CH$_3$), 13.6 (CH_3, CH$_2$*C*H$_3$).



1-Mesitylhept-2-yn-1-ol (1c): Following the general procedure B, from mesityl aldehyde (1.48 g, 10 mmol) and hex-1-yne (1.5 mL, 13 mmol), and after purification by column chromatography (cyclohexane/EtOAc, 90:10), a pale yellow oil was obtained (2.0 g, 87%).

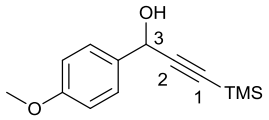
^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.84 (s, 2H, H^{Ar}), 5.90–5.88 (m, 1H, C*H*C≡C), 2.49 (s, 6H, *o*-Me^{Ar}), 2.25 (s, 3H, *p*-Me^{Ar}), 2.21 (td, *J* = 7.2; 2.0 Hz, 2H, C≡C–CH_2), 1.88 (d, *J* = 3.8 Hz, 1H, OH), 1.53–1.43 (m, 2H, C*H*$_2$CH$_2$CH$_3$), 1.42–1.34 (m, 2H, C*H*$_2$CH$_3$), 0.89 (t, *J* = 7.2 Hz, 3H, CH$_2$C*H*$_3$). ^{13}C{^{1}H} NMR ($CDCl_3$, 101 MHz) δ: 137.6 (C, C^{Ar}), 136.4 (C, C^{Ar}), 134.3 (C, C^{Ar}), 130 (CH, C^{Ar}), 86.6 (C, C^1), 79.7 (C, C^2), 60.6 (CH, C^3), 30.7 (CH_2, C≡C*C*H$_2$), 22.1 (CH_2, *C*H$_2$CH$_2$CH$_3$), 20.9 (CH_3, Me^{Ar}), 20.3 (CH_3, Me^{Ar}), 18.7 (CH_2, *C*H$_2$CH$_3$), 13.7 (CH_3, CH$_2$*C*H$_3$). HRMS (EI) calculated for $C_{16}H_{22}O$ 230.1671, found 230.1681.



1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-ol (1d): Following the general procedure B, from anisaldehyde (1.22 mL, 10 mmol) and ethynylbenzene (1.3 mL, 13 mmol), a white solid was isolated

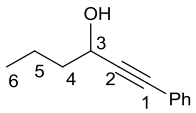
(2.34 g, 98%). Spectroscopic data for the title compound are in accordance with the literature.[3]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 7.46–7.48 (m, 2H, H^{Ar}), 7.30–7.34 (m, 3H, H^{Ar}), 6.93 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.56 (d, *J* = 6.1 Hz, 1H, CHC≡C), 3.81 (s, 3H, OMe), 2.2 (br s, 1H, OH). $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 101 MHz) δ: 159.8 (C, C^{Ar}), 133.1 (C, C^{Ar}), 131.5 (CH, CH^{Ar}), 128.6 (CH, CH^{Ar}), 128.4 (CH, CH^{Ar}), 128.3 (CH, CH^{Ar}), 122.6 (C, C^{Ar}), 114.1 (CH, CH^{Ar}), 89.5 (C, C^{2}), 86.5 (C, C^{1}), 64.8 (CH, C^{3}), 55.4 (CH_3, OMe).



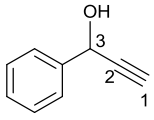
1-(4-Methoxyphenyl)-3-(trimethylsilyl)prop-2-yn-1-ol (1e): Following the general procedure B, from anisaldehyde (1.36 g, 10 mmol) and ethynyltrimethylsilane (1.84 mL, 13 mmol), the title compound was obtained, after purification by column chromatography (petroleum ether/EtOAc, 80:10), as a colourless oil (2.23 g, 95%). Spectroscopic data for the title compound are in accordance with the literature.[4]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.49 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 6.93 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 5.43 (d, *J* = 5.4 Hz, 1H, CHC≡C), 3.83 (s, 3H, OMe), 2.41 (d, *J* = 5.8 Hz, 1H, OH), 0.23 (s, 9H, TMS). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ: 159.8 (C, C^{Ar}), 132.8 (C, C^{Ar}), 128.3 (CH, CH^{Ar}), 114.0 (CH, CH^{Ar}), 105.2 (C, C^{1}), 91.4 (C, C^{2}), 64.7 (CH, C^{3}), 55.4 (CH_3, OMe), 0.0 (CH_3, TMS).



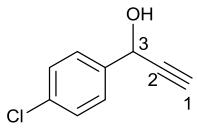
1-Phenylhex-1-yn-3-ol (1f): Following the general procedure B, from butyraldehyde (0.72 g, 10 mmol) and ethynylbenzene (1.3 mL, 13 mmol), the title compound was obtained as a colourless oil (1.65 g, 95%). Spectroscopic data for the title compound are in accordance with the literature.[5]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.46–7.40 (m, 2H, H^{Ar}), 7.33–7.28 (m, 3H, H^{Ar}), 4.61 (t, *J* = 6.6 Hz, 1H, CHC≡C), 1.83–1.73 (m, 3H, OH + H^{4}), 1.61–1.49 (m, 2H, H^{5}), 0.99 (t, *J* = 7.6 Hz, 3H, H^{6}). $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 101 MHz) δ: 131.7 (CH, CH^{Ar}), 128.2 (CH, CH^{Ar}), 127.9 (CH, CH^{Ar}), 122.9 (C, C^{Ar}), 88.6 (C, C^{2}), 85.4 (C, C^{1}), 67.5 (CH, C^{3}), 37.9 (CH_2, C^{4}), 18.6 (CH_3, C^{6}), 13.8 (CH_2, C^{5}).



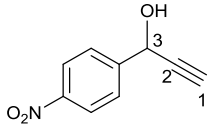
1-Phenylprop-2-yn-1-ol (1g): Following the general procedure A, from benzaldehyde (1.01 mL, 10 mmol) and ethynylmagnesium bromide (24 mL, 12 mmol), the title compound was obtained as a light brown oil (1.23 g, 92%). Spectroscopic data for the title compound are in accordance with the literature.[6]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 (dt, *J* = 8.4; 2.4 Hz, 2H, H^{Ar}), 7.43–7.32 (m, 3H, H^{Ar}), 5.48 (dd, *J* = 6.1; 2.1 Hz, 1H, CHC≡C), 2.68 (d, *J* = 2.1 Hz, 1H, C≡C–H), 2.23 (d, J = 6.1 Hz, 1H, OH). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ: 140.1 (C, C^{Ar}), 128.8 (CH, CH^{Ar}), 128.7 (C, C^{Ar}), 126.7 (CH, CH^{Ar}), 83.6 (C, C^{2}), 75.0 (CH, C^{1}), 64.5 (CH, C^{3}).



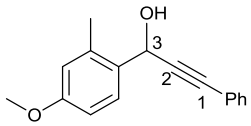
1-(4-Chlorophenyl)prop-2-yn-1-ol (1h): Following the general procedure A, from *p*-chlorobenzaldehyde (1.41 g, 10 mmol) and ethynylmagnesium bromide (24 mL, 12 mmol), the title compound was obtained as an orange oil, after purification by column chromatography (petroleum ether/EtOAc 80:10) (1.56 g, 93%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.49 (dd, *J* = 8.7; 2.0 Hz, 2H, H^{Ar}), 7.36 (dd, *J* = 8.7; 2.0 Hz, 2H, H^{Ar}), 5.44 (s, 1H, CHC≡C), 2.68 (d, *J* = 2.2 Hz, 1H, C≡C–H), 2.33 (br s, 1H, OH). ^{13}C{^{1}H} NMR ($CDCl_3$, 101 MHz) δ: 138.6 (C, C^{Ar}), 134.6 (C, C^{Ar}), 129.0 (CH, CH^{Ar}), 128.1 (CH, CH^{Ar}), 83.2 (C, C^2), 75.4 (CH, C^1), 63.8 (CH, C^3). HRMS (EI) calculated for C_9H_7ClO 166.0185, found 166.0191.



1-(4-Nitrophenyl)prop-2-yn-1-ol (1i): Following the general procedure A, from 4-nitrobenzaldehyde (1.51 g, 10 mmol) and ethynylmagnesium bromide (24 mL, 12 mmol), the title compound was isolated as an orange oil (0.89 g, 5 mmol, 50%). Spectroscopic data for the title compound are in accordance with the literature.[7]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.25 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 7.74 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.58 (s, 1H, CHC≡C), 2.74 (d, *J* = 2.1 Hz, 1H, C≡C–H), 2.53 (br s, 1H, OH). ^{13}C{^{1}H} NMR ($CDCl_3$, 101 MHz) δ: 148.0 (C, C^{Ar}), 146.8 (C, C^{Ar}), 127.5 (CH, CH^{Ar}), 124.0 (CH, CH^{Ar}), 82.4 (C, C^2), 76.1 (CH, C^1), 63.5 (CH, C^3).



1-(4-Methoxy-2-methylphenyl)-3-phenylprop-2-yn-1-ol (1j): Following the general procedure B, from 2-methyl-*p*-anisaldehyde (1.5 g, 10 mmol) and ethynylbenzene (1.3 mL, 13 mmol), the title compound was obtained as a pale yellow oil after purification by column chromatography (pentane/ethyl acetate, 90:10=>80:20) (2.0 g, 80%). Spectroscopic data for the title compound are in accordance with the literature.[3]

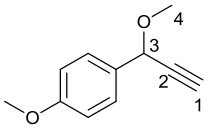
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.66 (d, *J* = 8.1 Hz, 1H, H^{Ar}), 7.49–7.45 (m, 2H, H^{Ar}), 7.33 (d, *J* = 2.1 Hz, 2H, H^{Ar}), 7.31 (d, *J* = 2.7 Hz, 1H, H^{Ar}), 6.79 (d, *J* = 2.7 Hz, 1H, H^{Ar}), 5.79 (d, *J* = 5.3 Hz, 1H, CH–C≡C), 3.81 (s, 3H, OMe), 2.49 (s, 3H, Me^{Ar}), 2.17 (d, *J* = 5.3 Hz, 1H, OH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.7 (C, C^{Ar}), 138.0 (C, C^{Ar}), 131.8 (CH, CH^{Ar}), 131.0 (C, C^{Ar}), 128.6 (CH, CH^{Ar}), 128.4 (CH, CH^{Ar}), 122.7 (C, C^{Ar}), 116.6 (CH, CH^{Ar}), 111.1 (CH, CH^{Ar}), 88.9 (C, C^2), 86.5 (C, C^1), 62.8 (CH, C^3), 55.3 (CH_3, OMe), 19.3 (CH_3, Me^{Ar}).

3. Nucleophilic Substitutions of Propargylic Alcohols

C) General procedure for the nucleophilic substitution reactions:

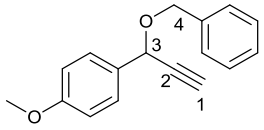
A vial fitted with a screw cap was loaded with propargylic alcohol **1** (1 mmol), technical acetone (2 mL) and nucleophile (2 mmol). Then, an HBF_4 aqueous solution (48% wt.) was added (1.24 μL, 1 mol%) and the reaction mixture was stirred at room temperature for 18 h. The resulting reaction mixture was quenched with saturated $NaHCO_{3(aq)}$ and

extracted with ethyl acetate. The combined organic phases were washed with brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure to give the title compound. If needed, the crude product was then purified by column chromatography.



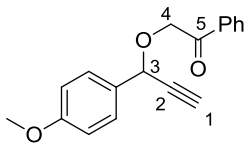
1-Methoxy-4-(1-methoxyprop-2-yn-1-yl)benzene (2a): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and methanol (0.081 mL, 2 mmol), the title compound was obtained as a pale yellow oil (0.15 g, 85%). Spectroscopic data for the title compound are in accordance with the literature.[8]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.45 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 6.91 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.05 (d, *J* = 2.2 Hz, 1H, CH–C≡C), 3.81 (s, 3H, OMe), 3.42 (s, 3H, H^4), 2.66 (d, *J* = 2.2 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.9 (C, C^{Ar}), 130.3 (C, C^{Ar}), 128.9 (CH, CH^{Ar}), 114.0 (CH, CH^{Ar}), 81.6 (C, C^2), 75.6 (CH, C^3), 72.4 (CH, C^1), 55.8 (CH_3, OMe), 55.4 (CH_3, C^4).



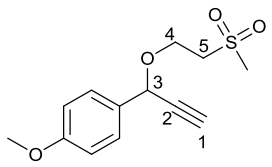
1-[1-(Benzyloxy)prop-2-yn-1-yl]-4-methoxybenzene (2b): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and benzyl alcohol (0.42 mL, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as a pale yellow oil (0.20 g, 80%). Spectroscopic data for the title compound are in accordance with the literature.[9]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.50 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 7.43–7.32 (m, 5H, H^{Ar}), 6.95 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.21 (d, *J* = 2.2 Hz, 1H, CH–C≡C), 4.71 (q, *J* = 11.7 Hz, 2H, H^4), 3.83 (s, 3H, OMe), 2.72 (d, *J* = 2.2 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.9 (C, C^{Ar}), 137.7 (C, C^{Ar}), 130.4 (C, C^{Ar}), 129.0 (CH, CH^{Ar}), 128.5 (CH, CH^{Ar}), 128.2 (CH, CH^{Ar}), 127.3 (CH, CH^{Ar}), 114.4 (CH, CH^{Ar}), 81.8 (C, C^2), 75.7 (CH, C^3), 69.9 (CH, C^1), 69.8 (CH_2, C^4), 55.4 (CH_3, OMe).



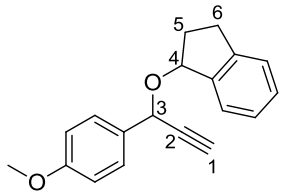
2-{[1-(4-Methoxyphenyl)prop-2-yn-1-yl]oxy}-1-phenylethan-1-one (2c): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and 2-hydroxy-1-phenylethan-1-one (0.27 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as an orange oil (0.21 g, 76%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.94–7.91 (m, 2H, H^{Ar}), 7.60–7.56 (m, 1H, H^{Ar}), 7.54 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 7.46 (t, *J* = 7.7 Hz, 2H, H^{Ar}), 6.91 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.49 (d, *J* = 2.2 Hz, 1H, CH–C≡C), 4.97 (d, *J* = 16.6 Hz, 1H, 1H^4), 4.82 (d, *J* = 16.6 Hz, 1H, 1H^4), 3.81 (s, 3H, OMe), 2.71 (d, *J* = 2.2 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 196.0 (C, CO^5), 160.2 (C, C^{Ar}), 135.1 (C, C^{Ar}), 133.7 (CH, CH^{Ar}), 129.4 (C, C^{Ar}), 129.3 (CH, CH^{Ar}), 128.8 (CH, CH^{Ar}), 128.1 (CH, CH^{Ar}), 114.1 (CH, CH^{Ar}), 80.9 (C, C^2), 76.8 (CH_2, C^4), 71.1 (CH, C^1), 69.9 (CH, C^3), 55.6 (CH_3, OMe). HRMS (ES-ToF) calculated for $C_{20}H_{19}NO_3Na$ (CH_3CN + Na adduct) 344.1263, found 344.1260.



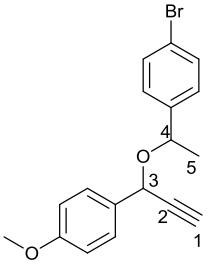
1-Methoxy-4-{1-[2-(methylsulfonyl)ethoxy]prop-2-yn-1-yl}benzene (2d): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and 2-(methylsulfonyl)ethanol (0.25 g, 2 mmol), and after purification by column chromatography (pentane/EtOAc, 40:60), the title compound was obtained as a yellow oil (0.24 g, 89%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.40 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 6.89 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 5.17 (d, *J* = 2.1 Hz, 1H, CH–C≡C), 4.13–4.02 (m, 1H, $1H^4$), 3.97–3.86 (m, 1H, $1H^4$), 3.79 (s, 3H, OMe), 3.32–3.16 (m, 2H, H^5), 2.93 (s, 3H, CH_3), 2.72 (d, *J* = 2.1 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 160.1 (C, C^{Ar}), 129.1 (C, C^{Ar}), 128.9 (CH, CH^{Ar}), 114.0 (CH, CH^{Ar}), 80.5 (C, C^2), 76.7 (CH, C^3), 71.7 (CH, C^1), 62.0 (CH_2, C^5), 55.3 (CH_2, C^4), 55.3 (CH_3, OMe), 43.0 (CH_3, Me). HRMS (EI) calculated for $C_{13}H_{17}O_4S$ 269.0848, found 269.0841.



1-{[1-(4-Methoxyphenyl)prop-2-yn-1-yl]oxy}-2,3-dihydro-1-indene (2e): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and 2,3-dihydro-1*H*-inden-1-ol (0.25 mL, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), a mixture of diastereoisomers in a 1:1 ratio of the title compound was obtained as a pale yellow oil (0.21 g, 74%).

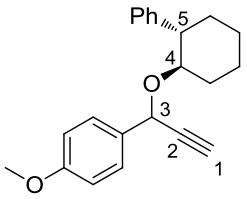
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.53–7.49 (m, 3H, H^{Ar}), 7.47–7.42 (m, 3H, H^{Ar}), 7.30 (d, *J* = 3.9 Hz, 2H, H^{Ar}), 7.27–7.22 (m, 4H, H^{Ar}), 6.92 (t, *J* = 8.9 Hz, 4H, H^{Ar}), 5.38 (t, *J* = 2.9 Hz, 1H, H^4), 5.34 (d, *J* = 2.1 Hz, 1H, CH–C≡C), 5.29 (d, *J* = 2.1 Hz, 1H, CH–C≡C), 5.27 (t, *J* = 2.9 Hz, 1H, H^4), 3.83 (s, 3H, OMe), 3.82 (s, 3H, OMe), 3.18–3.08 (m, 2H, H^6), 2.90–2.78 (m, 2H, H^6), 2.73 (d, *J* = 2.1 Hz, 1H, C≡CH), 2.69 (d, *J* = 2.1 Hz, 1H, C≡CH), 2.48–2.32 (m, 2H, H^5), 2.39–2.11 (m, 2H, H^5). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.8 (C, C^{Ar}), 144.6 (C, C^{Ar}), 143.0 (C, C^{Ar}), 131.1 (C, C^{Ar}), 128.9 (CH, CH^{Ar}), 128.6 (CH, CH^{Ar}), 126.5 (CH, CH^{Ar}), 125.4 (CH, CH^{Ar}), 125.1 (CH, CH^{Ar}), 114.0 (CH, CH^{Ar}), 82.8 (C, C^2), 81.1 (CH, C^4), 75.6 (CH, C^1), 69.4 (CH, C^3), 55.4 (CH_3, OMe), 33.6 (CH_2, C^5), 30.4 (CH_2, C^6). HRMS (EI) calculated for $C_{19}H_{19}O_2$ 279.1380, found 279.1384.



1-Bromo-4-{1-[(1-(4-methoxyphenyl)prop-2-yn-1-yl)oxy]ethyl} benzene (2f): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and 1-(4-bromophenyl)ethan-1-ol (0.40 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as a pale yellow oil (0.29 g, 86%) as a mixture of diastereoisomers in a 5:6 ratio.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.51 (t, *J* = 8.7 Hz, $4H^{maj}$, H^{Ar}), 7.36 (t, *J* = 8.7 Hz, $4H^{min}$, H^{Ar}), 7.29 (d, *J* = 8.4 Hz, $2H^{maj}$, H^{Ar}), 7.23 (d, *J* = 8.4 Hz, $2H^{min}$, H^{Ar}), 6.92 (d, *J* = 8.7 Hz, $2H^{min}$, H^{Ar}), 6.89 (d, *J* = 8.7 Hz, $2H^{maj}$, H^{Ar}), 4.95 (q, *J* = 6.5 Hz, $1H^{maj}$, C*H*CH_3), 4.93 (d, *J* = 2.3 Hz, $1H^{min}$, CH–C≡C), 4.82 (d, *J* = 2.3 Hz, $1H^{maj}$, CH–C≡C), 4.44 (q, *J* = 6.5

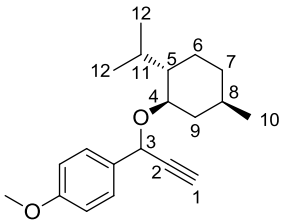
Hz, 1H^{min}, C*H*CH$_3$), 3.83 (s, 3H^{min}, OMe), 3.80 (s, 3H^{maj}, OMe), 2.63 (d, *J* = 2.3 Hz, 1H^{maj}, C≡CH), 2.57 (d, *J* = 2.3 Hz, 1H^{min}, C≡CH), 1.49 (d, *J* = 6.5 Hz, 3H^{maj}, CH$_3$), 1.42 (d, *J* = 6.5 Hz, 3H^{min}, CH$_3$). ^{13}C{^{1}H} NMR (101 MHz, CDCl$_3$) δ: 160.0 (C, C^{Ar}), 159.8 (C, C^{Ar}), 142.2 (C, C^{Ar}), 142.1 (C, C^{Ar}), 131.9 (CH, CH^{Ar}), 131.8 (CH, CH^{Ar}), 130.7 (C, C^{Ar}), 128.9 (CH, CH^{Ar}), 128.8 (CH, CH^{Ar}), 128.5 (CH, CH^{Ar}), 128.3 (CH, CH^{Ar}), 121.7 (C, C^{Ar}), 121.5 (C, C^{Ar}), 114.2 (CH, CH^{Ar}), 114.0 (CH, CH^{Ar}), 82.9 (C, C^2), 81.9 (C, C^2), 75.5 (CH, C^4), 75.2 (CH, C^4), 74.6 (CH, C^1), 74.2 (CH, C^1), 68.5 (CH, C^3), 68.2 (CH, C^3), 55.4 (CH$_3$, OMe), 24.2 (CH$_3$, Me), 23.7 (CH$_3$, Me). HRMS (EI) calculated for C$_{18}$H$_{18}$O$_2$Br 345.0485, found 345.0468.



1-Methoxy-4-{1[((2*S*)-2-phenylcyclohexyl)oxy]prop-2-yn-1-yl]benzene (2g): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **(1a)** (0.16 g, 1 mmol) and (2*S*)-2-phenylcyclohexan-1-ol (0.35 g, 2 mmol), after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as a white solid (0.28 g, 86%) as a mixture of inseparable diastereoisomers in a 2:1 ratio.

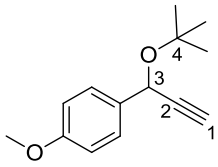
^{1}H NMR (400 MHz, CDCl$_3$) δ: 7.29 (dd, *J* = 19.5; 5.9 Hz, 12H$^{maj\ \&\ min}$, H^{Ar}), 7.13 (d, *J* = 8.5 Hz, 1H^{maj}, H^{Ar}), 6.82 (dd, *J* = 12.6; 8.5 Hz, 3H$^{maj\ \&\ min}$, H^{Ar}), 6.70 (d, *J* = 8.5 Hz, 2H^{maj}, H^{Ar}), 4.82 (s, 1H^{min}, C*H*–C≡C), 4.49 (s, 1H^{maj}, C*H*-C≡C), 3.78 (s, 3H^{min}, OMe), 3.76 (s, 3H^{maj}, OMe), 3.69 (td, *J* = 10.2; 4.5 Hz, 2H$^{maj\ \&\ min}$, CH^4), 2.71–2.54 (m, 2H$^{maj\ \&\ min}$, CH^5), 2.53 (d, *J* = 1.7 Hz, 1H^{maj}, C≡C*H*), 2.40 (d, *J* = 1.7 Hz, 1H^{min}, C≡CH), 1.97–1.27 (m, 16H$^{maj\ \&\ min}$, CH$_2$). ^{13}C{^{1}H} NMR (101 MHz, CDCl$_3$) δ: 159.5 (C, C^{Ar}), 144.7 (C, C^{Ar}), 130.7 (C, C^{Ar}), 128.8 (CH, CH^{Ar}), 128.4 (CH, CH^{Ar}), 128.2 (CH, CH^{Ar}), 126.4 (CH, CH^{Ar}), 113.5 (CH, CH^{Ar}), 83.3 (C, C^2), 82.2 (CH, C^4), 74.2 (CH, C^1), 70.8 (CH, C^3), 55.3 (CH$_3$, OMe), 51.7 (CH, C^5), 33.8 (CH$_2$), 32.1 (CH$_2$), 25.9 (CH$_2$), 25.4 (CH$_2$). HRMS (ES-ToF) calculated for C$_{19}$H$_{21}$O$_2$Na 343.1669, found 343.1658.

1-{1-[(*L*-Menthyl)oxy]prop-2-yn-1-yl}-4-methoxybenzene (2h): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and *L*-Menthol (0.35 mL, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as a pale yellow oil (0.18 g, 63%) and as a mixture of inseparable diastereoisomers in a 2:1 ratio.



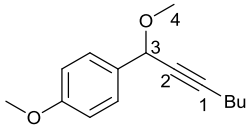
^{1}H NMR (400 MHz, CDCl$_3$) δ: 7.44–7.40 (m, 4H$^{maj\ \&\ min}$, H^{Ar}), 6.89 (d, *J* = 8.8 Hz, 4H$^{maj\ \&\ min}$, H^{Ar}), 5.24 (d, *J* = 2.1 Hz, 1H^{maj}, CH–C≡C), 5.11 (d, *J* = 2.1 Hz, 1H^{min}, CH–C≡C), 3.81 (s, 3H^{min}, OMe), 3.80 (s, 3H^{maj}, OMe), 3.60 (td, *J* = 10.5; 4.1 Hz, 1H^{maj}, H^4), 3.21 (td, *J* = 10.5; 4.1 Hz, 1H^{min}, H^4), 2.58 (d, *J* = 2.1 Hz, 1H^{min}, C≡CH), 2.55 (d, *J* = 2.1 Hz, 1H^{maj}, C≡CH), 2.41–2.26 (m, 2H$^{maj\ \&\ min}$, H^5), 2.22–2.11 (m, 2H$^{maj\ \&\ min}$, H^8), 1.73–1.53 (m, 4H$^{maj\ \&\ min}$, H^9), 1.46–1.34 (m, 2H$^{maj\ \&\ min}$, H^{11}), 1.33–1.22 (m, 2H$^{maj\ \&\ min}$, H^6), 1.04 (qd, *J* = 13.5; 3.4 Hz, 2H$^{maj\ \&\ min}$, H^6), 0.97–0.78 (m, 20H$^{maj\ \&\ min}$, H^7, CH$_3^{10}$, H^{12}), 0.50 (d, *J* = 6.9 Hz, 2H$^{maj\ \&\ min}$, H^7). ^{13}C{^{1}H} NMR (101 MHz, CDCl$_3$) δ: 159.8 (C, C^{Ar}), 159.7 (C, C^{Ar}), 131.8 (C, C^{Ar}), 131.6 (C, C^{Ar}), 129.1 (CH, CH^{Ar}), 128.7 (CH, CH^{Ar}), 114.0 (CH,

CH^{Ar}), 114.0 (CH, CH^{Ar}), 84.0 (C, C^2), 83.1 (C, C^2), 77.9 (CH, C^3), 76.6 (CH, C^3), 74.7 (CH, C^4), 74.2 (CH, C^4), 69.4 (CH, C^1), 67.6 (CH, C^1), 55.5 (CH_3, OMe), 48.6 (CH, C^5), 48.2 (CH, C^5), 41.2 (CH_2, C^9), 40.0 (CH_2, C^9), 34.7 (CH_2, C^7), 34.5 (CH_2, C^7), 31.8 (CH, C^8), 31.6 (CH, C^8), 25.3 (CH, C^{11}), 25.1 (CH, C^{11}), 23.3 (CH_2, C^6), 23.1 (CH_2, C^6), 22.6 (CH_3, C^{12}), 22.5 (CH_3, C^{12}), 21.3 (CH_3, C^{12}), 21.3 (CH_3, C^{12}), 16.3 (CH_3, C^{10}), 15.8 (CH_3, C^{10}). HRMS (ES-ToF) calculated for $C_{20}H_{29}O_3$ 317.2117, found 317.2131.



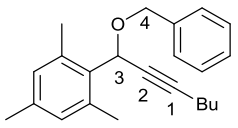
1-[1-(*tert*-Butoxy)prop-2-yn-1-yl)-4-methoxybenzene (2i): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and *tert*-butanol (0.19 mL, 2 mmol), and after purification by column chromatography (petroleum:EtOAc, 80:10), the title compound was obtained as a pale yellow oil (0.02 g, 9%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 6.88 (d, *J* = 8.5 Hz, 1H, H^{Ar}), 5.21 (d, *J* = 2.1 Hz, 1H, CH–C≡C), 3.80 (s, 3H, OMe), 2.55 (d, *J* = 2.1 Hz, 1H, C≡CH), 1.33 (s, 9H, Me). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.4 (C, C^{Ar}), 133.0 (C, C^{Ar}), 128.2 (CH, CH^{Ar}), 113.9 (CH, CH^{Ar}), 85.4 (C, C^2), 75.8 (C, C^4), 73.9 (CH, C^1), 63.6 (CH_3, OMe), 55.4 (CH, C^3), 28.5 (CH_3, *t*-Bu).



1-Methoxy-4-(1-methoxyhept-2-yn-1-yl) benzene (2j): Following the general procedure C, from 1-(4-methoxyphenyl)hept-2-yn-1-ol **1b** (0.24 g, 1 mmol) and methanol (0.081 mL, 2 mmol), the title compound was obtained as a pale yellow oil (0.19 g, 80%). Spectroscopic data for the title compound are in accordance with the literature.[8,10]

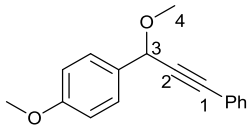
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 6.90 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.03 (t, *J* = 1.9 Hz, 1H, CH–C≡C), 3.80 (s, 3H, OMe), 3.38 (s, 3H, H^4), 1.60–1.50 (m, 2H, C≡CCH_2), 1.50–1.41 (m, 4H, CH_2C$H_2$$CH_3$), 0.93 (t, *J* = 7.3 Hz, 3H, CH_2CH_3). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.5 (C, C^{Ar}), 131.3 (C, C^{Ar}), 128.7 (CH, CH^{Ar}), 113.6 (CH, CH^{Ar}), 88.4 (C, C^1),77.7 (C, C^2), 72.7 (CH, C^3), 55.3 (CH_3, OMe), 55.2 (CH_3, OMe), 30.7 (CH_2, C≡C–CH_2), 21.9 (CH_2, $CH_2CH_2CH_3$), 18.5 (CH_2, CH_2CH_3), 13.7 (CH_3, CH_2CH_3).



2-(1-(Benzyloxy)hept-2-yn-1-yl)-1,3,5-trimethylbenzene (2k): Following the general procedure C, from 1-mesitylhept-2-yn-1-ol **1c** (0.23 g, 1 mmol) and phenylmethanol (0.42 mL, 2 mmol), after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained as a pale yellow oil (0.29 g, 90%).

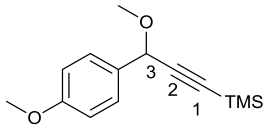
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43–7.27 (m, 5H, H^{Ar}), 6.87 (s, 2H, H^{Ar}), 5.51 (t, *J* = 2.0 Hz, 1H, CH–C≡C), 4.68 (d, *J* = 11.9 Hz, 1H, OCH_2), 4.58 (d, *J* = 11.9 Hz, 1H, OCH_2), 2.43 (s, 6H, Me^{Ar}), 2.29 (s, 3H, Me^{Ar}), 2.26 (dd, *J* = 7.1; 2.0 Hz, 2H, C≡CCH_2), 1.55 (m, 2H, C$H_2$$CH_2CH_2CH_3$), 1.50–1.40 (m, 2H, C$H_2$$CH_2CH_3$), 0.95 (t, *J* = 7.1 Hz, 3H, CH_2CH_3). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 138.3 (C, C^{Ar}), 137.5 (C, C^{Ar}), 137.1 (C, C^{Ar}), 132.5

(C, C^{Ar}), 129.9 (CH, CH^{Ar}), 128.4 (CH, CH^{Ar}), 128.2 (CH, CH^{Ar}), 127.7 (C, C^{Ar}), 87.4 (C, C^1), 77.8 (C, C^2), 69.8 (CH_2, C^4), 66.2 (CH, C^3), 30.8 (CH_2, C≡C*C*H_2), 22.2 (CH_2, *C*$H_2CH_2CH_3$), 21.0 (CH_2, *C*H_2CH_3), 20.4 (CH_3, Me^{Ar}), 18.8 (CH_3, Me^{Ar}), 13.7 (CH_3,CH_2*C*H_3). HRMS (EI) calculated for $C_{23}H_{29}O$ 321.2213, found 321.2219.



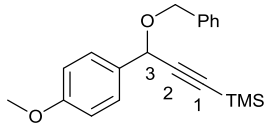
1-Methoxy-4-(1-methoxy-3-phenylprop-2-yn-1-yl)benzene (2l): Following the general procedure C, from 1-(4-methoxyphenyl)-3-phenylprop-2-yn-1-ol **1d** (0.24 g, 1 mmol) and methanol (0.081 mL, 2 mmol), the title compound was obtained as a pale yellow oil (0.22 g, 86%). Spectroscopic data for the title compound are in accordance with the literature.[11]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.58–7.51 (m, 4H, H^{Ar}), 7.38–7.32 (m, 3H, H^{Ar}), 6.96 (d, *J* = 8.8 Hz, 2H, H^{Ar}), 5.31 (s, 1H, CH–C≡C), 3.83 (s, 3H, OMe), 3.51 (s, 3H, H^4). ^{13}C{^{1}H} NMR (400 MHz, $CDCl_3$) δ: 159.7 (C, C^{Ar}), 133.0 (C, C^{Ar}), 130.7 (CH, CH^{Ar}), 128.5 (CH, CH^{Ar}), 128.3 (CH, CH^{Ar}), 128.2 (CH, CH^{Ar}), 122.4 (C, C^{Ar}), 114.0 (CH, CH^{Ar}), 87.7 (C, C^2), 86.4 (C, C^1), 73.1 (CH, C^3), 55.7 (CH_3, OMe), 55.3 (CH_3, C^4).



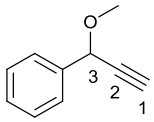
[3-Methoxy-3-(4-methoxyphenyl)prop-1-yn-1-yl]trimethylsilane (2m): Following the general procedure C, from 1-(4-methoxyphenyl)-3-(trimethylsilyl)prop-2-yn-1-ol **1e** (0.24 g, 1 mmol) and methanol (0.82 mL, 2 mmol), the title compound was obtained as a pale brown oil (0.16 g, 86%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 6.90 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.03 (s, 1H, CH–C≡C), 3.81 (s, 3H, OMe^{Ar}), 3.38 (s, 3H, OMe), 0.21 (s, 9H, TMS). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.9 (C, C^{Ar}), 130.6 (C, C^{Ar}), 129.1 (CH, CH^{Ar}), 113.9 (CH, CH^{Ar}), 103.1 (C, C^1), 92.6 (C, C^2), 73.1 (CH, C^3), 55.6 (CH_3, OMe^{Ar}), 55.4 (CH_3, OMe), 0.0 (CH_3, TMS). HRMS (EI) calculated for $C_{13}H_{17}O_2Si$ $[M\text{-}Me]^+$ 233.0992, found 233.0981.



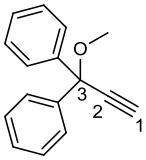
[3-(Benzyloxy)-3-(4-methoxyphenyl)prop-1-yn-1-yl]trimethylsilane (2n): Following the general procedure C, from 1-(4-methoxyphenyl)-3-(trimethylsilyl)prop-2-yn-1-ol **1e** (0.24 g, 1 mmol) and phenylmethanol (0.42 mL, 2 mmol), after purification by column chromatography (petroleum ether/EtOAc, 60:10=>40:10), the title compound was obtained as a yellow-orange oil (0.13 g, 79%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.45 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 7.42–7.26 (m, 5H, H^{Ar}), 6.90 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.17 (s, 1H, CH–C≡C), 4.65 (q, *J* = 11.7 Hz, 2H, OC*H*$_2$Ph), 3.82 (s, 3H, OMe), 0.23 (s, 9H, TMS). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.8 (C, C^{Ar}), 138.0 (C, C^{Ar}), 130.7 (C, C^{Ar}), 129.2 (CH, CH^{Ar}), 128.5 (CH, CH^{Ar}), 128.3 (CH, CH^{Ar}), 127.8 (CH, CH^{Ar}), 113.9 (CH, CH^{Ar}), 103.3 (C, C^1), 92.8 (C, C^2), 70.5 (CH, C^3), 69.8 (CH_2, OCH_2), 55.4 (CH_3, OMe), 0.1 (CH_3, TMS). HRMS (EI) calculated for $C_{20}H_{25}O_2Si$ 325.1618, found 325.1619.



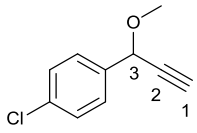
(1-Methoxyprop-2-yn-1-yl)benzene (2o): Following the general procedure C in toluene at 80°C from 1-phenylprop-2-yn-1-ol **1g** (0.12 mL, 1 mmol) and methanol (0.081 mL, 2 mmol), the title compound was obtained as an orange oil (0.10 g, 70%). Spectroscopic data for the title compound are in accordance with the literature.[12]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.53–7.51 (m, 2H, H^{Ar}), 7.40–7.34 (m, 3H, H^{Ar}), 5.10 (d, *J* = 2.1 Hz, 1H, CH–C≡CH), 3.45 (s, 3H, OMe), 2.66 (d, *J* = 2.1 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 137.9 (C, C^{Ar}), 128.8 (CH, CH^{Ar}), 128.7 (C, C^{Ar}), 126.7 (CH, CH^{Ar}), 81.3 (C, C^2), 75.8 (CH, C^3), 73.0 (CH, C^1), 55.8 (CH_3, OMe).



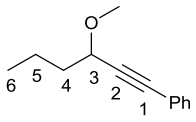
(1-Methoxyprop-2-yne-1,1-diyl)dibenzene (2p): Following the general procedure C in acetone at 30°C from 1,1-diphenyl-2-propyn-1-ol (0.17 g, 1 mmol), methanol (0.081 mL, 2 mmol) and HBF_4 (6.2 µL, 5 mol %), and after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained (0.13 g, 59%). Spectroscopic data for the title compound are in accordance with the literature.[13]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57–7.54 (m, 5H, H^{Ar}), 7.35–7.25 (m, 5H, H^{Ar}), 3.36 (s, 3H, OMe), 2.90 (s, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 144.5 (C, C^{Ar}), 127.9 (CH, CH^{Ar}), 127.8 (CH, CH^{Ar}), 127.2 (C, C^{Ar}), 126.0 (CH, CH^{Ar}), 86.5 (C, C^2), 75.6 (CH, C^3), 74.3 (CH, C^1), 52.5 (CH_3, OMe).



1-Chloro-4-(1-methoxyprop-2-yn-1-yl)benzene (2q): Following the general procedure C in toluene at 80°C from 1-(4-chlorophenyl)prop-2-yn-1-ol **(1f)** (0.17 mL, 1 mmol), methanol (0.082 mL, 2 mmol) and HBF_4 (6.2 µL, 5 mol %), and after purification by column (petroleum ether/EtOAc,80:20), the title compound was obtained as yellow solid (0.16 g, 88%). Spectroscopic data for the title compound are in accordance with the literature.[14]

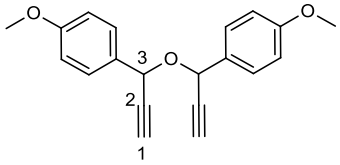
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.46 (d, *J* = 8.4 Hz, 2H, H^{Ar}), 7.35 (d, *J* = 8.4 Hz, 2H, H^{Ar}), 5.06 (d, *J* = 2.2 Hz, 1H, C*H*–C≡CH), 3.44 (s, 3H, OMe), 2.67 (d, *J* = 2.2 Hz, 1H, C≡C*H*). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 136.7 (C, C^{Ar}), 134.5 (C, C^{Ar}), 128.8 (CH, CH^{Ar}), 80.9 (C, C^2), 76.2 (CH, C^3), 72.1 (CH, C^1), 56.0 (CH_3, OMe).



(3-Methoxyhex-1-yn-1-yl)benzene (2s): Following the general procedure C in toluene at 80°C, from 1-phenylhex-1-yn-3-ol **1f** (0.17 g, 1 mmol), methanol (0.081 mL, 2 mmol) and HBF_4 (6.2 µL, 5 mol %), the title compound was obtained (0.13 g, 70%). Spectroscopic data for the title compound are in accordance with the literature.[15]

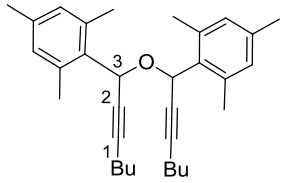
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.52–7.44 (m, 2H, H^{Ar}), 7.38–7.28 (m, 3H, H^{Ar}), 4.20 (t, *J* = 6.6 Hz, 1H, CH–C≡C), 3.49 (s, 3H, OMe), 1.81 (dt, *J* = 13.8; 6.6 Hz, 2H, H^4), 1.64–1.50 (m, 2H, H^5), 1.00 (t, *J* = 6.6 Hz, 3H, H^6). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 131.8 (C, C^{Ar}), 128.6 (CH, CH^{Ar}), 128.4 (CH, CH^{Ar}), 123.0 (CH, CH^{Ar}), 88.2 (C, C^1), 86.0 (C, C^2), 71.7 (CH, C^3), 56.5 (CH_3, OMe), 37.8 (CH_2, C^4), 18.7 (CH_3, C^6), 14.0 (CH_2, C^5).

4,4'-[Oxy-bis-(prop-2-yne-1,1-diyl)]-bis-(methoxybenzene) (3a): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.162 g, 1 mmol) and after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained as a pale yellow oil (0.105 g, 70%) and as a 10:9 mixture of diastereoisomers.



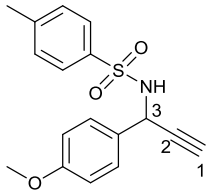
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.7 Hz, 4H^{maj}, H^{Ar}), 7.43 (d, *J* = 8.7 Hz, 4H^{min}, H^{Ar}), 6.95–6.85 (m, 8H$^{maj\ \&\ min}$, H^{Ar}), 5.56 (d, *J* = 2.0 Hz, 2H^{maj}, CH–C≡C), 5.18 (d, *J* = 2.2 Hz, 2H^{min}, CH–C≡C), 3.81 (s, 6H^{min}, OMe), 3.80 (s, 6H^{maj}, OMe), 2.70 (d, *J* = 2.0 Hz, 2H^{maj}, C≡CH), 2.65 (d, *J* = 2.2 Hz, 2H^{min}, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 160.1 (C, C^{Ar}), 159.9 (C, C^{Ar}), 130.2 (CH, CH^{Ar}), 129.9 (CH, CH^{Ar}), 129.4 (C, C^{Ar}), 129.2 (C, C^{Ar}), 114.1 (CH, CH^{Ar}), 114.0 (CH, CH^{Ar}), 81.9 (C, C^{2}), 81.5 (C, C^{2}), 76.1 (CH, C^{3}), 75.7 (CH, C^{3}), 69.0 (CH, C^{1}), 68.3 (CH, C^{1}), 55.5 (CH_3, OCH_3). HRMS (ES-ToF) calculated for $C_{20}H_{19}O_3$ 307.1334, found 307.1332.

2,2'-[Oxy-bis-(hept-2-yne-1,1-diyl)]-bis-(1,3,5-trimethyl benzene) (3b): Following the general procedure C, from 1-mesitylhept-2-yn-1-ol **1c** (0.23 g, 1 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained as a pale yellow oil (0.16 g, 73%) and a 10:7 mixture of diasteroisomers.



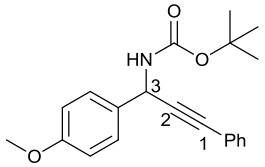
^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.82 (s, 8H$^{maj\ \&\ min}$, H^{Ar}), 5.71 (s, 2H^{maj}, CH–C≡C), 5.57 (s, 2H^{min}, CH–C≡C), 2.36 (s, 18H^{maj}, Me^{Ar}), 2.26 (s, 18H^{min}, Me^{Ar}), 2.25–2.16 (m, 8H$^{maj\ \&\ min}$, C≡CCH_2), 1.57–1.33 (m, 16H$^{maj\ \&\ min}$, CH_2C$H_2$$CH_3$), 0.90 (t, *J* = 7.2 Hz, 12H$^{maj\ \&\ min}$, CH_2CH_3). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 137.4 (CH, CH^{Ar}), 137.4 (CH, CH^{Ar}), 132.5 (C, C^{Ar}), 129.7 (CH, CH^{Ar}), 87.0 (C, C^{1}), 86.9 (C, C^{1}), 78.4 (C, C^{2}), 78.1 (C, C^{2}), 64.5 (CH, C^{3}), 63.9 (CH, C^{3}), 30.9 (CH_2, C≡CCH_2), 22.2 (CH_2, $CH_2CH_2CH_3$), 21.0 (CH_2, CH_2CH_3), 20.3 (CH_3, Me^{Ar}), 20.3 (CH_3, Me^{Ar}), 18.9 (CH_3, Me^{Ar}), 18.8 (CH_3, Me^{Ar}), 13.7 (CH_3, CH_2CH_3). HRMS (ES-ToF) calculated for $C_{32}H_{43}O$ 443.3314, found 443.3326.

***N*-[1-(4-Methoxyphenyl)prop-2-yn-1-yl]-4-methylbenzenesulfonamide (4a):** Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and 4-methylbenzenesulfonamide (0.34 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as a pale yellow solid (0.27 g, 85%). Spectroscopic data for the title compound are in accordance with the literature.[16]



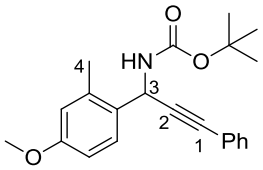
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.74 (d, *J* = 8.3 Hz, 2H, H^{Ar}), 7.34 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 7.26 (d, *J* = 8.3 Hz, 2H, H^{Ar}), 6.81 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 5.25 (dd, *J* = 8.7; 2.3 Hz, 1H, CH–C≡C), 5.17 (d, *J* = 8.7 Hz, 1H, NH), 3.77 (s, 3H, OMe), 2.42 (s, 3H, Me^{Ar}), 2.30 (d, *J* = 2.3 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.9 (C, C^{Ar}), 143.7 (C, C^{Ar}), 137.5 (C, C^{Ar}), 129.7 (CH, CH^{Ar}), 129.2 (C, C^{Ar}), 128.7 (CH, CH^{Ar}), 127.6

(CH, CH^{Ar}), 114.2 (CH, CH^{Ar}), 80.8 (C, C^2), 74.7 (CH, C^1), 55.5 (CH_3, OMe), 48.6 (CH, C^3), 21.7 (CH_3, Me^{Ar}).



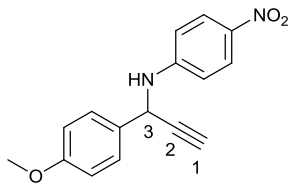
***tert*-Butyl[1-(4-methoxyphenyl)-3-phenylprop-2-yn-1-yl] carbamate (4b):** Following the general procedure C, from 1-(4-methoxyphenyl)-3-phenylprop-2-yn-1-ol **1d** (0.24 g, 1 mmol) and *tert*-butyl carbamate (0.24 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained as a white solid (0.26 g, 78%). Spectroscopic data for the title compound are in accordance with the literature.[17]

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55–7.42 (m, 4H, H^{Ar}), 7.36–7.28 (m, 3H, H^{Ar}), 6.90 (d, *J* = 8.8 Hz, 2H, H^{Ar}), 5.84 (br s, 1H, CH–C≡C), 5.10 (br s, 1H, NH), 3.81 (s, 3H, OMe), 1.48 (s, 9H, $OCCH_3$). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.4 (C, C^{Ar}), 154.9 (C, CO), 131.8 (CH, CH^{Ar}), 128.4 (CH, CH^{Ar}), 128.3 (CH, CH^{Ar}), 128.2 (CH, CH^{Ar}), 122.7 (C, C^{Ar}), 114.0 (CH, CH^{Ar}), 87.9 (C, C^2), 84.6 (C, C^1), 80.1 (C, *C*Me_3), 55.3 (CH_3, OMe), 46.3 (CH, C^3), 28.4 (CH_3, C*Me*$_3$).



***tert*-Butyl[1-(4-methoxy-2-methylphenyl)-3-phenylprop-2-yn-1-yl]carbamate (4c):** Following the general procedure C, from 1-(4-methoxy-2-methylphenyl)-3-phenylprop-2-yn-1-ol **1j** (0.25 g, 1 mmol) and *tert*-butyl carbamate (0.24 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as white solid (0.31 g, 88%).

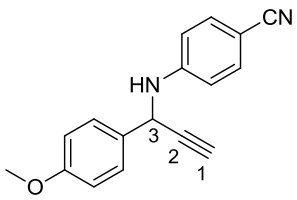
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 (d, *J* = 8.1 Hz, 1H, H^{Ar}), 7.51–7.39 (m, 2H, H^{Ar}), 7.37–7.30 (m, 3H, H^{Ar}), 6.82–6.70 (m, 2H, H^{Ar}), 5.89 (br s, 1H, CH–C≡C), 4.97 (br s, 1H, NH), 3.80 (s, 3H, OMe), 2.42 (s, 3H, H^4), 1.46 (s, 9H, OCMe). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.4 (C, C^{Ar}), 154.6 (C, CO), 137.7 (C, C^{Ar}), 131.9 (CH, CH^{Ar}), 129.8 (C, C^{Ar}), 128.4 (CH, CH^{Ar}), 128.4 (CH, CH^{Ar}), 123.0 (C, C^{Ar}), 116.6 (CH, CH^{Ar}), 111.3 (CH, CH^{Ar}), 88.2 (C, C^2), 84.3 (C, C^1), 80.2 (C, *C*Me_3), 55.4 (CH_3, OMe), 44.4 (CH, C^3), 28.5 (CH_3, C*Me*$_3$), 19.5 (CH_3, C^4). HRMS (ES-ToF) calculated for $C_{16}H_{20}NO_3Na$ 296.1263, found 296.1283.



***N*-[1-(4-Methoxyphenyl)prop-2-yn-1-yl)-4-nitroaniline (4d):** Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and *p*-nitroaniline (0.27 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as a yellow solid (0.22 g, 77%).

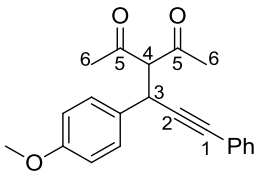
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.11 (d, *J* = 9.2 Hz, 2H, H^{Ar}), 7.49 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 6.94 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 6.69 (d, *J* = 9.2 Hz, 2H, H^{Ar}), 5.32 (dd, *J* = 6.6; 2.2 Hz, 1H, CH–C≡C), 4.79 (d, *J* = 6.6 Hz, 1H, NH), 3.83 (s, 3H, OMe), 2.54 (d, *J* = 2.2 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 160.1 (C, C^{Ar}), 151.4 (C, C^{Ar}), 139.3 (C, C^{Ar}), 129.5 (C, C^{Ar}), 128.6 (CH, CH^{Ar}), 126.3 (CH, CH^{Ar}), 114.5 (CH, CH^{Ar}), 112.5 (CH,

CH^{Ar}), 81.5 (C, C^2), 74.0 (CH, C^1), 55.5 (CH_3, OMe), 48.9 (CH, C^3). HRMS (ES-ToF) calculated for $C_{16}H_{15}N_2O_3$ 283.1083, found 283.1084.



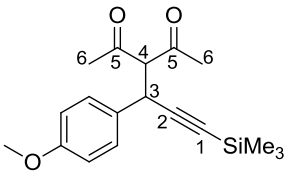
***N*-[1-(4-methoxyphenyl)prop-2-yn-1-yl]-4-aminobenzonitrile (4e):** Following the general procedure C at 60°C from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol), 4-aminobenzonitrile (0.24 g, 2 mmol), and HBF_4 (6.2 μL, 5 mol %), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as an orange solid (0.22 g, 85%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.48 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 7.46 (d, *J* = 8.8 Hz, 2H, H^{Ar}), 6.93 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 6.70 (d, *J* = 8.8 Hz, 2H, H^{Ar}), 5.26 (dd, *J* = 6.6; 2.2 Hz, 1H, CH–C≡C), 4.56 (d, *J* = 6.6 Hz, 1H, NH), 3.81 (s, 3H, OMe), 2.52 (d, *J* = 2.2 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 159.8 (C, C^{Ar}), 149.3 (C, C^{Ar}), 133.6 (CH, CH^{Ar}), 129.6 (C, C^{Ar}), 128.4 (CH, CH^{Ar}), 120.1 (C, CN), 114.3 (CH, CH^{Ar}), 113.4 (CH, CH^{Ar}), 100.3 (C, C^{Ar}) 81.8 (C, C^2), 73.7 (CH, C^1), 55.4 (CH_3, OMe), 48.6 (CH, C^3). HRMS (ES-ToF) calculated for $C_{17}H_{15}N_2O$ 263.1184, found 263.1224.



3-[1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-yl]pentane-2,4-dione (5a): Following the general procedure C, from 1-(4-methoxyphenyl)-3-phenylprop-2-yn-1-ol **1d** (0.24 g, 1 mmol) and acetylacetone (0.21 mL, 2 mmol), the title compound was obtained as a pale orange solid (0.29 g, 91%). Spectroscopic data for the title compound are in accordance with the literature.[18]

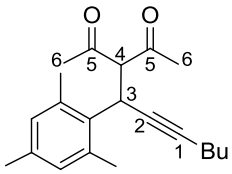
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.35–7.33 (m, 2H, H^{Ar}), 7.31 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 7.28 (dd, *J* = 5.3; 1.9 Hz, 3H, H^{Ar}), 6.86 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 4.63 (d, *J* = 11.0 Hz, 1H, CH–C≡C), 4.19 (d, *J* = 11.0 Hz, 1H, H^4), 3.81 (s, 3H, OMe), 2.41 (s, 3H, H^6), 1.97 (s, 3H, H^6). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 201.9 (C, C^5), 159.2 (C, C^{Ar}), 131.8 (CH, CH^{Ar}), 130.3 (C, C^{Ar}), 129.3 (CH, CH^{Ar}), 128.4 (C, C^{Ar}), 128.4 (CH, CH^{Ar}), 122.9 (C, C^{Ar}), 114.4 (CH, CH^{Ar}), 88.5 (C, C^2), 84.9 (C, C^1), 76.0 (CH, C^4), 55.4 (CH_3, O*Me*), 37.5 (CH_3, C^6), 31.3 (CH_3, C^6), 28.8 (CH_3, C^3).



3-[1-(4-Methoxyphenyl)-3-(trimethylsilyl)prop-2-yn-1-yl] pentane-2,4-dione (5b): Following the general procedure C, from 1-(4-methoxyphenyl)-3-(trimethylsilyl)prop-2-yn-1-ol **1e** (0.24 g, 1 mmol) and acetylacetone (0.21 mL, 2 mmol), the title compound was obtained as a yellow oil (0.13 g, 80%).

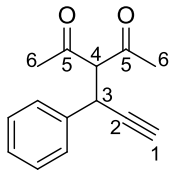
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.23 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 6.83 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 4.42 (d, *J* = 11.0 Hz, 1H, CH–C≡C), 4.07 (d, *J* = 11.0 Hz, 1H, H^4), 3.78 (s, 3H, OMe), 2.33 (s, 3H, H^6), 1.90 (s, 3H, H^6), 0.13 (s, 9H, TMS). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 201.8 (C, C^5), 201.8 (C, C^5), 159.1 (C, C^{Ar}), 129.9 (C, C^{Ar}), 129.3 (CH, CH^{Ar}), 114.3 (CH, CH^{Ar}), 105.1 (C, C^1), 89.6 (C, C^2), 76.0 (CH, C^4), 55.4 (CH_3, OMe), 37.8

(CH_3, C^6), 31.3 (CH_3, C^6), 28.9 (CH, C^3), 0.0 (CH_3, TMS). HRMS (EI) calculated for $C_{18}H_{25}O_3Si$ 317.1567, found 317.1574.



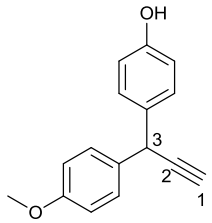
3-(1-Mesitylhept-2-yn-1-yl)pentane-2,4-dione (5c): Following the general procedure C, from 1-mesitylhept-2-yn-1-ol **1c** (0.23 g, 1 mmol) and acetylacetone (0.21 mL, 2 mmol), the title compound was obtained as a pale yellow oil (0.27 g, 87%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.80 (s, 2H, H^{Ar}), 4.87 (dt, *J* = 11.4; 2.2 Hz, 1H, CH–C≡C), 4.50 (d, *J* = 11.4 Hz, 1H, H^4), 2.56 (br s, 3H, H^6), 2.41 (br s, 3H, H^6), 2.37 (s, 3H, Me^{Ar}), 2.21 (s, 3H, Me^{Ar}), 2.10 (td, *J* = 7.0; 2.2 Hz, 2H, C≡CCH_2), 1.82 (s, 3H, Me^{Ar}), 1.47–1.29 (m, 4H, CH_2C$H_2$$CH_3$), 0.87 (t, *J* = 7.0 Hz, 3H, CH_2CH_3). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 202.4 (C, C^5), 202.0 (C, C^5), 136.9 (CH, CH^{Ar}), 136.7 (C, C^{Ar}), 136.5 (C, C^{Ar}), 131.3 (CH, CH^{Ar}), 131.2 (C, C^{Ar}), 129.7 (C, C^{Ar}), 84.0 (C, C^2), 78.0 (C, C^1), 71.8 (CH, C^4), 31.8 (CH_3, C^6), 31.0 (CH, C^3), 30.8 (CH_3, C^6), 28.8 (CH_2, C≡CCH_2), 22.0 (CH_3, Me^{Ar}), 21.1 (CH_2, $CH_2CH_2CH_3$), 20.8 (CH_3, Me^{Ar}), 20.7 (CH_2, CH_2CH_3), 18.5 (CH_3, Me^{Ar}), 13.6 (CH_3, CH_2CH_3). HRMS (EI) calculated for $C_{21}H_{27}O_2$ 311.2213, found 311.2025.



3-(1-Phenylprop-2-yn-1-yl)pentane-2,4-dione (5d): Following the general procedure C in acetonitrile at 80°C from 1-phenylprop-2-yn-1-ol **1g** (0.12 mL, 1 mmol), acetylacetone (0.21 mL, 2 mmol), and HBF_4 (6.2 μL, 5 mol %), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as a yellow-orange oil (0.11 g, 52%). Spectroscopic data for the title compound are in accordance with the literature.[19]

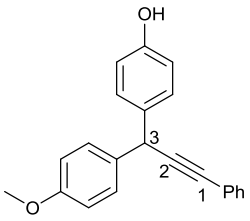
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.39–7.24 (m, 5H, H^{Ar}), 4.46 (dd, *J* = 11.0; 2.5 Hz, 1H, CH–C≡C), 4.16 (d, *J* = 11.0 Hz, 1H, H^4), 2.34 (s, 3H, H^6), 2.30 (d, *J* = 2.5 Hz, 1H, C≡CH), 1.90 (s, 3H, H^6). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 201.6 (C, C^5), 201.4 (C, C^5), 137.7 (C, C^{Ar}), 129.1 (CH, CH^{Ar}), 128.2 (CH, CH^{Ar}), 128.0 (CH, CH^{Ar}), 83.0 (C, C^2), 75.4 (CH, C^4), 73.0 (CH, C^1), 37.3 (CH_3, C^6), 31.1 (CH_3, C^6), 28.9 (CH, C^3).



4-[1-(4-Methoxyphenyl)prop-2-yn-1-yl]phenol (5e): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and phenol (0.18 mL, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained as a dark orange oil (0.15 g, 63%). Spectroscopic data for the title compound are in accordance with the literature.[20]

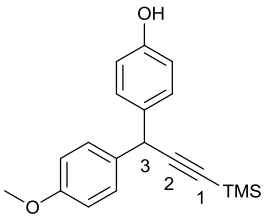
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.28 (d, *J* = 8.4 Hz, 2H, H^{Ar}), 7.24 (d, *J* = 8.4 Hz, 2H, H^{Ar}), 6.76 (d, *J* = 8.8 Hz, 2H, H^{Ar}), 6.68 (d, *J* = 8.8 Hz, 2H, H^{Ar}), 4.91 (d, *J* = 2.6 Hz, 1H, CH–C≡C), 4.82 (br s, 1H, OH), 3.78 (s, 3H, OMe), 2.48 (d, *J* = 2.6 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR ($CDCl_3$, 101 MHz) δ: 158.6 (C, C^{Ar}), 154.5 (C, C^{Ar}), 133.9 (C, C^{Ar}), 133.7

(C, C^{Ar}), 129.1 (CH, CH^{Ar}), 128.9 (CH, CH^{Ar}), 115.5 (CH, CH^{Ar}), 114.1 (CH, CH^{Ar}), 85.3 (C, C^2), 72.6 (CH, C^1), 55.4 (CH_3, OMe), 41.4 (CH, C^3).



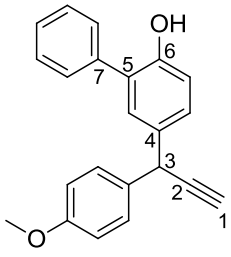
4-[1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-yl]phenol (5f): Following the general procedure C, from 1-(4-methoxyphenyl)-3-phenylprop-2-yn-1-ol **1d** (0.24 g, 1 mmol) and phenol (0.18 mL, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained as an orange oil (0.13 g, 80%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.48 (dd, *J* = 6.7; 3.0 Hz, 2H, H^{Ar}), 7.36–7.28 (m, 7H, H^{Ar}), 6.87 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 6.79 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 5.12 (s, 1H, CH–C≡C), 5.01 (br s, 1H, OH), 3.80 (s, 3H, OMe). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 158.5 (C, C^{Ar}), 154.7 (C, C^{Ar}), 134.4 (C, C^{Ar}), 134.3 (C, C^{Ar}), 131.8 (CH, CH^{Ar}), 129.1 (CH, CH^{Ar}), 128.9 (CH, CH^{Ar}), 128.3 (CH, CH^{Ar}), 128.0 (CH, CH^{Ar}), 123.7 (C, C^{Ar}), 115.5 (CH, CH^{Ar}), 114.1 (CH, CH^{Ar}), 90.9 (C, C^2), 84.7 (C, C^1), 55.4 (CH_3, OMe), 42.2 (CH, C^3). HRMS (EI) calculated for $C_{22}H_{19}O_2$ 315.1385, found 315.1385.



4-[1-(4-Methoxyphenyl)-3-(trimethylsilyl)prop-2-yn-1-yl]phenol (5g): Following the general procedure C, from 1-(4-methoxyphenyl)-3-(trimethylsilyl)prop-2-yn-1-ol **(1e)** (0.24 g, 1 mmol) and phenol (0.19 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained as a brown oil (0.11 g, 65%). Spectroscopic data for the title compound are in accordance with the literature.[20]

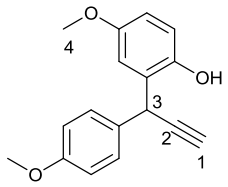
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.25 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 7.21 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 6.83 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 6.75 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 4.91 (s, 1H, CH–C≡C), 3.78 (s, 3H, OMe), 2.05 (s, 1H, OH), 0.19 (s, 9H, TMS). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 158.5 (C, C^{Ar}), 154.4 (C, C^{Ar}), 134.4 (C, C^{Ar}), 129.1 (CH, CH^{Ar}), 128.9 (CH, CH^{Ar}), 115.4 (CH, CH^{Ar}), 114.0 (CH, CH^{Ar}), 107.4 (C, C^{Ar}), 88.8 (C, C^1), 83.6 (C, C^2), 55.4 (CH_3, OMe), 42.6 (CH, C^3), 0.3 (CH_3, TMS). HRMS (EI) calculated for $C_{19}H_{23}O_2Si$ 311.1462, found 311.1470.



5-[1-(4-Methoxyphenyl)prop-2-yn-1-yl]biphenyl-2-ol (5h): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and biphenyl-2-ol (0.34 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as a brown oil (0.29 g, 92%).

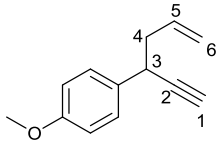
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.54–7.46 (m, 4H, H^{Ar}), 7.46–7.40 (m, 1H, H^{Ar}), 7.35 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 7.32–7.26 (m, 2H, H^{Ar}), 6.96 (d, *J* = 8.2 Hz, 1H, H^{Ar}), 6.89 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 5.28 (br s, 1H, OH), 4.99 (d, *J* = 2.6 Hz, 1H, CH–C≡C), 3.82 (s, 3H, OMe), 2.52 (d, *J* = 2.6 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 158.7 (C, C^{Ar}), 151.5 (C, C^6), 137.1 (C, C^4), 134.0 (C, C^7), 133.6 (C, C^{Ar}), 129.6 (CH, CH^{Ar}), 129.4

(CH, CH^{Ar}), 129.2 (CH, CH^{Ar}), 128.9 (CH, CH^{Ar}), 128.5 (CH, CH^{Ar}), 128.2 (C, C^5), 128.0 (CH, CH^{Ar}), 116.1 (CH, CH^{Ar}), 114.1 (CH, CH^{Ar}), 85.2 (C, C^2), 72.7 (CH, C^1), 55.4 (CH_3, OMe), 41.5 (CH, C^3). HRMS (EI) calculated for $C_{19}H_{21}O_2$ 313.1367, found 313.3664.



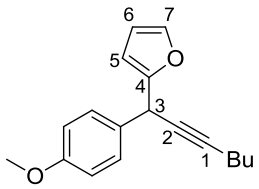
4-Methoxy-2-[1-(4-methoxyphenyl)prop-2-yn-1-yl]phenol (5i): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and 4-methoxyphenol (0.25 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained as an orange oil (0.17 g, 64%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.34 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 6.96 (d, *J* = 2.7 Hz, 1H, H^{Ar}), 6.85 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 6.74–6.68 (m, 2H, H^{Ar}), 5.21 (d, *J* = 2.7 Hz, 1H, CH–C≡C), 5.09 (br s, 1H, OH), 3.78 (s, 3H, OMe), 3.76 (s, 3H, OMe), 2.52 (d, *J* = 2.7 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 158.8 (C, C^{Ar}), 154.0 (C, C^{Ar}), 147.0 (C, C^{Ar}), 131.8 (C, C^{Ar}), 128.8 (CH, CH^{Ar}), 128.5 (C, C^{Ar}), 117.4 (CH, CH^{Ar}), 115.1 (CH, CH^{Ar}), 114.2 (CH, CH^{Ar}), 113.4 (CH, CH^{Ar}), 84.2 (C, C^2), 73.0 (CH, C^1), 55.8 (CH_3, OMe), 55.4 (CH_3, OMe), 36.9 (CH, C^3). HRMS (EI) calculated for $C_{17}H_{17}O_3$ 269.1172, found 269.1166.



1-(Hex-5-en-1-yn-3-yl)-4-methoxybencene (5j): Following the general procedure C, in MeCN at 80°C from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol), allyltrimethylsilane (0.32 mL, 2 mmol) and HBF_4 (6.2 μL, 5 mol %), and after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained as a yellow oil (0.09 g, 50%). Spectroscopic data for the title compound were consistent with the literature.[21]

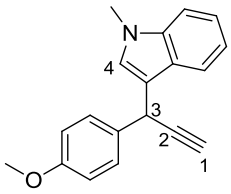
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.28 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 6.87 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 5.85 (ddt, *J* = 17.0; 10.5; 7.0 Hz, 1H, H^5), 5.13–5.01 (m, 2H, H^6), 3.80 (s, 3H, OMe), 3.67 (td, *J* = 7.0; 2.5 Hz, 1H, CH–C≡C), 2.50 (td, *J* = 7.0; 2.5 Hz, 2H, H^4), 2.30 (d, *J* = 2.5 Hz, 1H, C≡CH). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ: 158.6 (C, C^{Ar}), 135.4 (CH, C^5), 133.0 (C, C^{Ar}), 128.6 (CH, CH^{Ar}), 117.3 (CH_2, C^6), 114.0 (CH, CH^{Ar}), 85.9 (C, C^2), 71.3 (CH, C^1), 55.4 (CH_3, OMe), 42.6 (CH_2, C^4), 37.0 (CH, C^3).



2-[1-(4-Methoxyphenyl)hept-2-yn-1-yl)furan (5k): Following the general procedure C, from 1-(4-methoxyphenyl)hept-2-yn-1-ol **1b** (0.22 g, 1 mmol) and furan (0.15 mL, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:15), the title compound was obtained as a dark yellow oil (0.11 g, 41%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.35–7.29 (m, 3H, H^{Ar} + H^7), 6.86 (d, *J* = 8.8 Hz, 2H, H^{Ar}), 6.28 (dd, *J* = 3.2; 1.9 Hz, 1H, H^6), 6.17 (d, *J* = 3.2 Hz, 1H, H^5), 4.96 (s, 1H, CH–C≡C), 3.79 (s, 3H, OMe), 2.27 (td, *J* = 7.0; 2.3 Hz, 2H, C≡CCH_2), 1.58–1.50 (m, 2H, C$H_2$$CH_2CH_3$) 1.48–1.38 (m, 2H, C$H_2$$CH_3$), 0.92 (t, *J* = 7.3 Hz, 3H, CH_2CH_3).

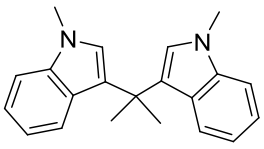
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ: 158.8 (C, C^{Ar}), 155.2 (C, C^4), 142.1 (CH, C^7), 131.9 (C, C^{Ar}), 128.9 (CH, CH^{Ar}), 114.0 (CH, CH^{Ar}), 110.3 (CH, C^6), 106.1 (CH, C^5), 84.2 (C, C^1), 78.3 (C, C^2), 55.4 (CH_3, OMe), 36.7 (CH, *C*H–C≡C), 31.1 (CH_2, C≡C*C*H_2), 22.4 (CH_2, *C*$H_2CH_2CH_3$), 18.7 (CH_2, *C*H_2CH_3), 13.8 (CH_3, CH_2*C*H_3). HRMS (ES-ToF) calculated for $C_{18}H_{21}O_2$ 269.1542, found 269.1537.



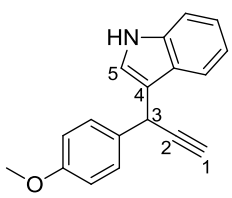
3-[1-(4-Methoxyphenyl)prop-2-yn-1-yl]-1-methyl-1*H*-indole (5l): Following the general procedure C in DCM, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and 1-methyl indole (0.25 mL, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as a brown oil (0.20 g, 74%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.52 (d, *J* = 8.2 Hz, 1H, H^{Ar}), 7.39 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 7.29 (d, *J* = 8.2 Hz, 1H, H^{Ar}), 7.22 (t, *J* = 7.0 Hz, 1H, H^{Ar}), 7.06 (t, *J* = 7.0 Hz, 1H, H^{Ar}), 6.96 (s, 1H, H^{Ar}), 6.86 (d, *J* = 8.7 Hz, 2H, H^{Ar}), 5.22 (d, *J* = 2.6 Hz, 1H, CH–C≡C), 3.79 (s, 3H, OMe), 3.75 (s, 3H, NMe), 2.43 (d, *J* = 2.6 Hz, 1H, C≡CH). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ: 158.6 (C, C^{Ar}), 137.6 (C, C^{Ar}), 133.1 (C, C^{Ar}), 128.9 (CH, CH^{Ar}), 127.4 (CH, CH^{Ar}), 126.4 (C, C^{Ar}), 121.9 (CH, CH^{Ar}), 119.7 (CH, CH^{Ar}), 119.2 (CH, CH^{Ar}), 115.1 (C, C^{Ar}), 114.0 (CH, C^4), 109.5 (CH, CH^{Ar}), 85.5 (C, C^2), 71.1 (CH, C^1), 55.4 (CH_3, OMe), 33.9 (CH, C^3), 32.8 (CH_3, N–Me). HRMS (ES-ToF) calculated for $C_{19}H_{18}NO$ 276.1388, found 276.1383.

The reaction using acetone as solvent also gave byproduct **6**, (0.041 g, 0.13 mmol, 13% yield with respect to 1-methylindole) as an off white solid. Spectroscopic data for the title compound were consistent with the literature.[22]



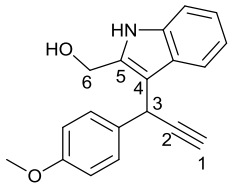
^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.52 (d, *J* = 8.2 Hz, 2H, H^{Ar}), 7.32 (d, *J* = 8.2 Hz, 2H, H^{Ar}), 7.19 (t, *J* = 7.6 Hz, 2H, H^{Ar}), 6.98 (t, *J* = 7.6 Hz, 2H, H^{Ar}), 6.95 (s, 2H, H^{Ar}), 3.78 (s, 6H, N–CH_3), 1.98 (s, 6H, C–CH_3). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ: 137.9 (C, C^{Ar}), 126.8 (C, C^{Ar}), 125.6 (CH, CH^{Ar}), 124.2 (C, C^{Ar}), 121.6 (CH, CH^{Ar}), 121.0 (CH, CH^{Ar}), 118.2 (CH, CH^{Ar}), 109.2 (CH, CH^{Ar}), 35.04 (C, *C*$(Me)_2$), 32.72 (CH_3, N–Me), 30.45 (CH_3, C–*Me*).



3-[1-(4-Methoxyphenyl)prop-2-yn-1-yl]-1*H*-indole (5m): Following the general procedure C in DCM, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and indole (0.23 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:10), the title compound was obtained as an orange oil (0.18 g, 70%).

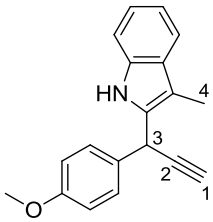
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.01 (br s, 1H, NH), 7.52 (d, *J* = 8.2 Hz, 1H, H^{Ar}), 7.4 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 7.36 (d, *J* = 8.2 Hz, 1H, H^{Ar}), 7.2 (t, *J* = 7.6 Hz, 1H, H^{Ar}), 7.13 (d, *J* = 2.2 Hz, 1H, H^{Ar}), 7.08 (t, *J* = 7.6 Hz, 1H, H^{Ar}), 6.88 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.23 (s, *J* = 2.4 Hz, 1H, CH–C≡C), 3.79 (s, 3H, OMe), 2.44 (d, *J* = 2.4 Hz, 1H, C≡CH).

$^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 101 MHz) δ: 158.6 (C, C^{Ar}), 136.8 (C, C^{Ar}), 133.0 (C, C^{Ar}), 128.9 (CH, CH^{Ar}), 122.4 (CH, CH^{Ar}), 120.6 (CH, CH^{Ar}), 119.7 (CH, C^5), 118.7 (CH, CH^{Ar}), 116.3 (C, C^{Ar}), 114.0 (CH, CH^{Ar}), 111.4 (C, C^4), 111.2 (CH, CH^{Ar}), 85.3 (C, C^2), 71.2 (CH, C^1), 55.4 (CH_3, OMe), 34.0 (CH, C^3). HRMS (ES-ToF) calculated for $C_{18}H_{16}NO$ 262.1232, found 262.1235.



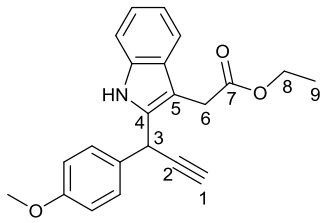
{3-[1-(4-Methoxyphenyl)prop-2-yn-1-yl]indol-2-yl}methanol (5n): Following the general procedure C, in toluene at 80°C from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol), (indol-2-yl)methanol (0.30 g, 2 mmol) and HBF_4 (6.2 μL, 5 mol %), and after purification by column chromatography (petroleum ether/EtOAc, 40:10), the title compound was obtained as a brown oil (0.02 g, 8%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.34 (br s, 1H, NH), 7.59 (d, *J* = 8.0 Hz, 1H, H^{Ar}), 7.37 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 7.34 (d, *J* = 8.0 Hz, 1H, H^{Ar}), 7.18 (t, *J* = 7.6 Hz, 1H, H^{Ar}), 7.08 (t, *J* = 7.6 Hz, 1H, H^{Ar}), 6.82 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.36 (d, *J* = 2.4 Hz, 1H, CH–C≡C), 4.91 (d, *J* = 13.7 Hz, 1H, $1H^6$), 4.77 (d, *J* = 13.7 Hz, 1H, $1H^6$), 3.77 (s, 3H, OMe), 2.45 (d, *J* = 2.7 Hz, 1H, C≡CH). Compound **5n** decomposed rapidly after purification and only ^{1}H NMR could be recorded.



2-[1-(4-Methoxyphenyl)prop-2-yn-1-yl]-3-methyl-1*H*-indole (5o): Following the general procedure C, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol) and 3-methylindole (0.26 g, 2 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as an orange oil (0.16 g, 58%).

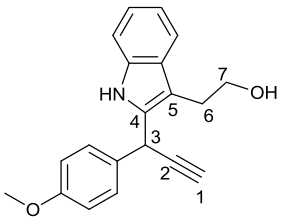
^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.03 (s, 1H, NH), 7.57 (d, *J* = 7.9 Hz, 1H, H^{Ar}), 7.35 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 7.31 (d, *J* = 7.9 Hz, 1H, H^{Ar}), 7.21 (t, *J* = 7.5 Hz, 1H, H^{Ar}), 7.15 (t, *J* = 7.5 Hz, 1H, H^{Ar}), 6.89 (d, *J* = 8.6 Hz, 2H, H^{Ar}), 5.35 (d, *J* = 2.5 Hz, 1H, CH–C≡C), 3.83 (s, 3H, OMe), 2.55 (d, *J* = 2.5 Hz, 1H, C≡CH), 2.33 (s, 3H, Me^4). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ: 159.0 (C, C^{Ar}), 135.4 (C, C^{Ar}), 132.2 (C, C^{Ar}), 131.0 (C, C^{Ar}), 129.4 (C, C^{Ar}), 128.6 (CH, CH^{Ar}), 122.0 (CH, CH^{Ar}), 119.4 (CH, CH^{Ar}), 118.7 (CH, CH^{Ar}), 114.2 (CH, CH^{Ar}), 110.9 (CH, CH^{Ar}), 108.0 (C, C^{Ar}), 82.8 (C, C^2), 72.6 (CH, C^1), 55.3 (CH_3, OMe), 33.7 (CH, C^3), 8.5 (CH_3, C^4). HRMS (ES-ToF) calculated for $C_{19}H_{18}NO$ 276.1388, found 276.1396.



Ethyl 2-(2-(1-(4-methoxyphenyl)prop-2-yn-1-yl)-1*H*-indol-3-yl)acetate (5p): Following the general procedure C in DCM, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol), (indol-2-yl)methanol (0.30 g, 2 mmol) and HBF_4 (6.2 μL, 2.5 mol %), and after purification by preparative TLC (petroleum ether/DCM, 50:50), the title compound was obtained as an orange oil (0.11 g, 30%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.08 (br s, 1H, NH), 7.59 (d, *J* = 7.7 Hz, 1H, H^{Ar}), 7.34 (d, *J* = 8.5 Hz, 1H, H^{Ar}), 7.29 (d, *J* = 7.7 Hz, 2H, H^{Ar}), 7.17 (td, *J* = 7.7; 2.1 Hz, 1H, H^{Ar}),

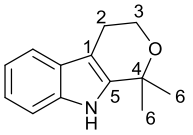
7.12 (td, *J* = 7.7; 2.1 Hz, 1H, H^{Ar}), 6.86 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 5.42 (d, *J* = 2.5 Hz, 1H, CH–C≡C), 4.10 (qd, *J* = 7.1; 1.6 Hz, 2H, H^{8}), 3.79 (s, 3H, OMe), 3.71 (dd, *J* = 36.1; 15.6 Hz, 2H, H^{6}), 2.51 (d, *J* = 2.5 Hz, 1H, C≡CH), 1.22 (t, *J* = 7.1 Hz, 3H, H^{9}). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ: 171.7 (C, CO), 159.1 (C, C^{Ar}), 135.2 (C, C^{Ar}), 134.0 (C, 4C), 130.5 (C, C^{Ar}), 128.8 (CH, CH^{Ar}), 128.6 (C, C^{Ar}), 122.3 (CH, CH^{Ar}), 120.0 (CH, CH^{Ar}), 118.9 (CH, CH^{Ar}), 114.3 (CH, CH^{Ar}), 111.0 (CH, CH^{Ar}), 105.4 (C, C^{5}), 82.7 (C, C^{2}), 73.1 (CH, C^{1}), 61.0 (CH_2, C^{8}), 55.5 (CH_3, OMe), 34.0 (CH, C^{3}), 30.5 (CH_2, C^{6}), 14.3 (CH_3, C^{9}). HRMS (EI) calculated for $C_{22}H_{22}O_3N$ 348.1594, found 348.1598.



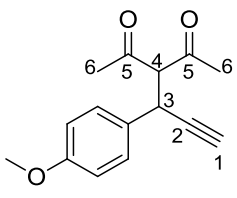
2-{2-[1-(4-Methoxyphenyl)prop-2-yn-1-yl]indol-3-yl}ethan-1-ol (5q): Following the general procedure C in DCM, from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (0.16 g, 1 mmol), tryptophol (0.32 g, 2 mmol), and HBF_4 (6.2 μL, 2.5 mol %), and after purification by column chromatography (petroleum ether/EtOAc, 2:1), the title compound was obtained as an orange oil (0.18 g, 60%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.12 (br s, 1H, NH), 7.58 (d, *J* = 7.8 Hz, 1H, H^{Ar}), 7.32 (t, *J* = 9.0 Hz, 3H, H^{Ar}), 7.21–7.16 (m, 1H, H^{Ar}), 7.15–7.10 (m, 1H, H^{Ar}), 6.87 (d, *J* = 9.0 Hz, 2H, H^{Ar}), 5.38 (d, *J* = 2.5 Hz, 1H, CH–C≡C), 3.84 (t, *J* = 6.3 Hz, 2H, H^{7}), 3.79 (s, 3H, OMe), 3.14–2.95 (m, 2H, H^{6}), 2.51 (d, *J* = 2.5 Hz, 1H, C≡CH), 1.61 (br s, 1H, OH). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ: 159.1 (C, C^{Ar}), 135.6 (C, C^{Ar}), 134.1 (C, C^{4}), 130.8 (C, C^{Ar}), 128.6 (CH, CH^{Ar}), 122.3 (CH, CH^{Ar}), 119.8 (CH, CH^{Ar}), 118.8 (CH, CH^{Ar}), 114.3 (CH, CH^{Ar}), 111.1 (CH, CH^{Ar}), 108.6 (C, C^{5}), 83.0 (C, C^{2}), 73.1 (CH, C^{1}), 62.8 (CH_2, C^{7}), 55.5 (CH_3, OMe), 33.9 (CH, C^{3}), 27.8 (CH_2, C^{6}). HRMS (ES-ToF) calculated for $C_{20}H_{20}NO_2$ 306.1494, found 306.1504.

In the reaction using acetone as solvent the initial tryptophol reacted with the solvent, giving by-product **7** in 70% yield with respect with tryptophol (0.28 g, 1.4 mmol), as well as product **5q** in 40% yield with respect to the propargylic alcohol (0.12 g, 0.4 mmol, yellow solid). Spectroscopic data for **7** were consistent with the literature.[23]



^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85 (br s, 1H, NH), 7.55 (d, *J* = 7.5 Hz, 1H, H^{Ar}), 7.34 (d, *J* = 7.5 Hz, 1H, H^{Ar}), 7.21 (td, *J* = 7.5: 1.2 Hz, 1H, H^{Ar}), 7.16 (td, *J* = 7.5; 1.2 Hz, 1H, H^{Ar}), 4.09 (t, *J* = 5.5 Hz, 2H, H^{3}), 2.85 (t, *J* = 5.5 Hz, 2H, H^{2}), 1.60 (s, 6H, H^{6}). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ: 139.0 (C, C^{5}), 135.8 (C, C^{Ar}), 127.1 (C, C^{Ar}), 121.8 (CH, CH^{Ar}), 119.7 (CH, CH^{Ar}), 118.4 (CH, CH^{Ar}), 111.0 (CH, CH^{Ar}), 106.6 (C, C^{1}), 72.4 (C, C^{4}), 60.6 (CH_2, C^{3}), 27.9 (CH_3, C^{6}), 22.5 (CH_2, C^{2}).



3-[1-(4-Methoxyphenyl)prop-2-yn-1-yl]pentane-2,4-dione (5r): Following the general procedure C from 1-(4-methoxyphenyl)prop-2-yn-1-ol **1a** (1 g, 6.16 mmol) and acetylacetone (1.26 mL, 13.23 mmol), and after purification by column chromatography (petroleum ether/EtOAc, 80:20), the title compound was obtained as a yellow oil (1.38 g, 5.65 mmol, 92%).

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.23 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 6.82 (d, *J* = 8.5 Hz, 2H, H^{Ar}), 4.39 (dd, *J* = 11.0; 2.0 Hz, 1H, CH–C≡C), 4.11 (d, *J* = 11.0 Hz, 1H, H^4), 3.76 (s, 3H, OMe) 2.31 (s, 3H, H^6), 2.29 (d, *J* = 2.0 Hz, 1H, C≡CH), 1.89 (s, 3H, H^6). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ: 201.5 (C, C^5), 201.4 (C, C^5), 159.1 (C, C^{Ar}), 129.1 (CH, CH^{Ar}), 127.7 (C, C^{Ar}), 114.3 (CH, CH^{Ar}), 83.2 (C, C^2), 75.4 (CH, C^4), 72.7 (CH, C^1), 36.4 (CH_3, C^6), 31.0 (CH_3, C^6), 28.9 (CH, C^3). HRMS (ES-ToF) calculated for $C_{15}H_{17}O_3$ 245.1178, found 245.1174.

4. References

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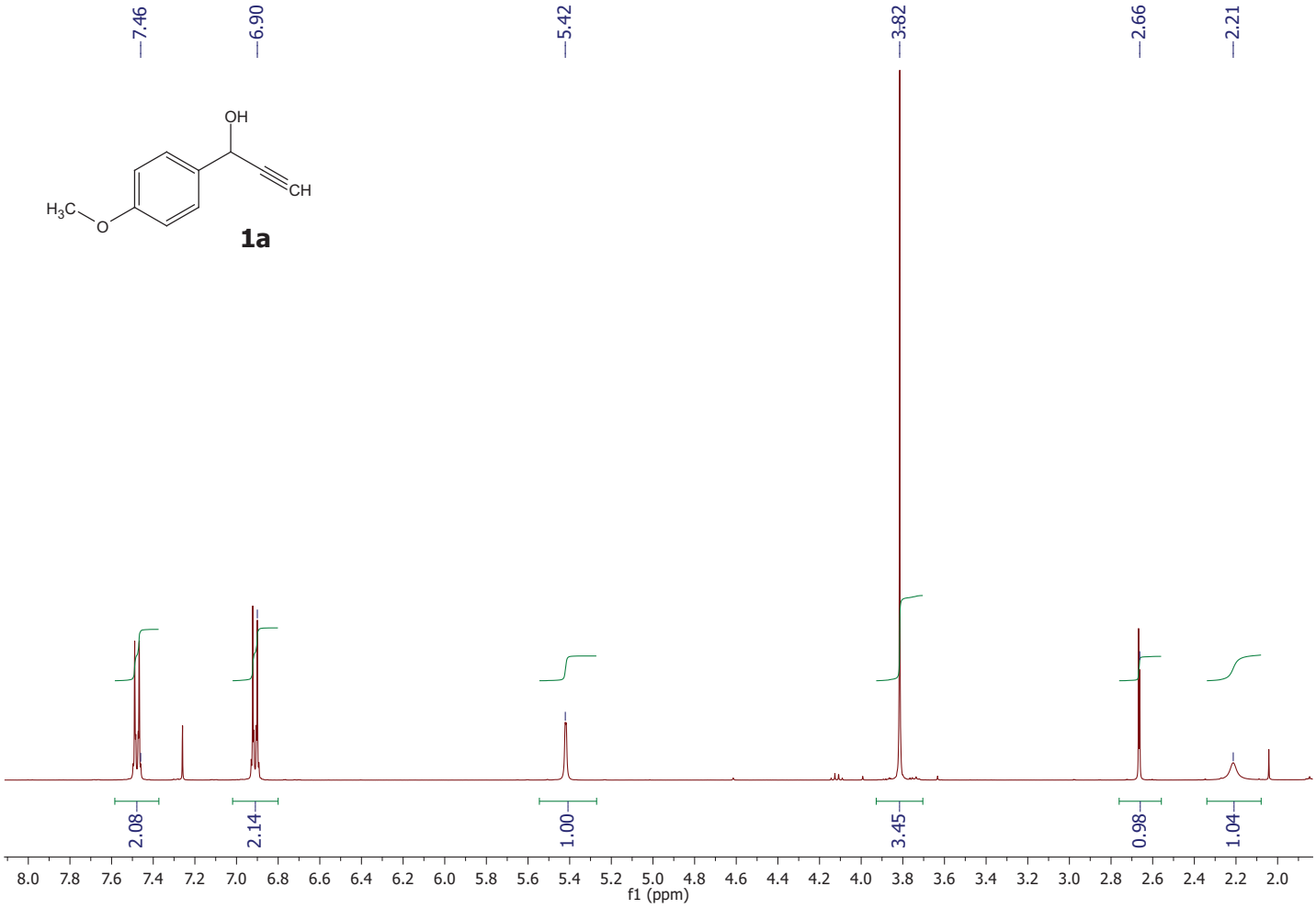
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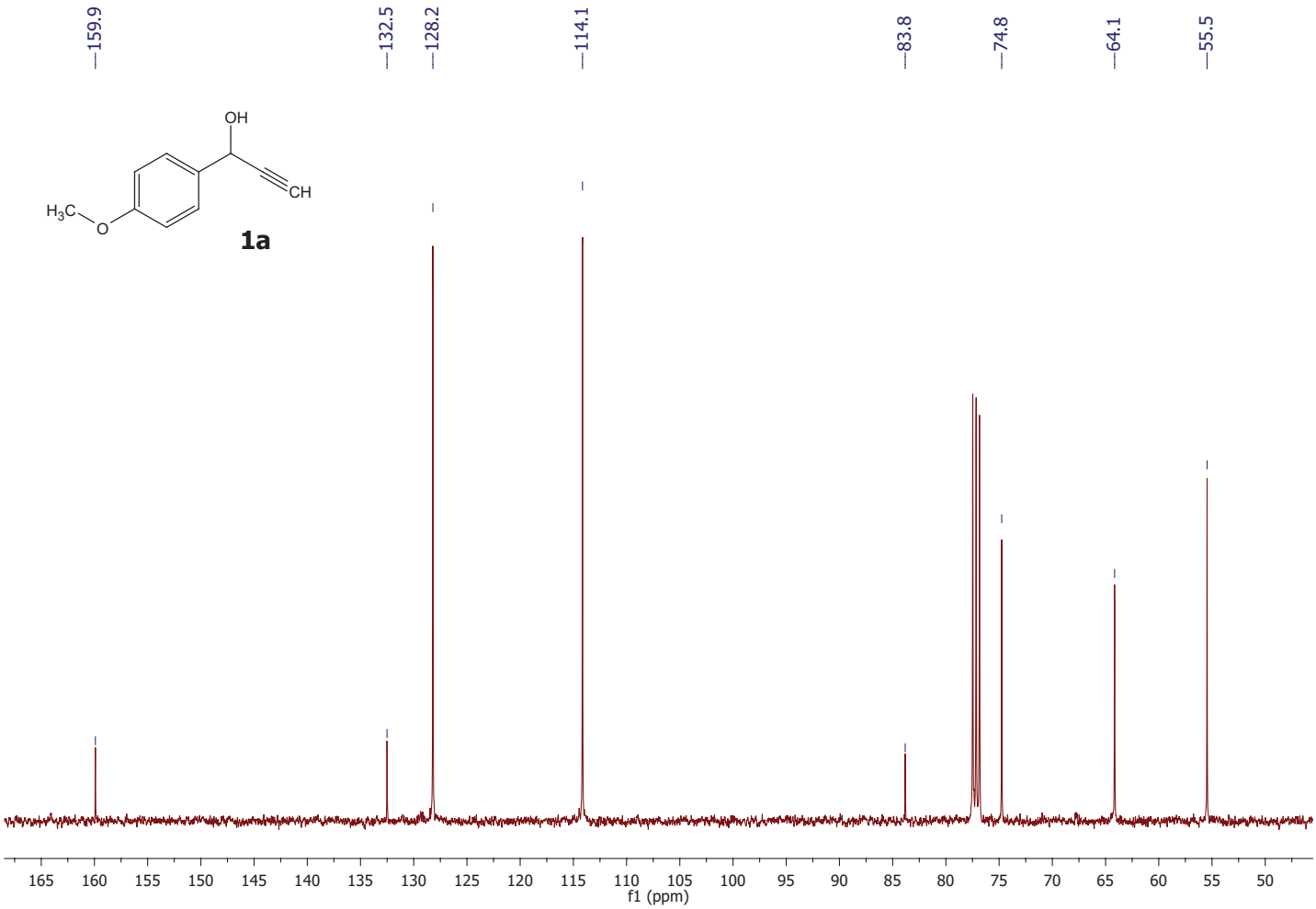
f1 (ppm)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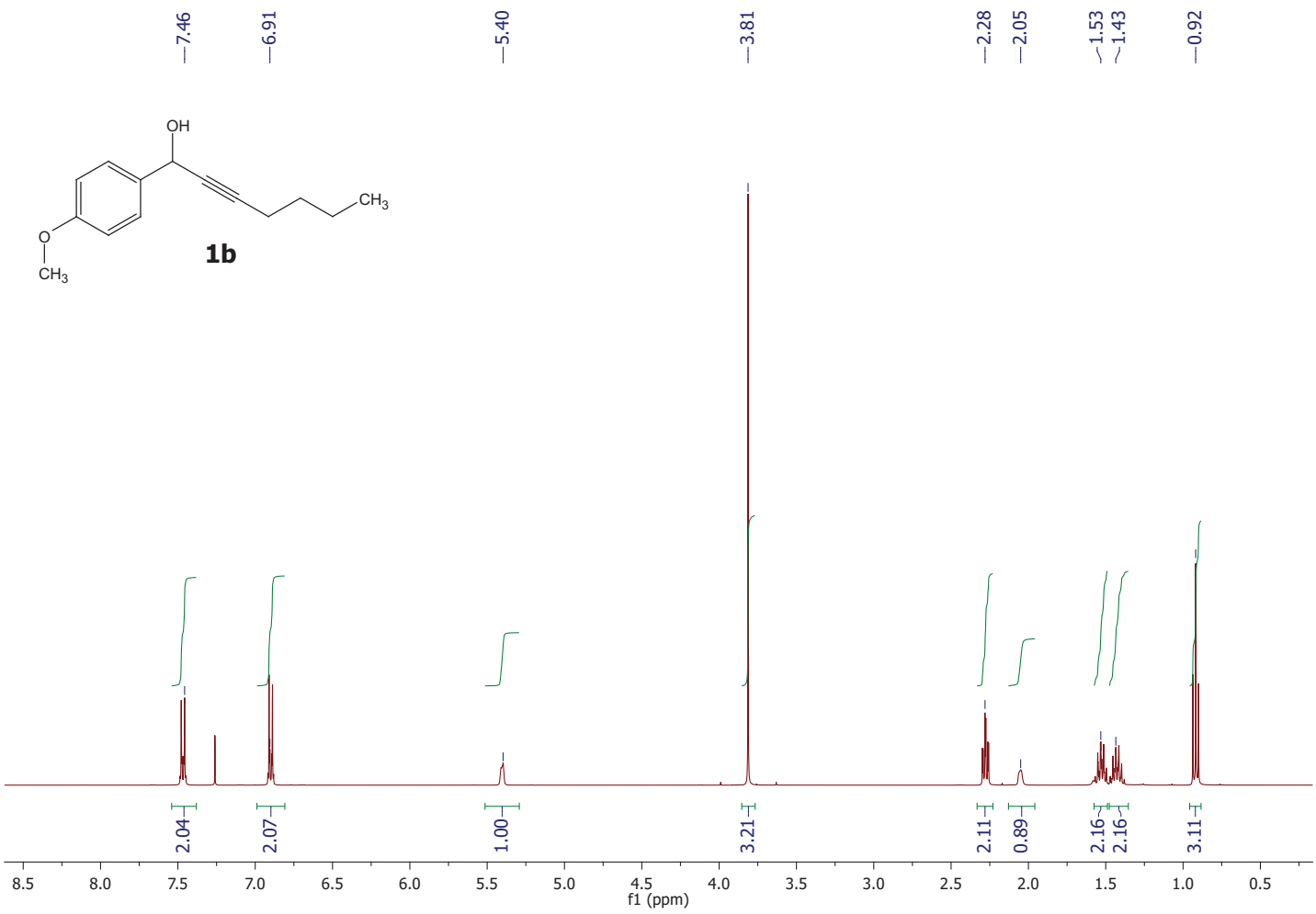
1a
f1 (ppm)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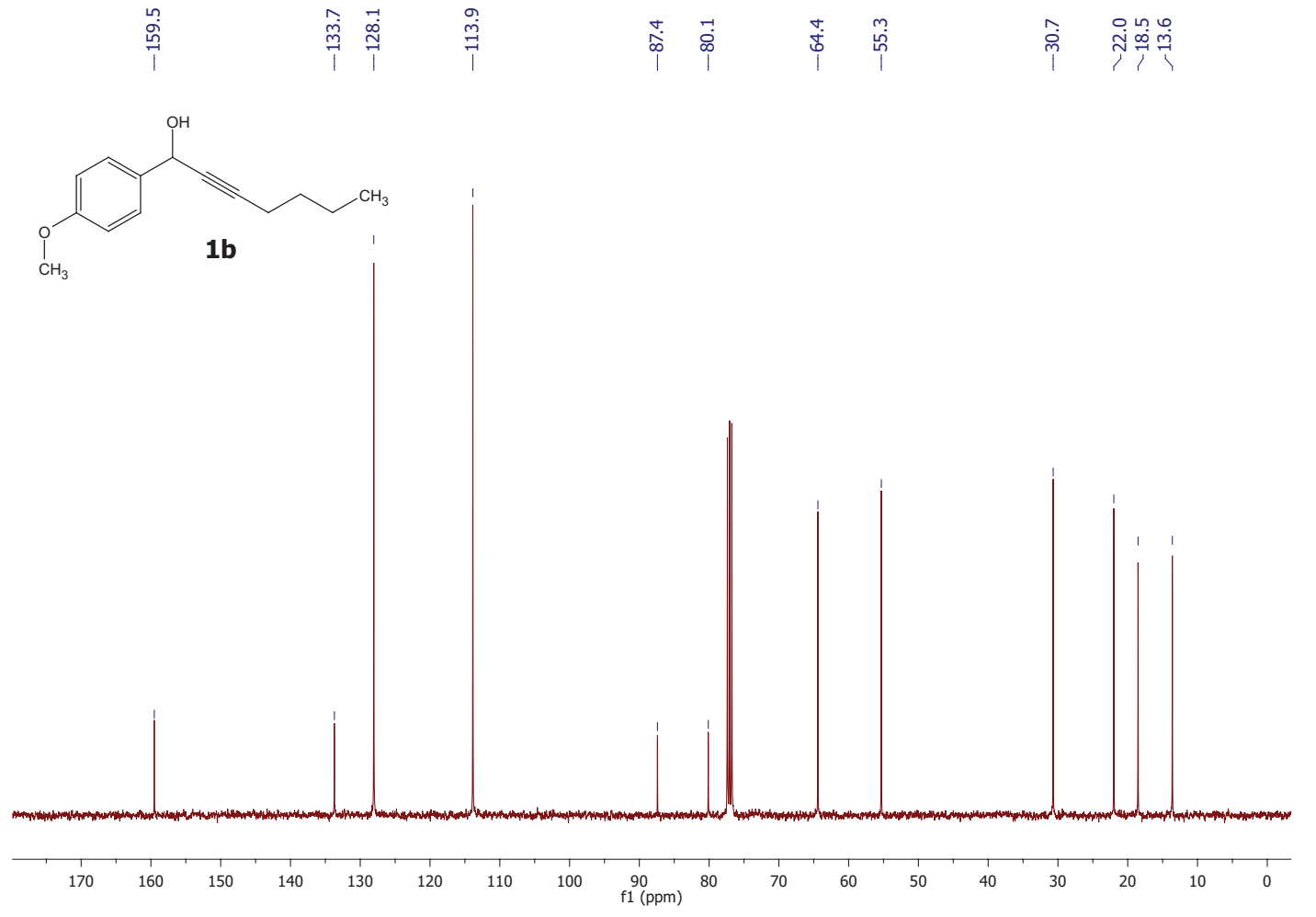
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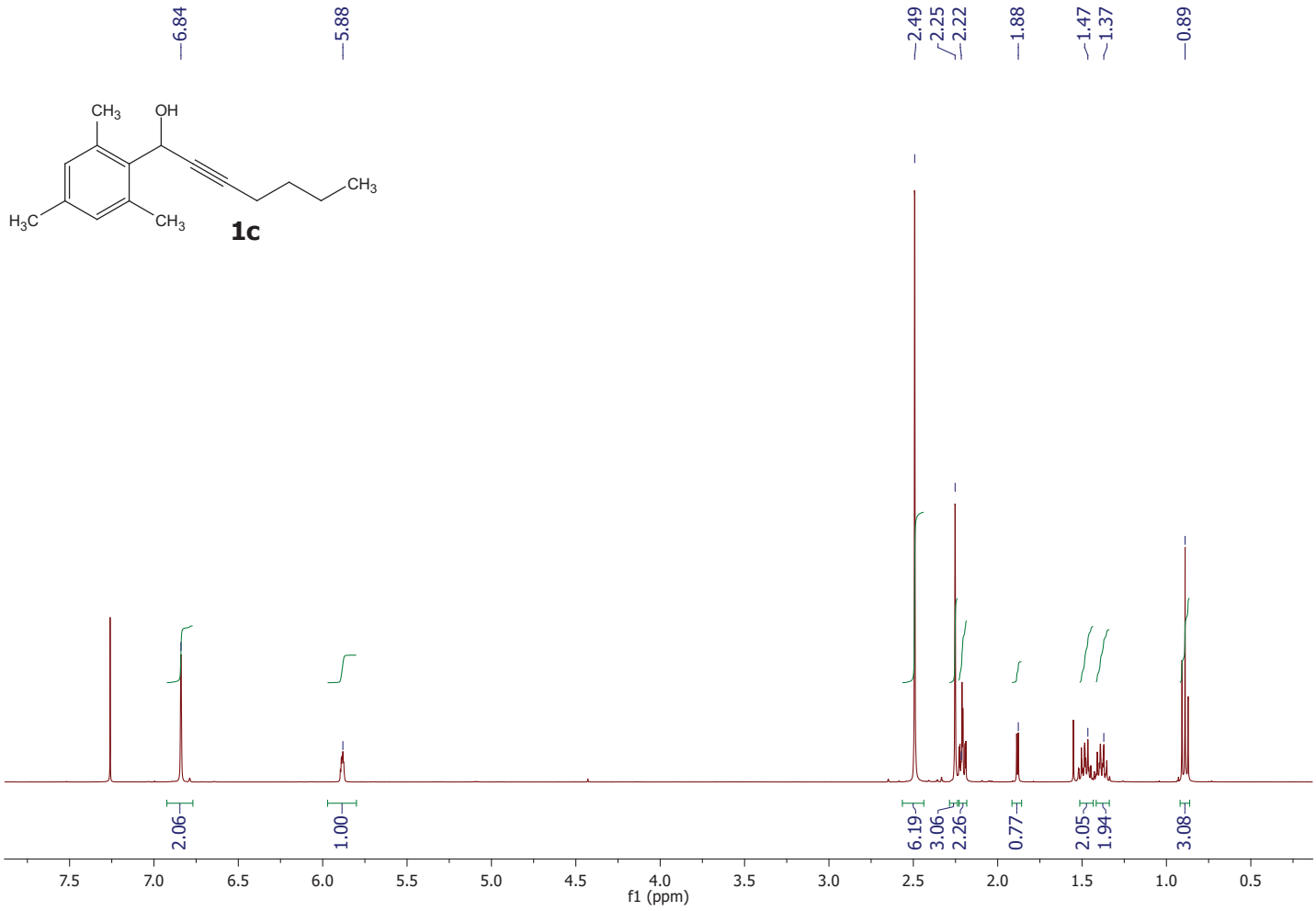
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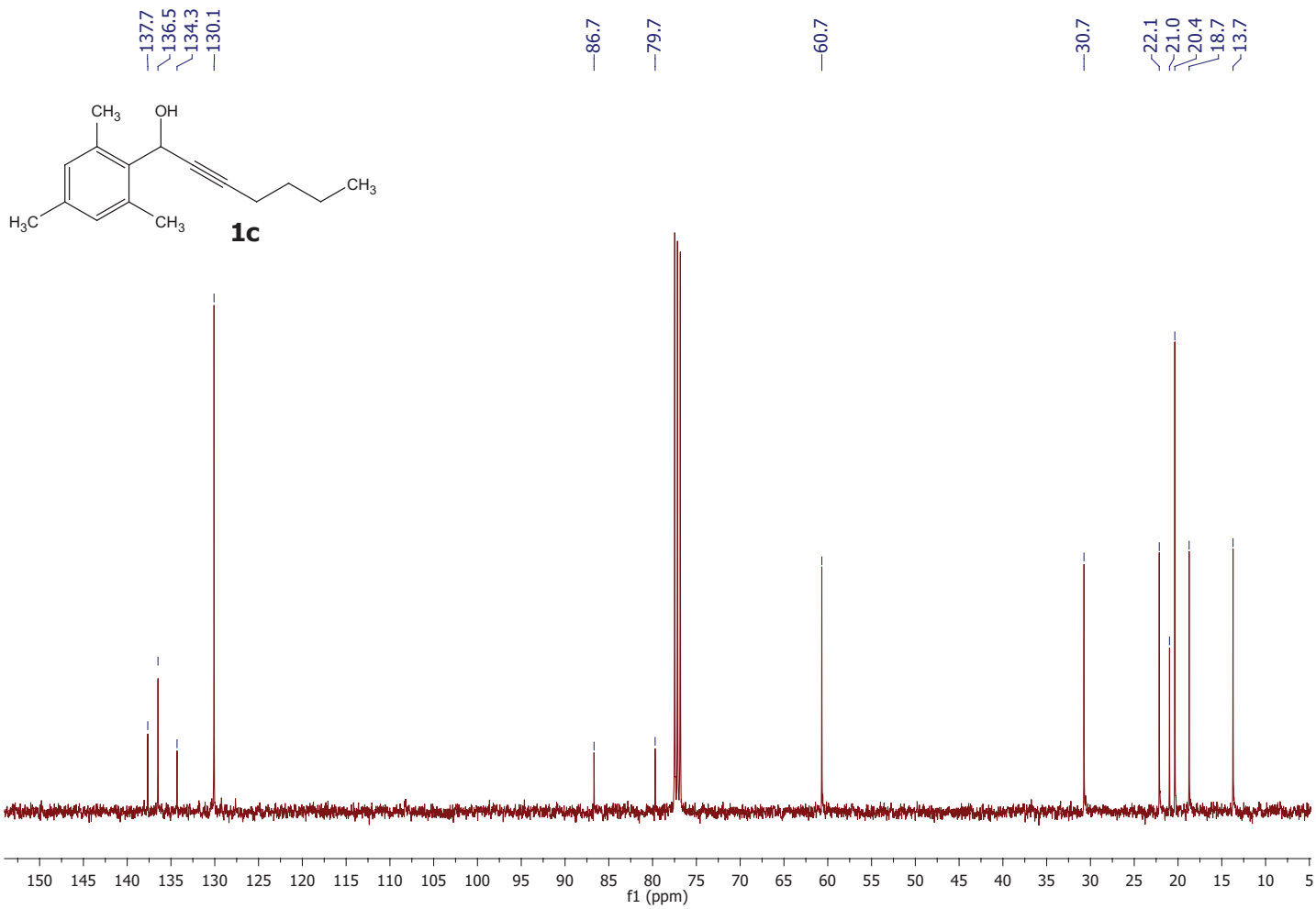
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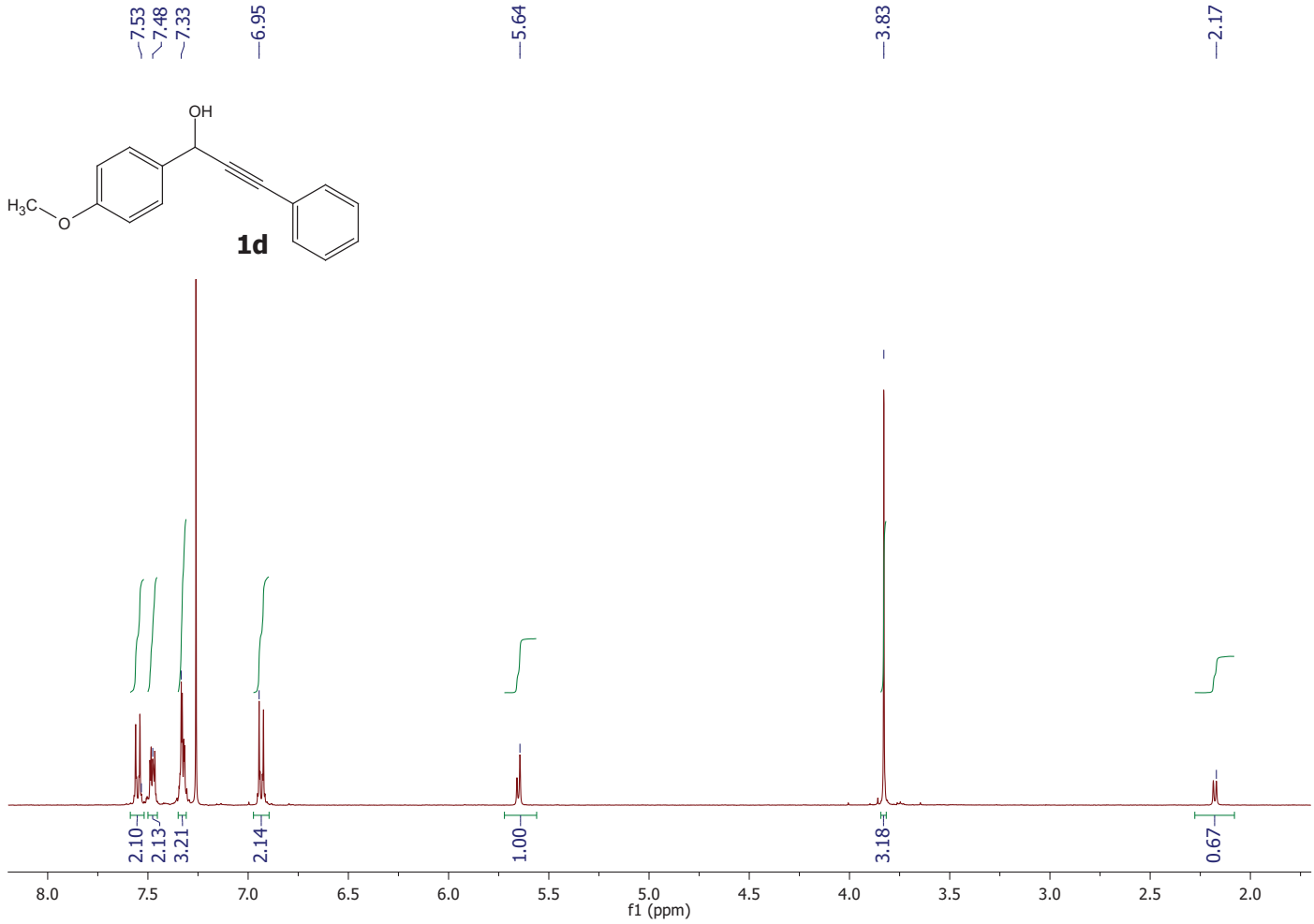
f1 (ppm)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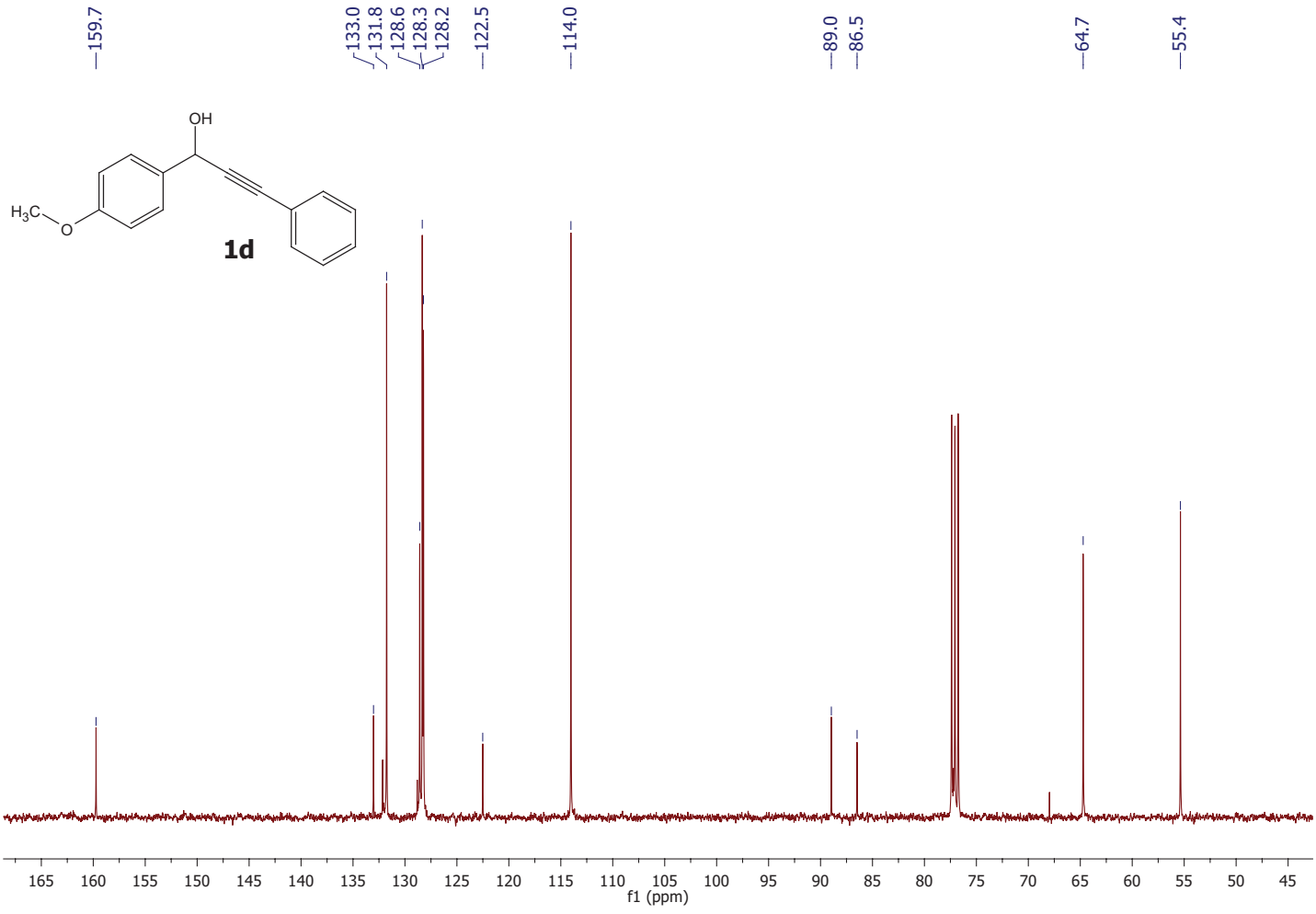
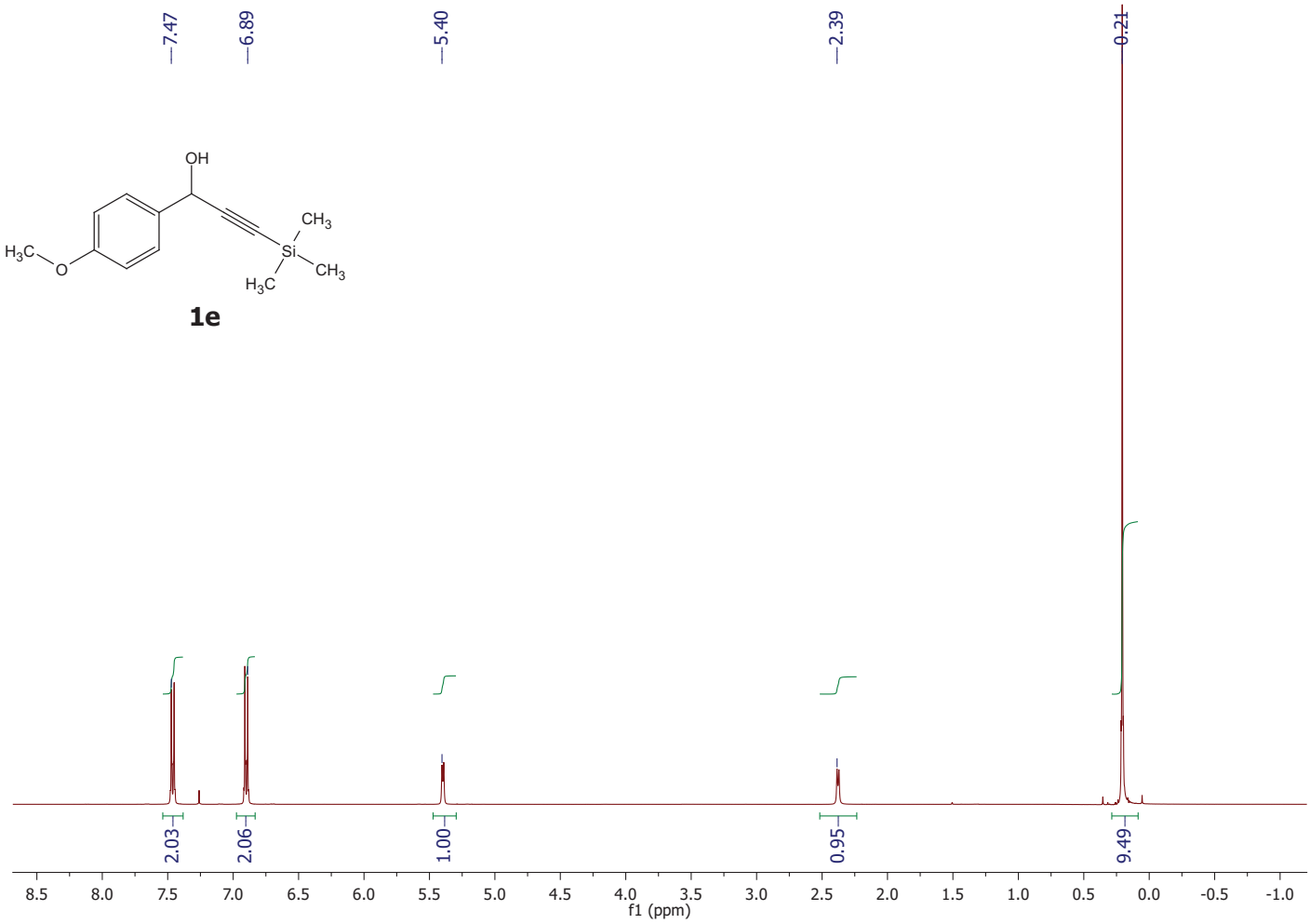
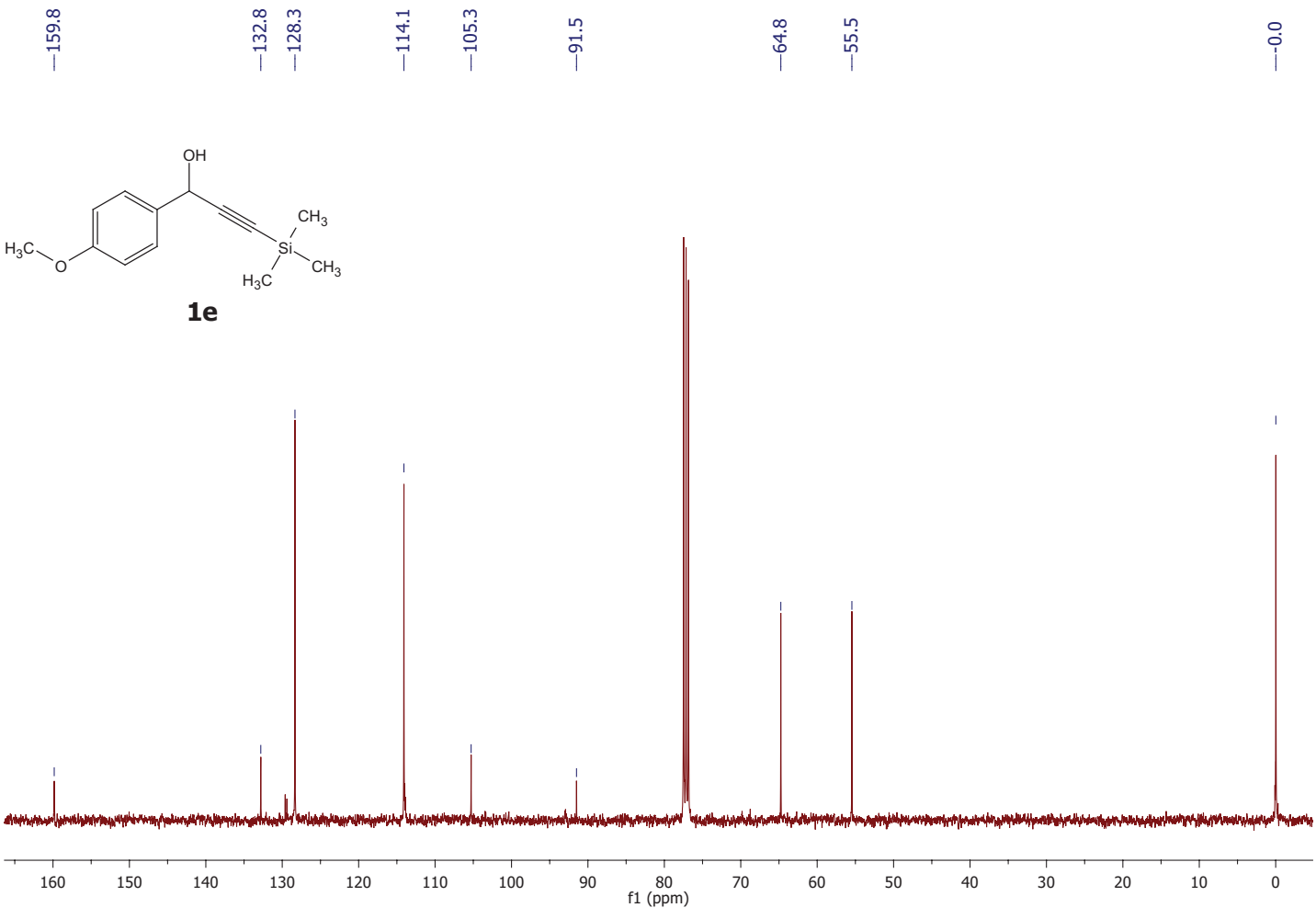
2.5
2.0
f1 (ppm)

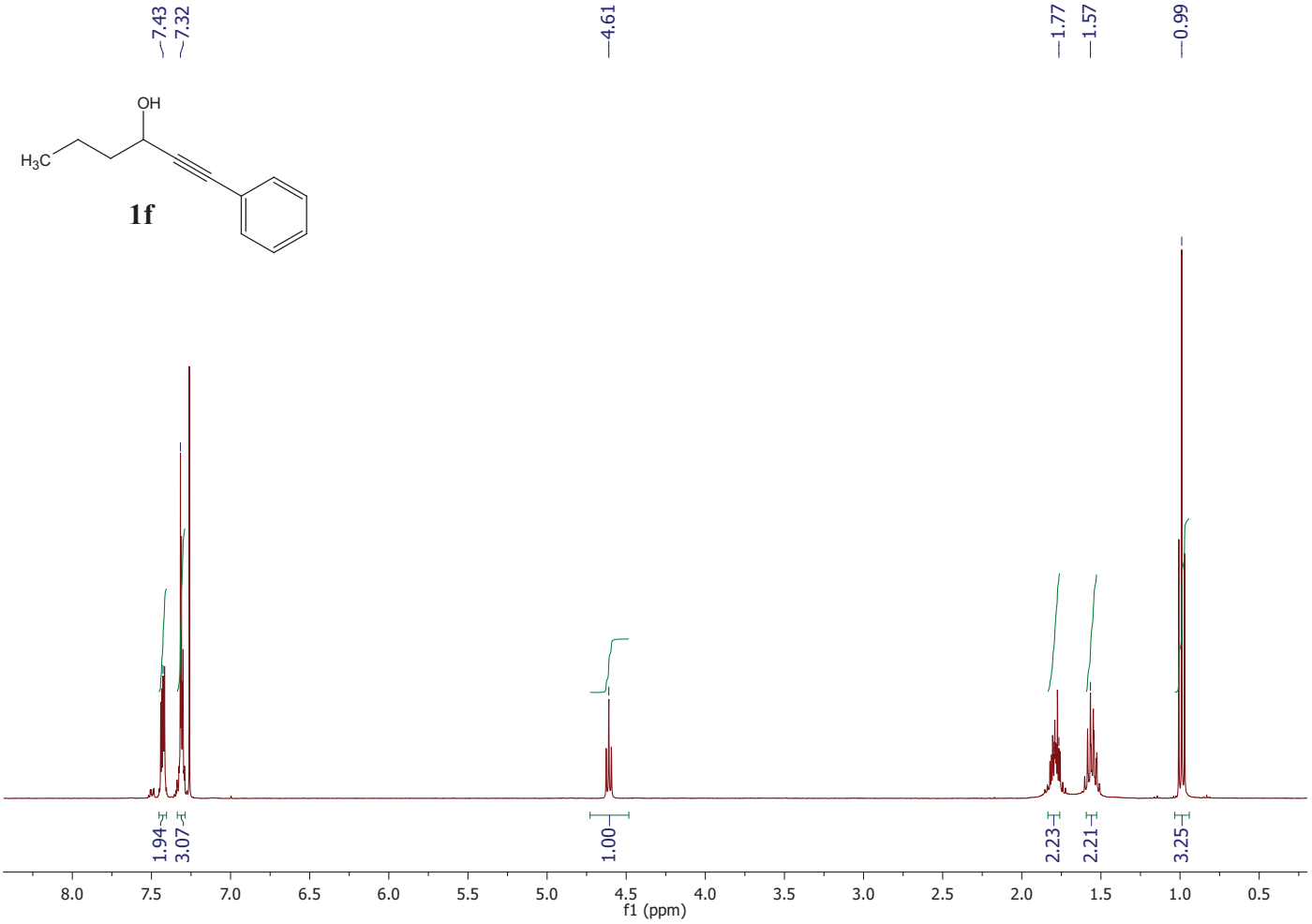

159.7
133.0
131.8
128.6
128.3
128.2
122.5
114.0
89.0
86.5
64.7
55.4
OH
H3C
O
1d
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
f1 (ppm)

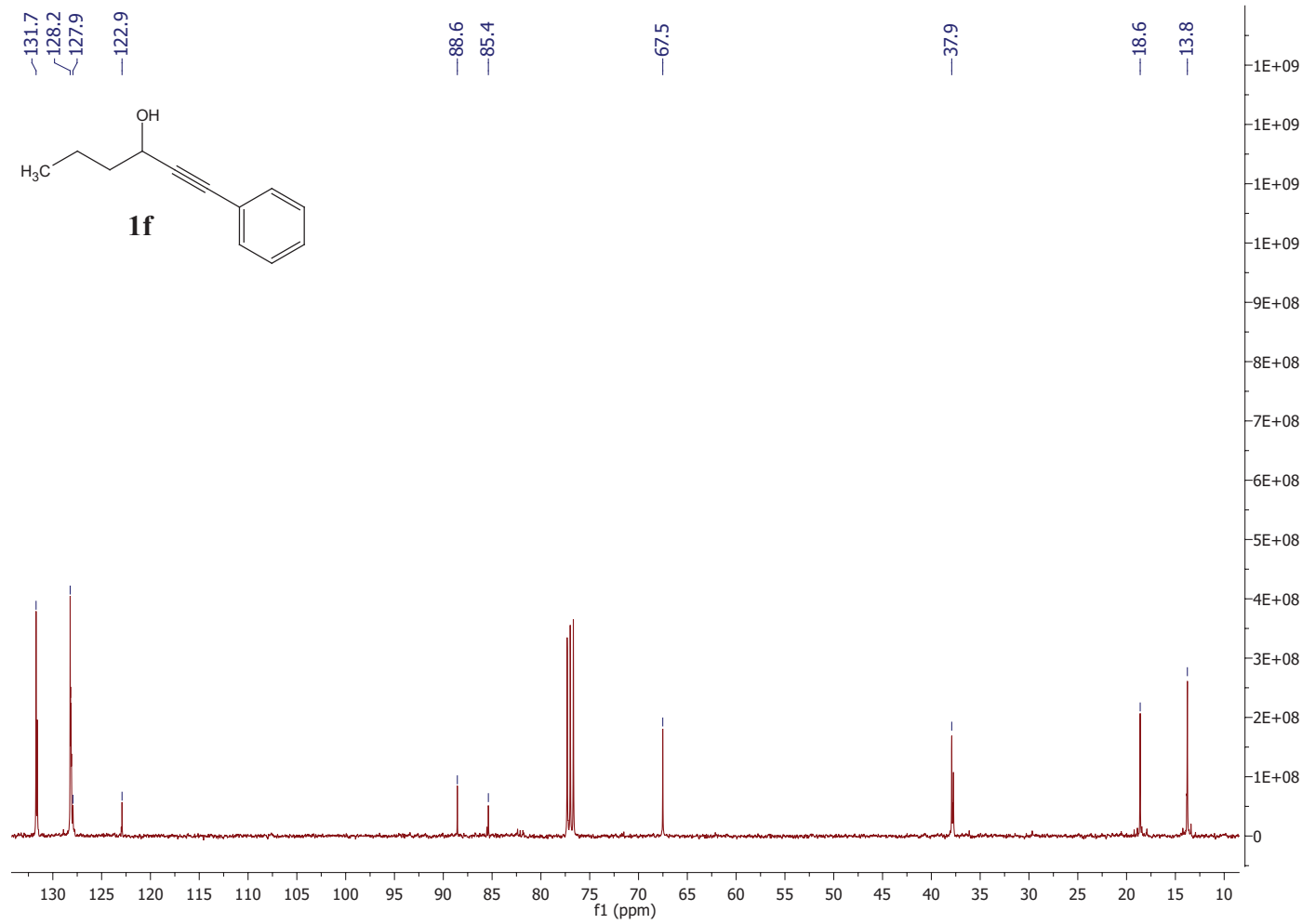

7.47
6.89
5.40
2.39
0.21
OH
CH3
H3C
O
Si
CH3
H3C
2.03
2.06
1.00
0.95
9.49
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

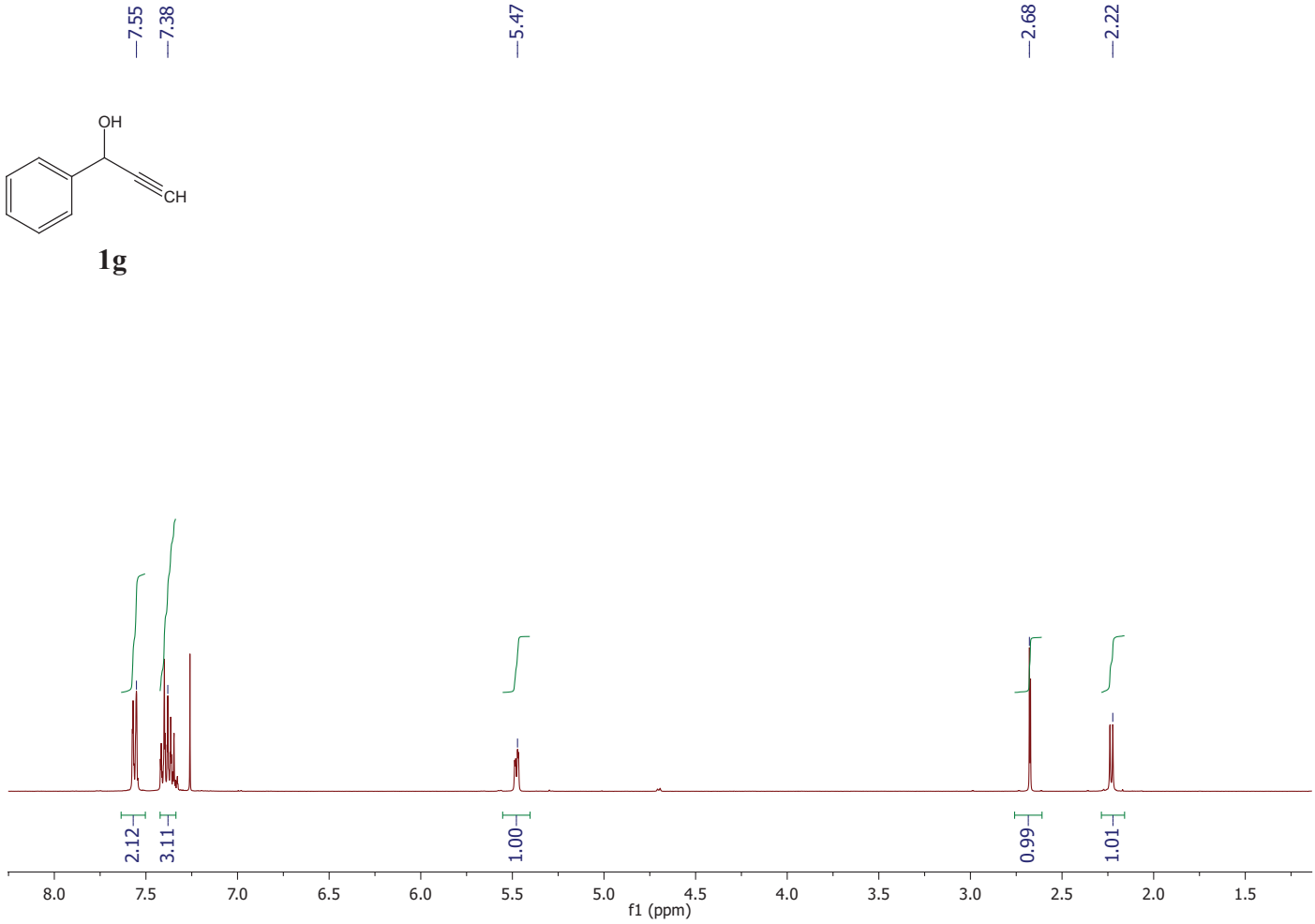
1e


159.8
132.8
128.3
114.1
105.3
91.5
64.8
55.5
0.0
OH
CH3
H3C
O
Si
CH3
H3C
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

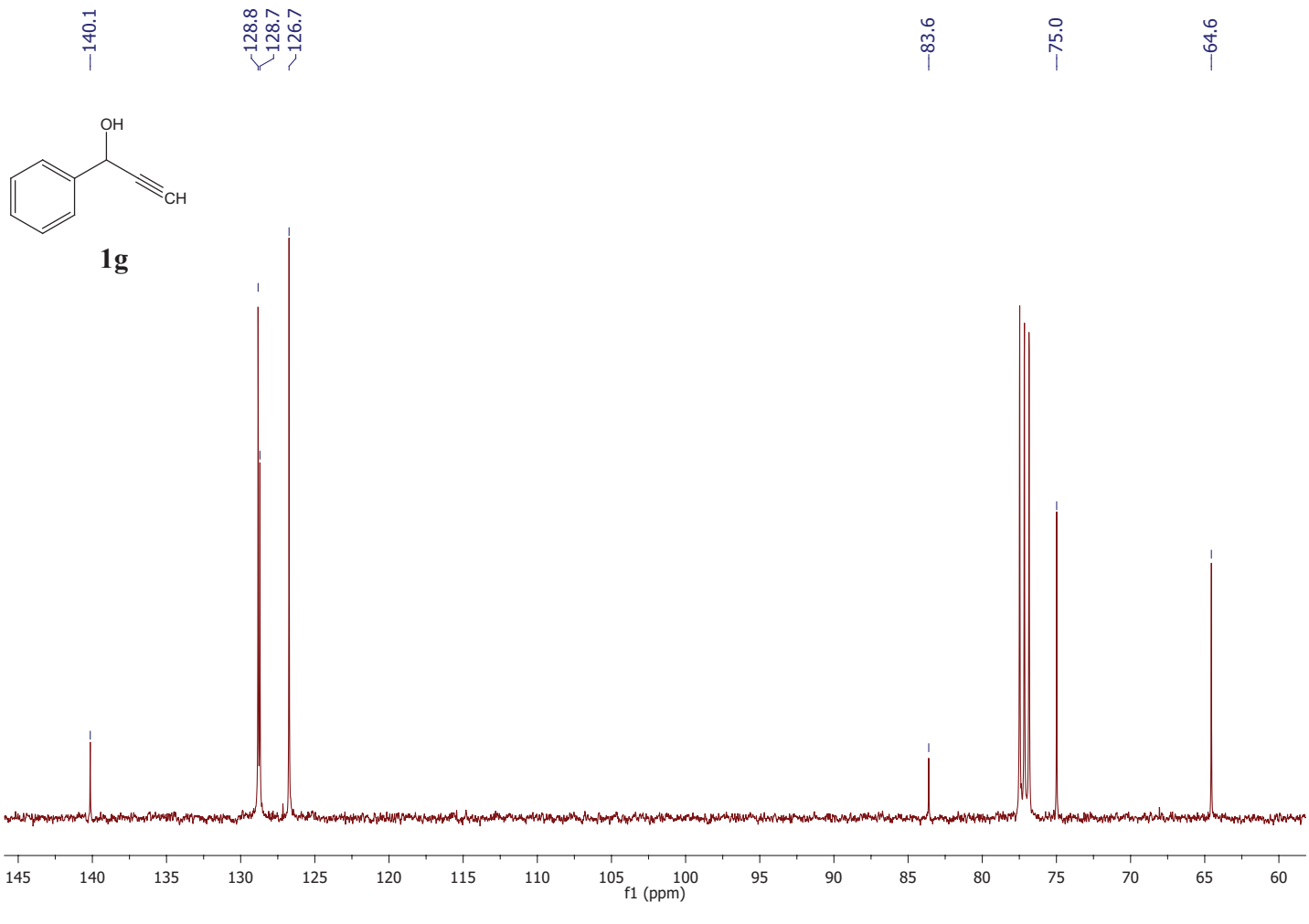
1e


7.43
7.32
4.61
1.77
1.57
0.99
OH
H3C
1f
1.94
3.07
1.00
2.23
2.21
3.25
f1 (ppm)

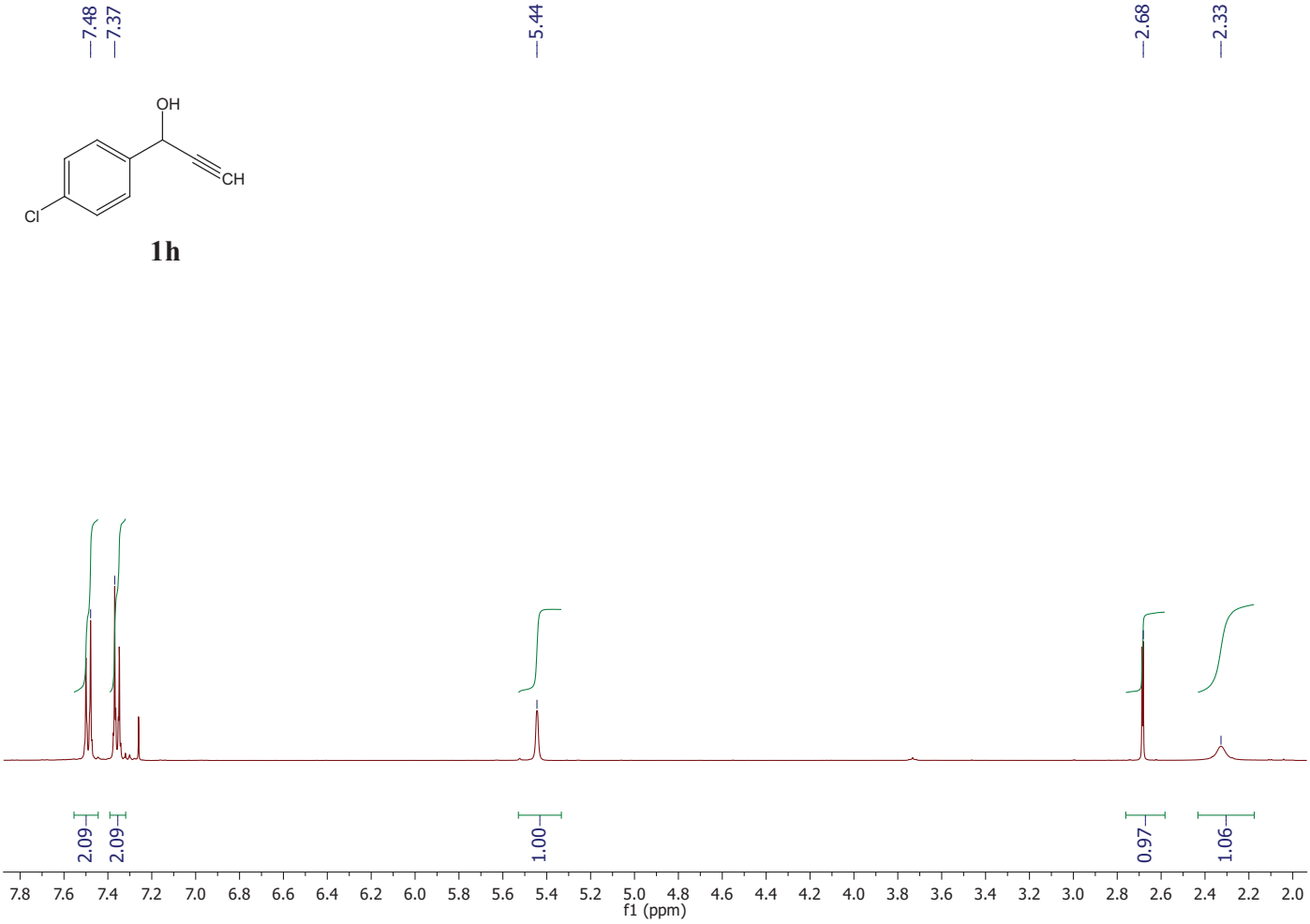

131.7
128.2
127.9
122.9
88.6
85.4
67.5
37.9
18.6
13.8
OH
H3C
1f
f1 (ppm)


7.55
7.38
5.47
2.68
2.22
OH
CH
2.12
3.11
1.00
0.99
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
f1 (ppm)

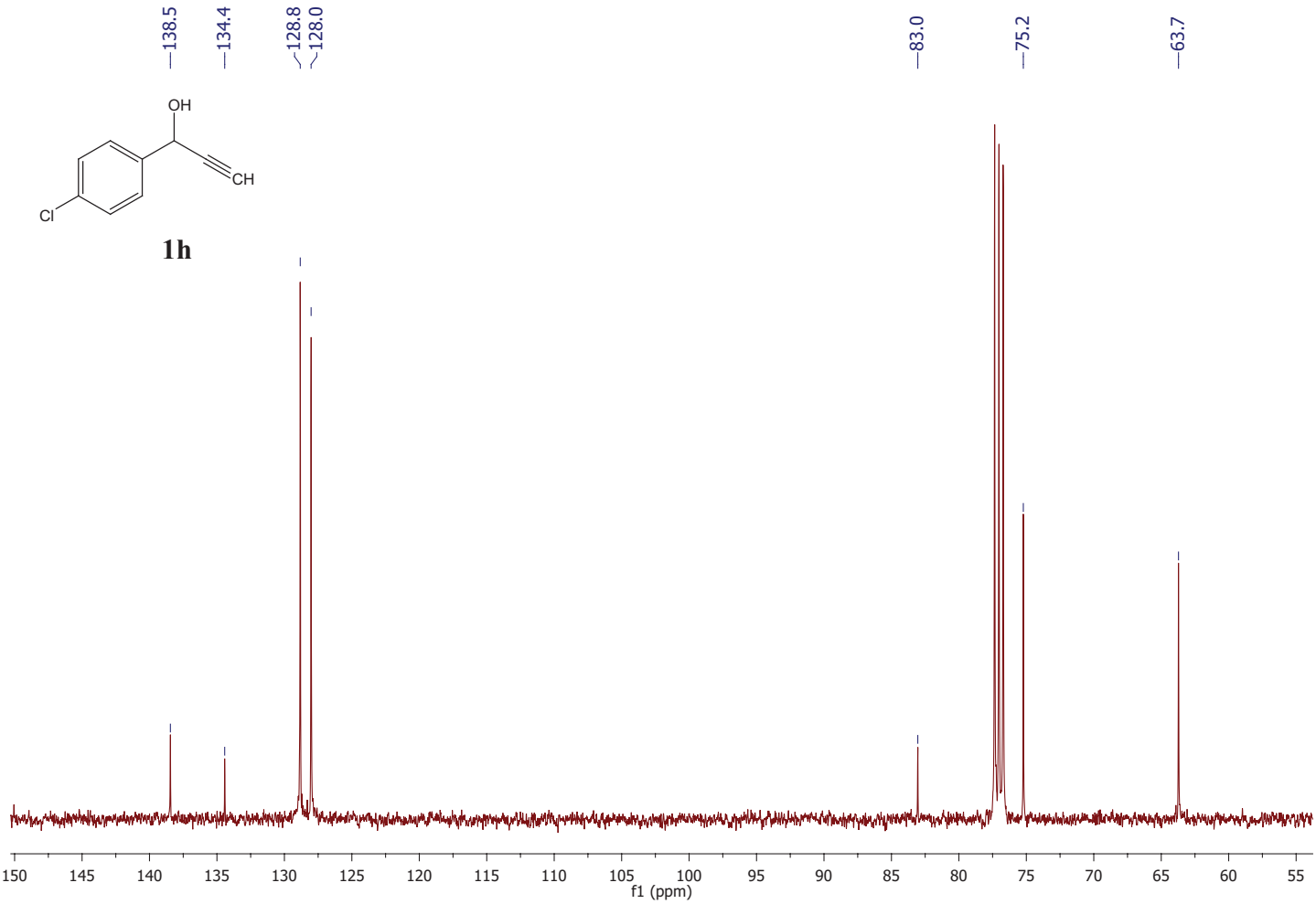
1g


140.1
128.8
128.7
126.7
83.6
75.0
64.6
OH
CH
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
f1 (ppm)

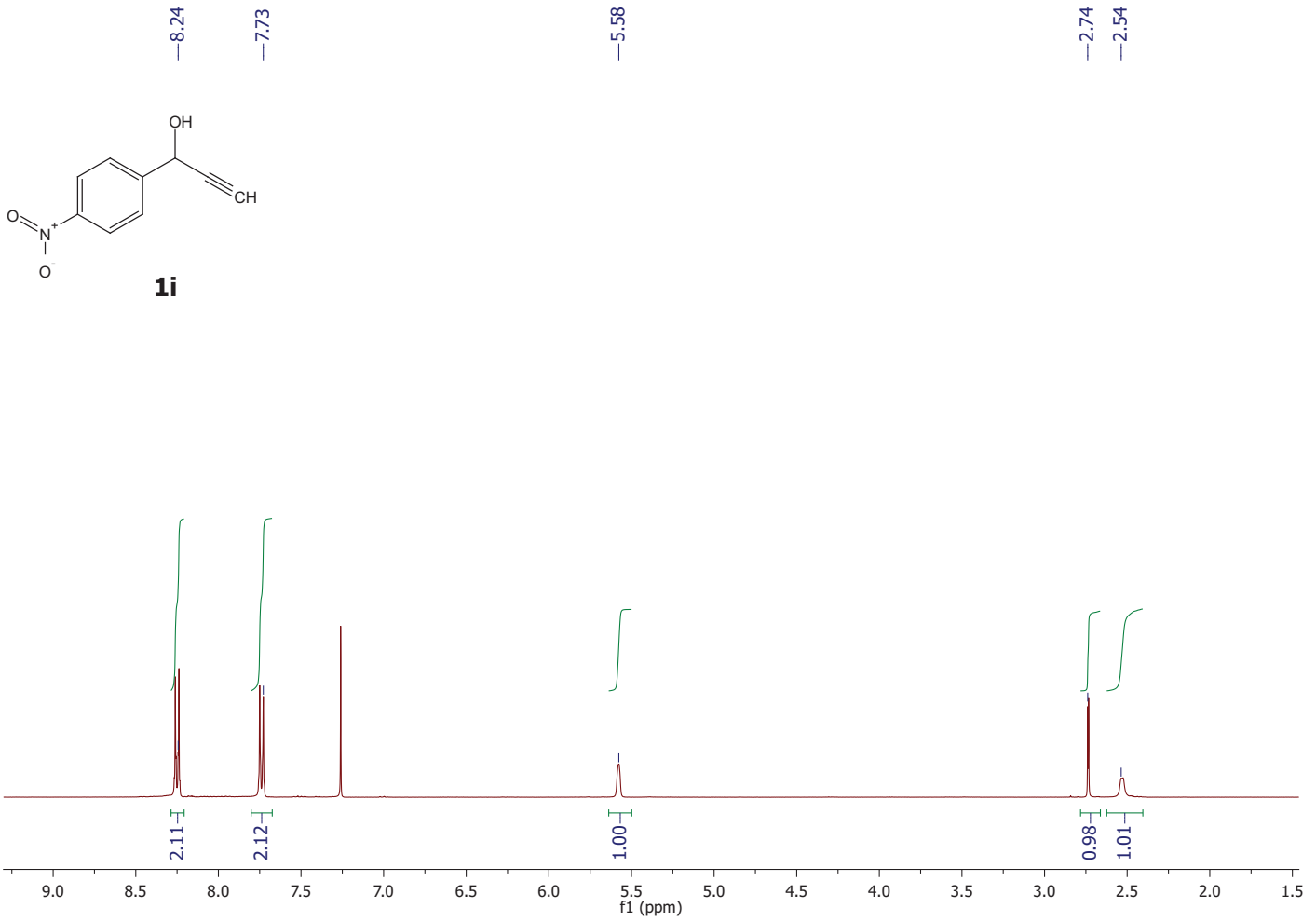
1g


7.48
7.37
5.44
2.68
2.33
OH
CH
Cl
2.09
2.09
1.00
0.97
1.06
f1 (ppm)

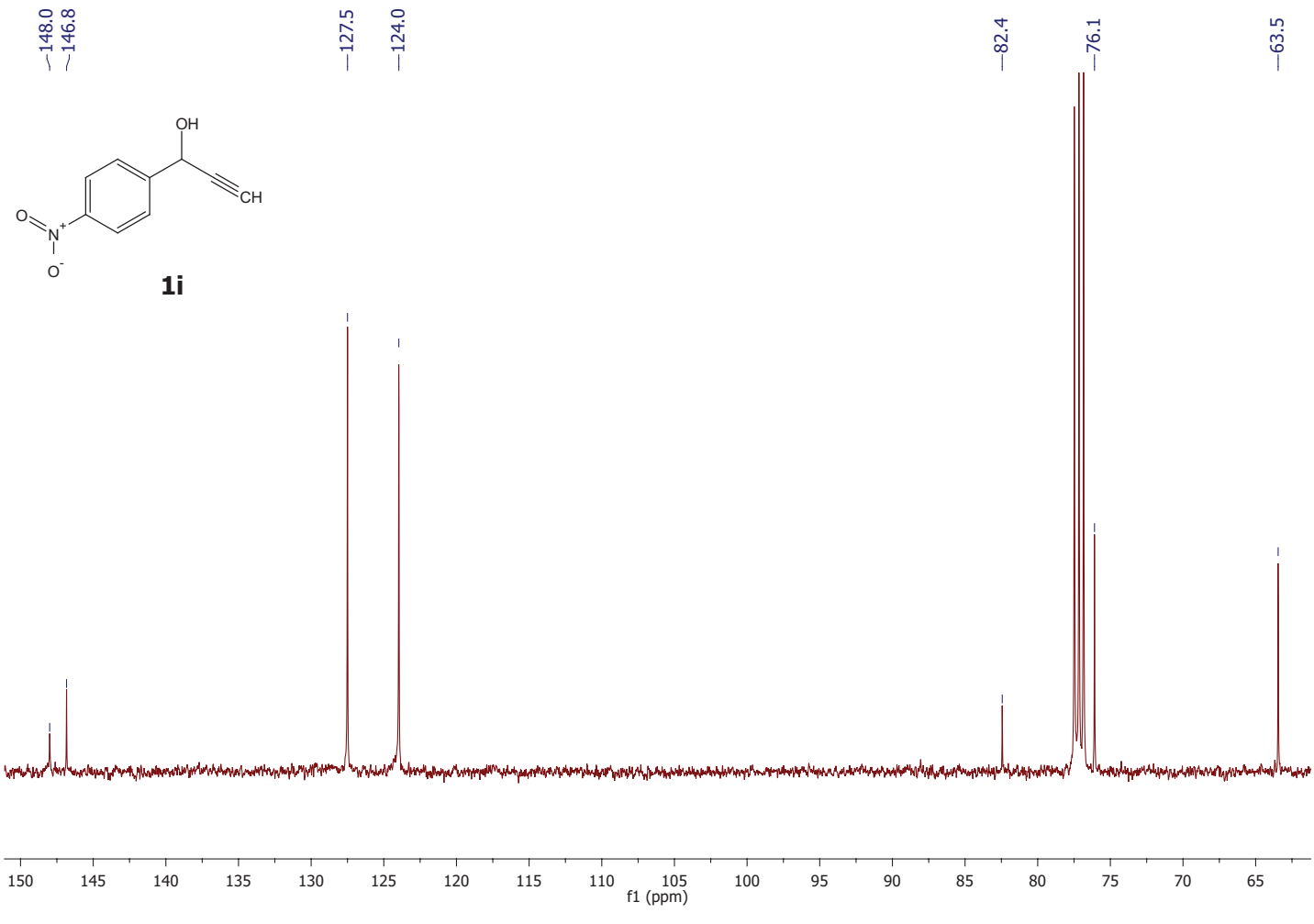
1h


138.5
134.4
128.8
128.0
83.0
75.2
63.7
OH
CH
Cl
f1 (ppm)

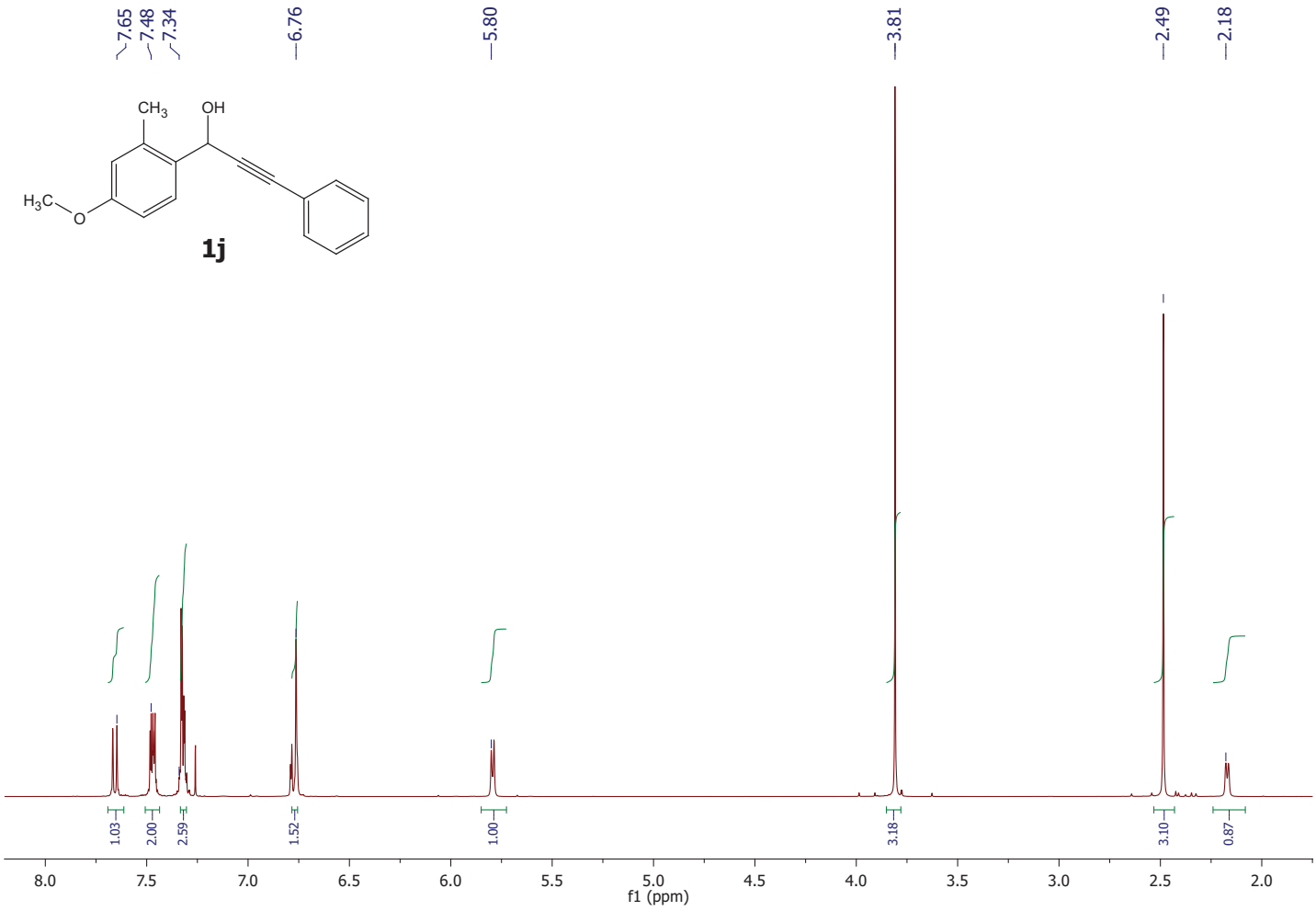
1h

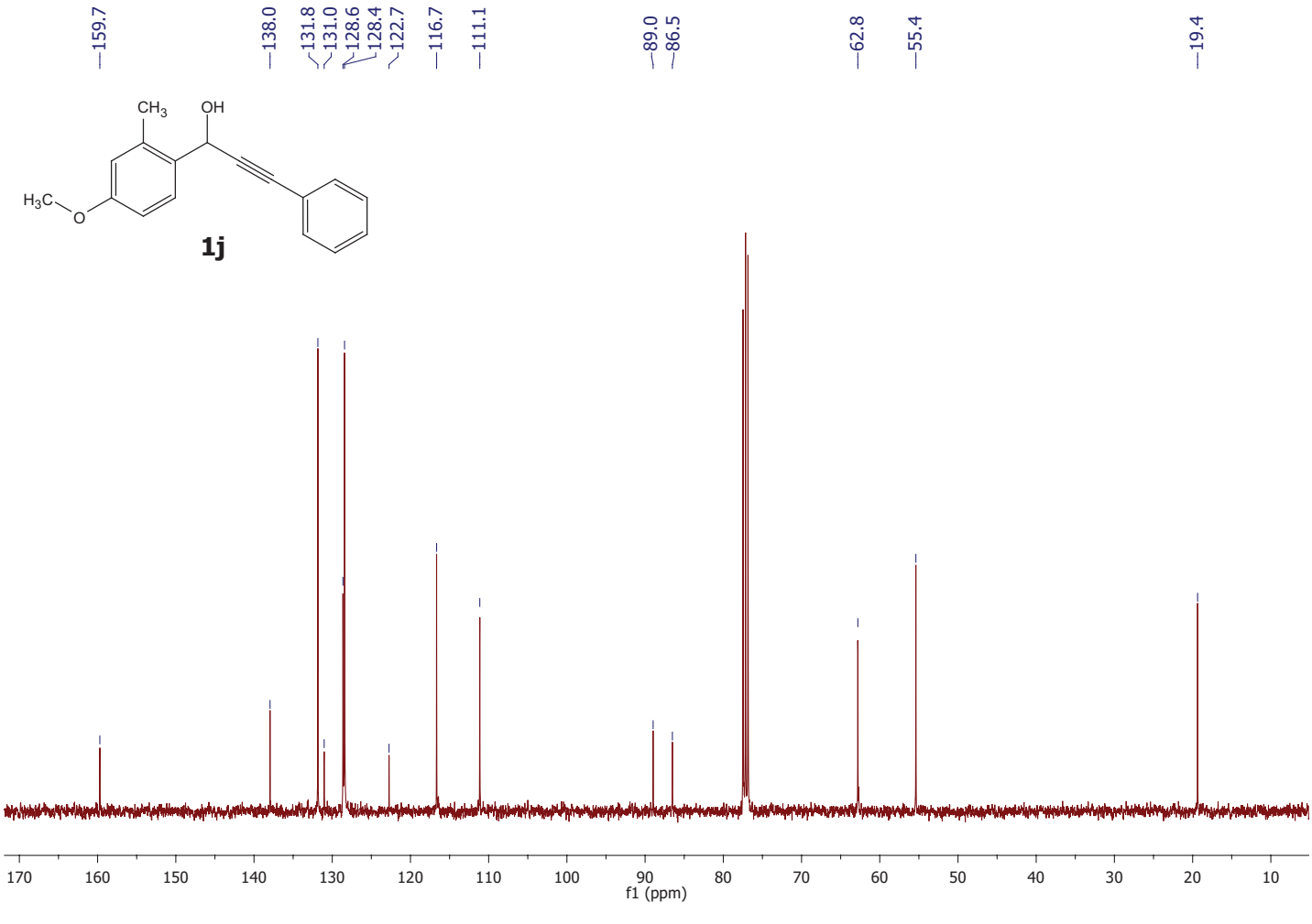

8.24
7.73
5.58
2.74
2.54
OH
CH
O
N+
O-
2.11
2.12
1.00
0.98
1.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f1 (ppm)

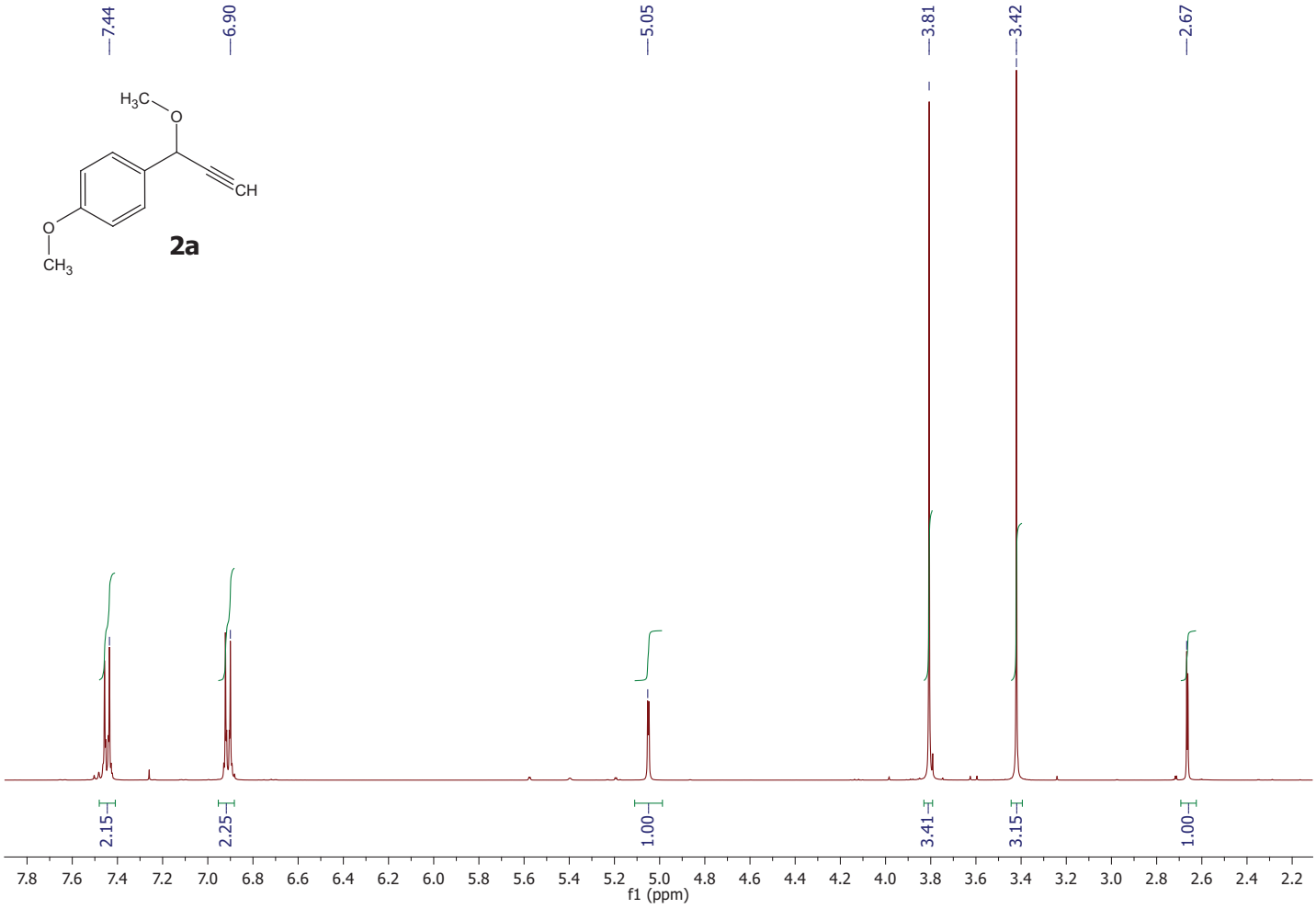
1i

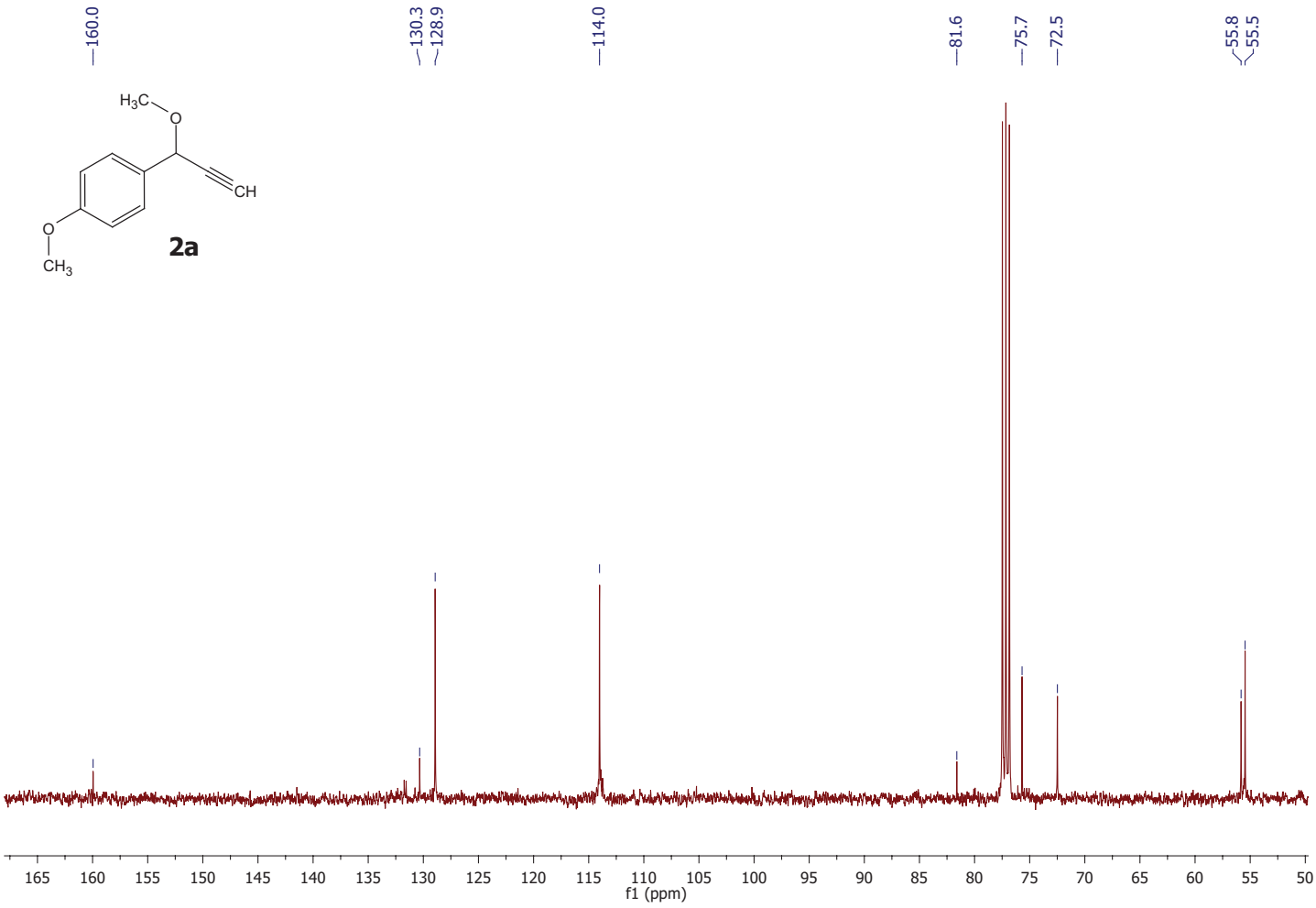

148.0
146.8
127.5
124.0
82.4
76.1
63.5
OH
CH
O
N+
O-
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
f1 (ppm)

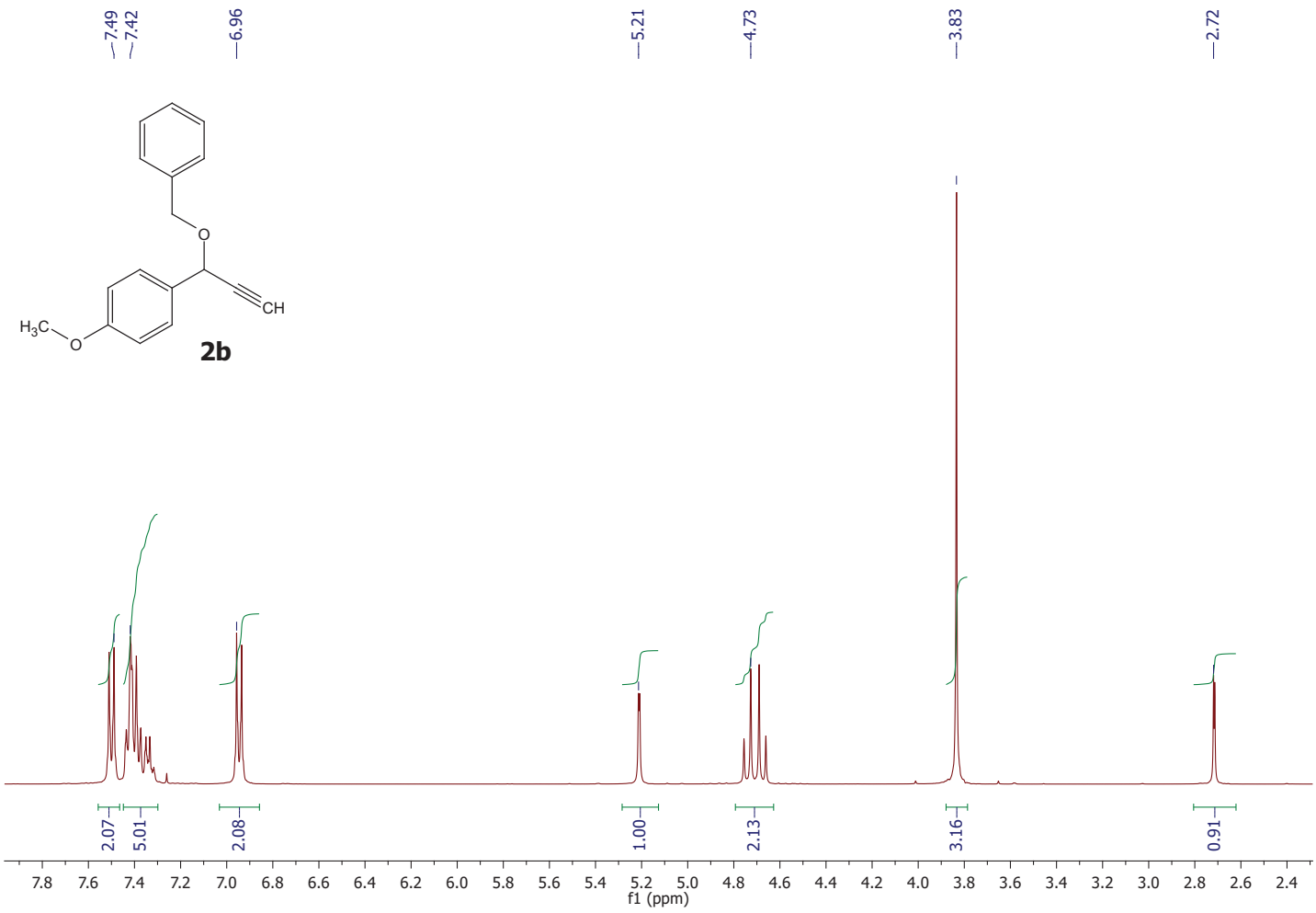
1i

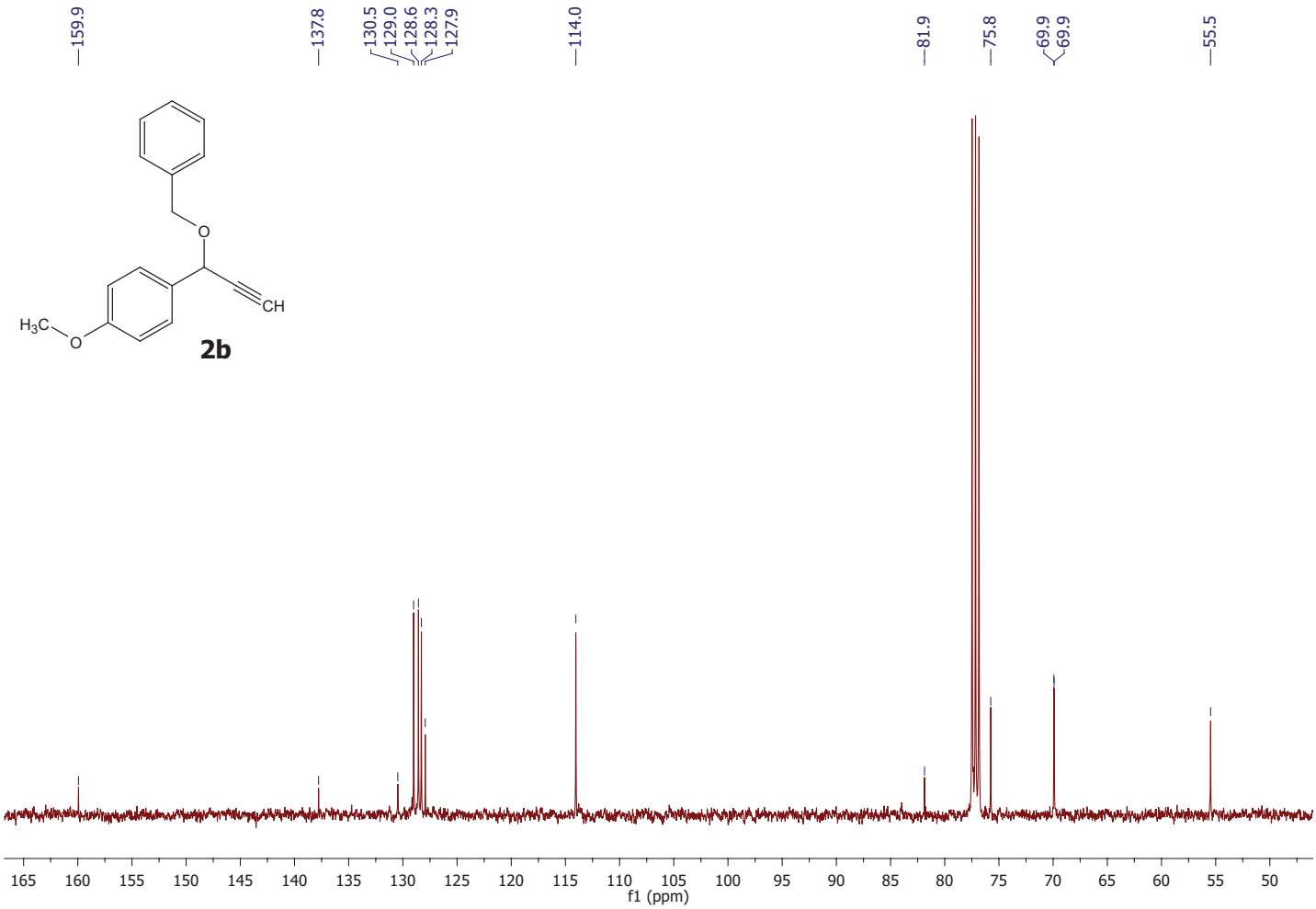

7.65
7.48
7.34
6.76
5.80
3.81
2.49
2.18
CH3
OH
H3C
O
1j
1.03
2.00
2.59
1.52
1.00
3.18
3.10
0.87
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f1 (ppm)

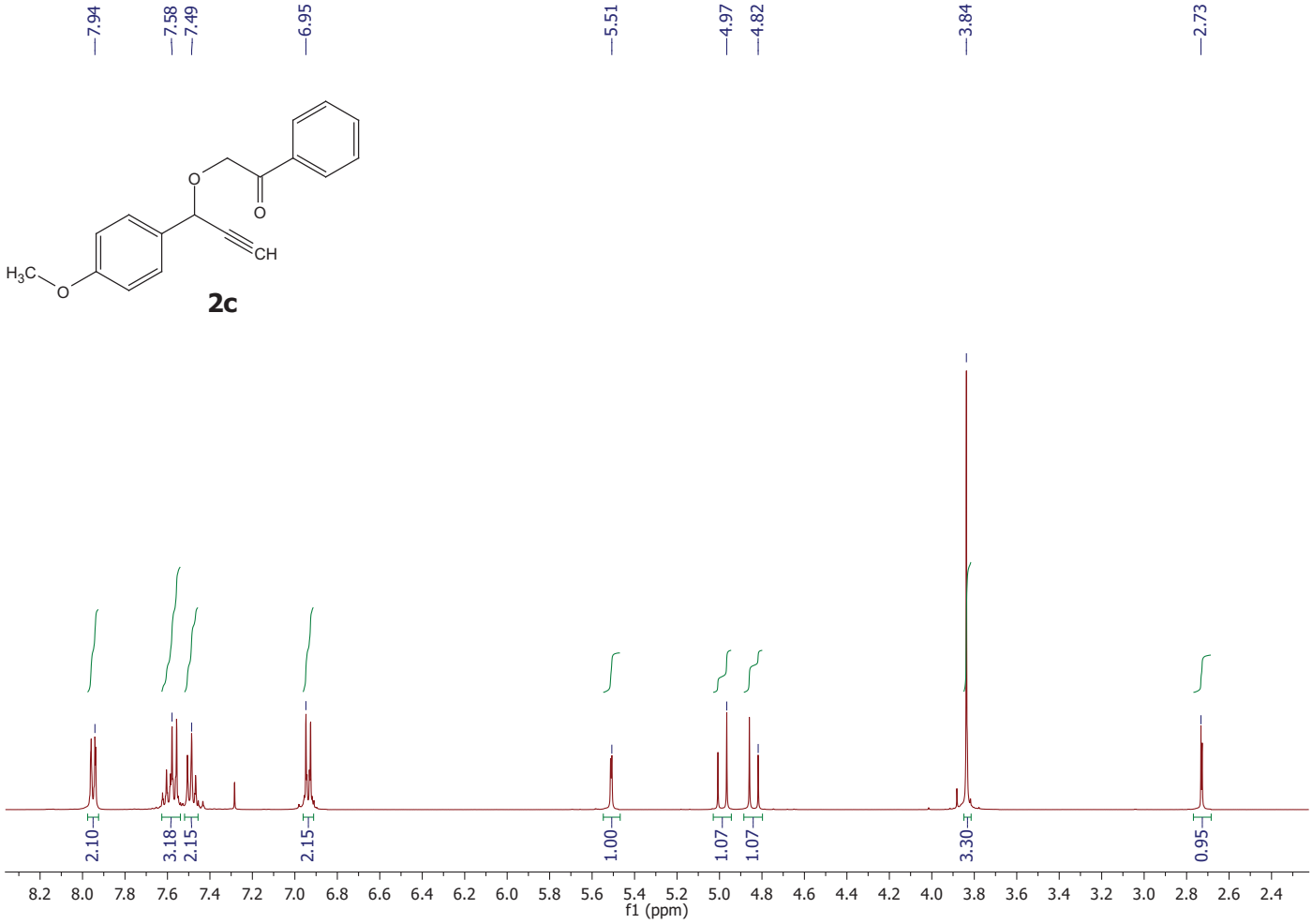

159.7
138.0
131.8
131.0
128.6
128.4
122.7
116.7
111.1
89.0
86.5
62.8
55.4
19.4
CH3
OH
H3C
O
1j
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.44
6.90
5.05
3.81
3.42
2.67
H3C
O
CH
O
CH3
2a
2.15
2.25
1.00
3.41
3.15
1.00
7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2
f1 (ppm)

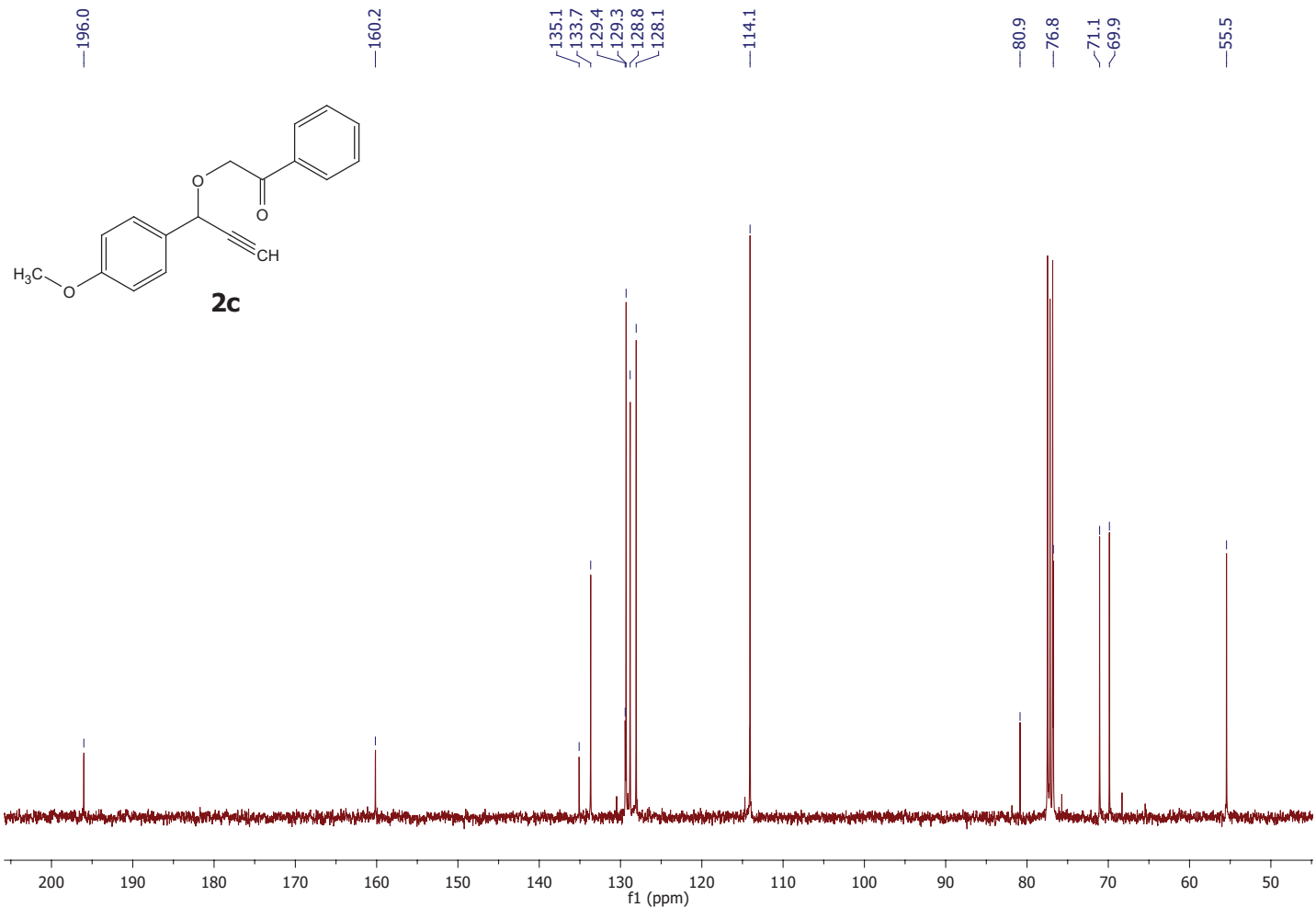

160.0
130.3
128.9
114.0
81.6
75.7
72.5
55.8
55.5
H3C
O
CH
O
CH3
2a
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50
f1 (ppm)


7.49
7.42
6.96
5.21
4.73
3.83
2.72
O
CH
H3C
O
2b
2.07
5.01
2.08
1.00
2.13
3.16
0.91
f1 (ppm)

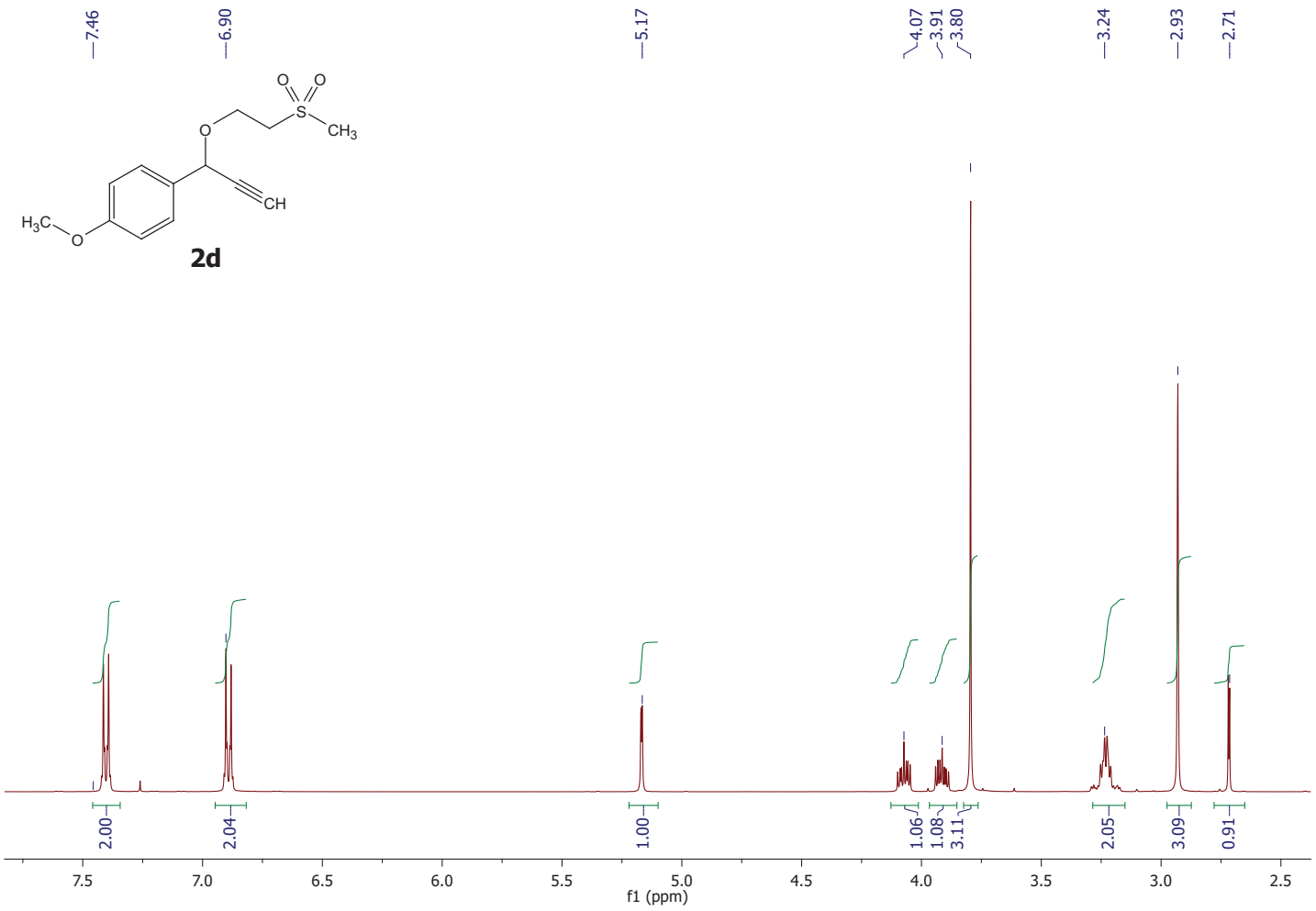

159.9
137.8
130.5
129.0
128.6
128.3
127.9
114.0
81.9
75.8
69.9
69.9
55.5
O
CH
H3C
O
2b
f1 (ppm)


7.94
7.58
7.49
6.95
5.51
4.97
4.82
3.84
2.73
O
O
CH
H3C
O
2.10
3.18
2.15
2.15
1.00
1.07
1.07
3.30
0.95
8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4
f1 (ppm)

2c

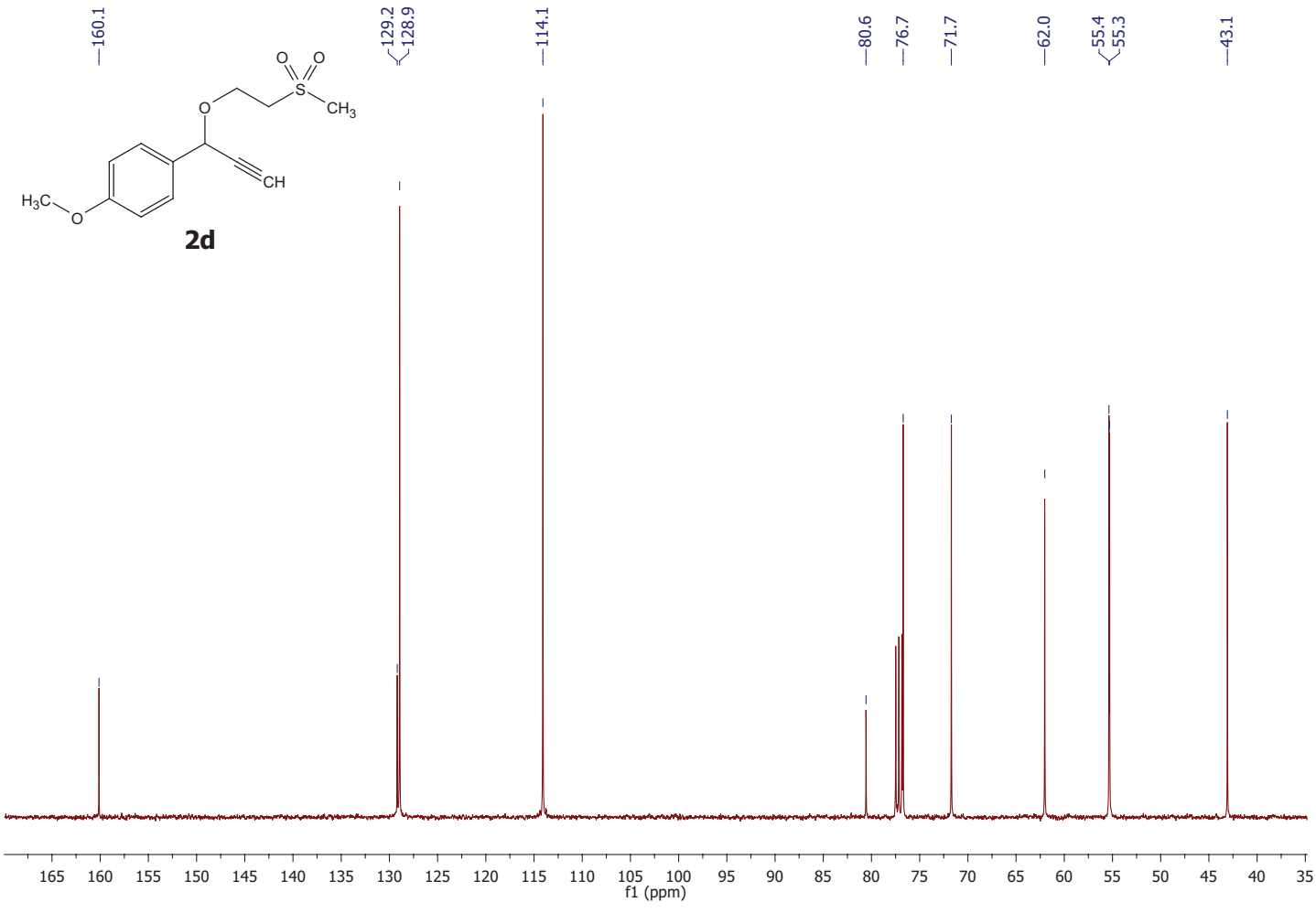

196.0
160.2
135.1
133.7
129.4
129.3
128.8
128.1
114.1
80.9
76.8
71.1
69.9
55.5
O
O
CH
H3C
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50
f1 (ppm)

2c


7.46
6.90
5.17
4.07
3.91
3.80
3.24
2.93
2.71
2.00
2.04
1.00
1.06
1.08
3.11
2.05
3.09
0.91
f1 (ppm)

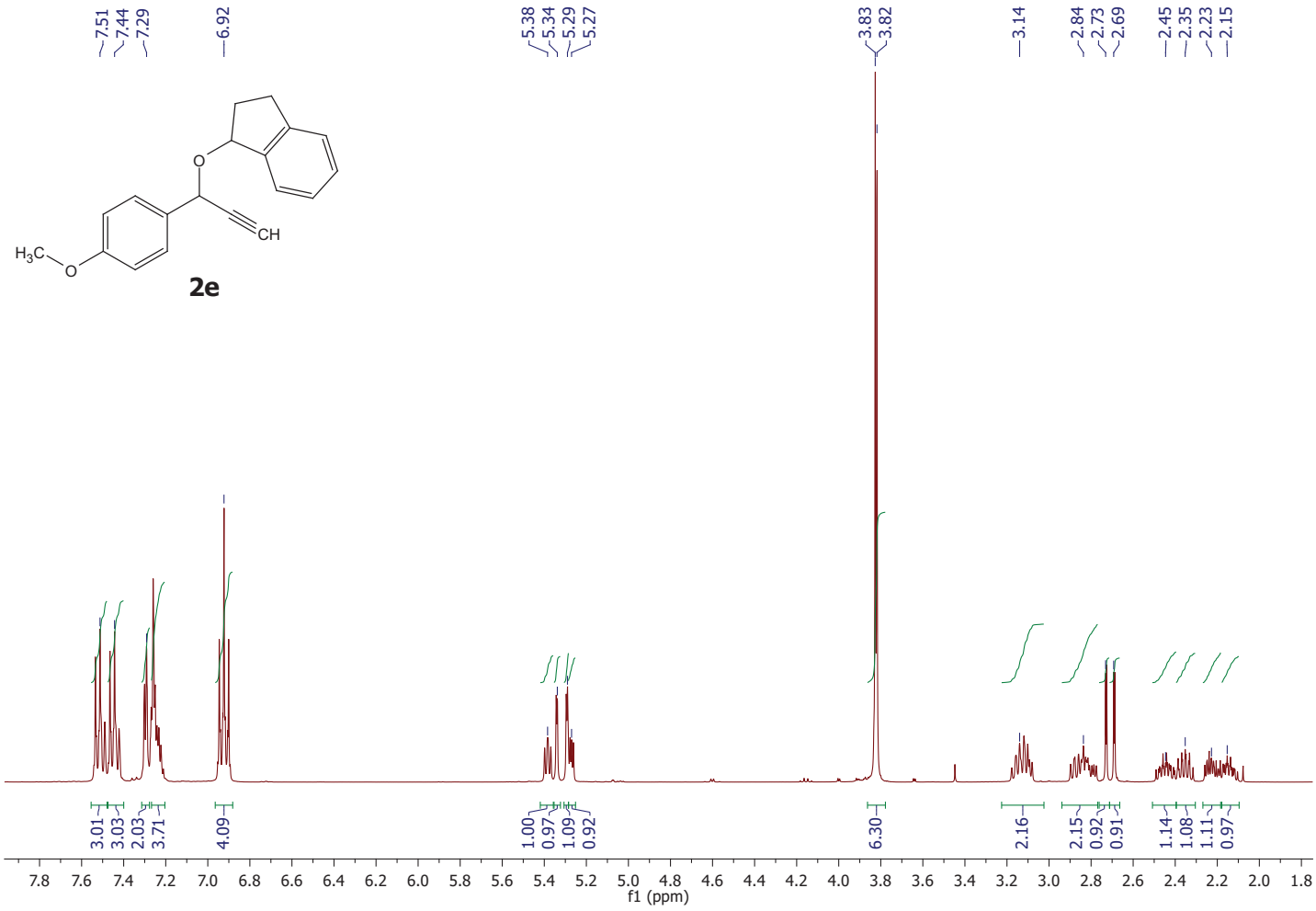
O
O
S
CH3
O
CH
H3C
O

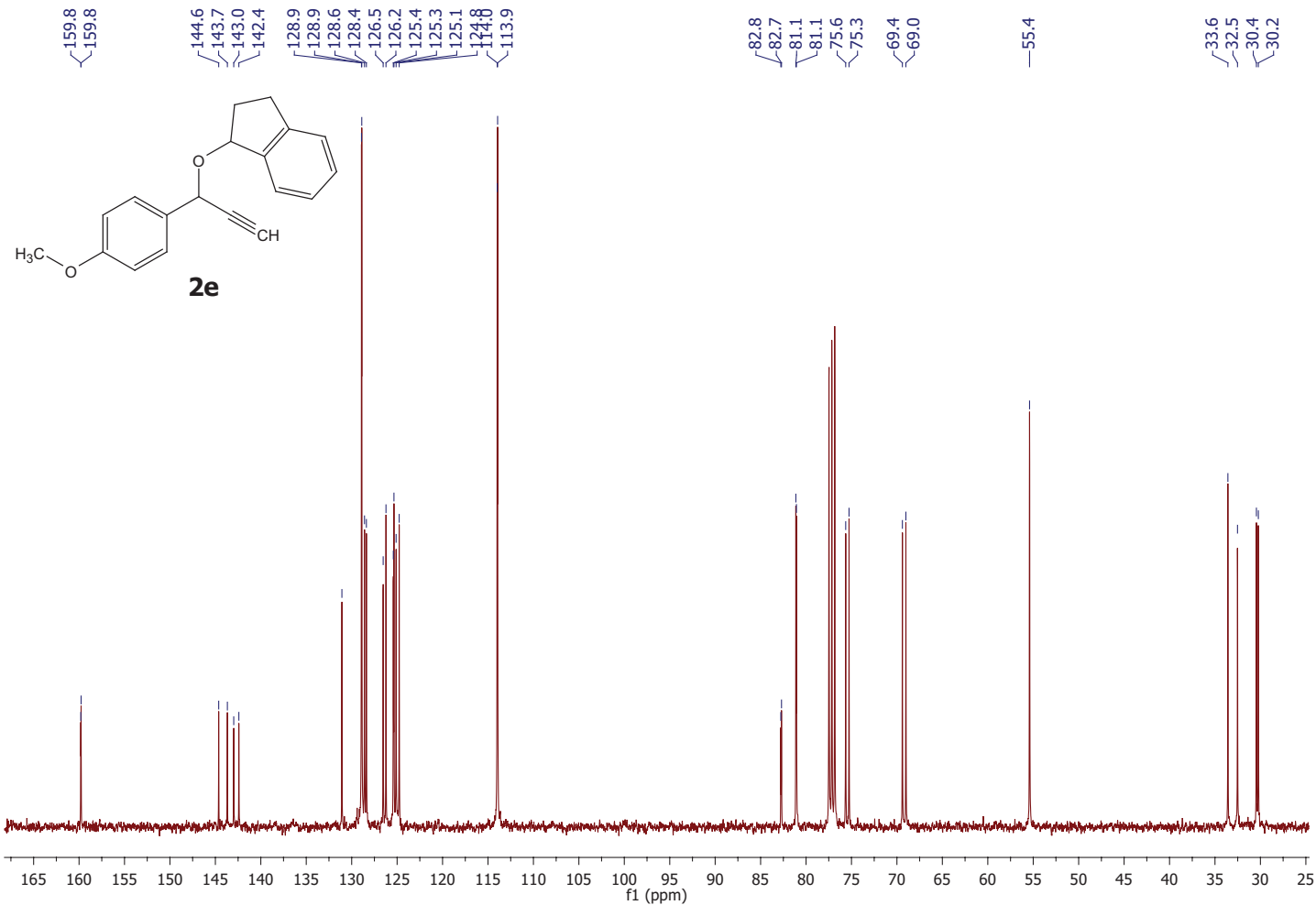
2d

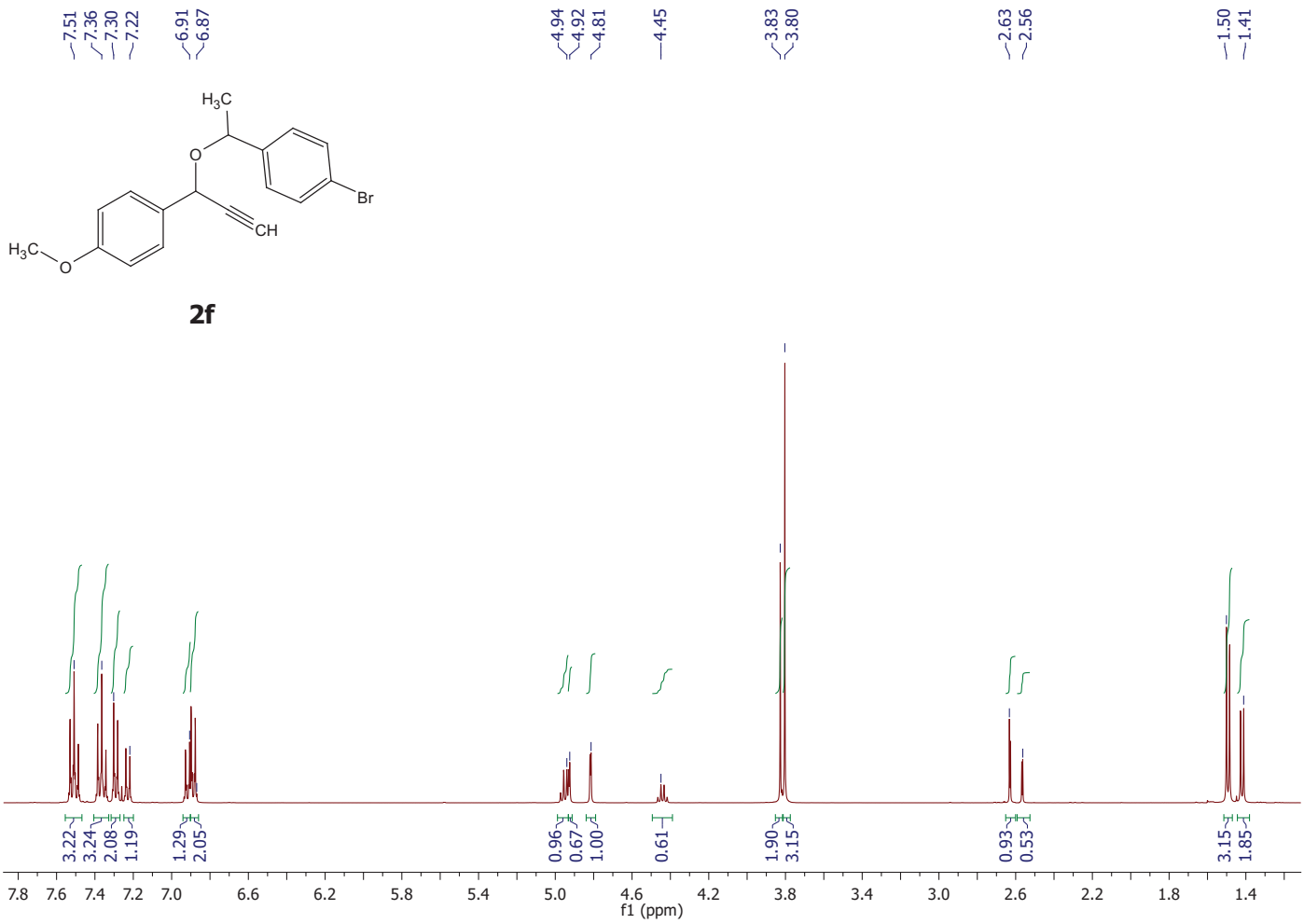

160.1
129.2
128.9
114.1
80.6
76.7
71.7
62.0
55.4
55.3
43.1
f1 (ppm)

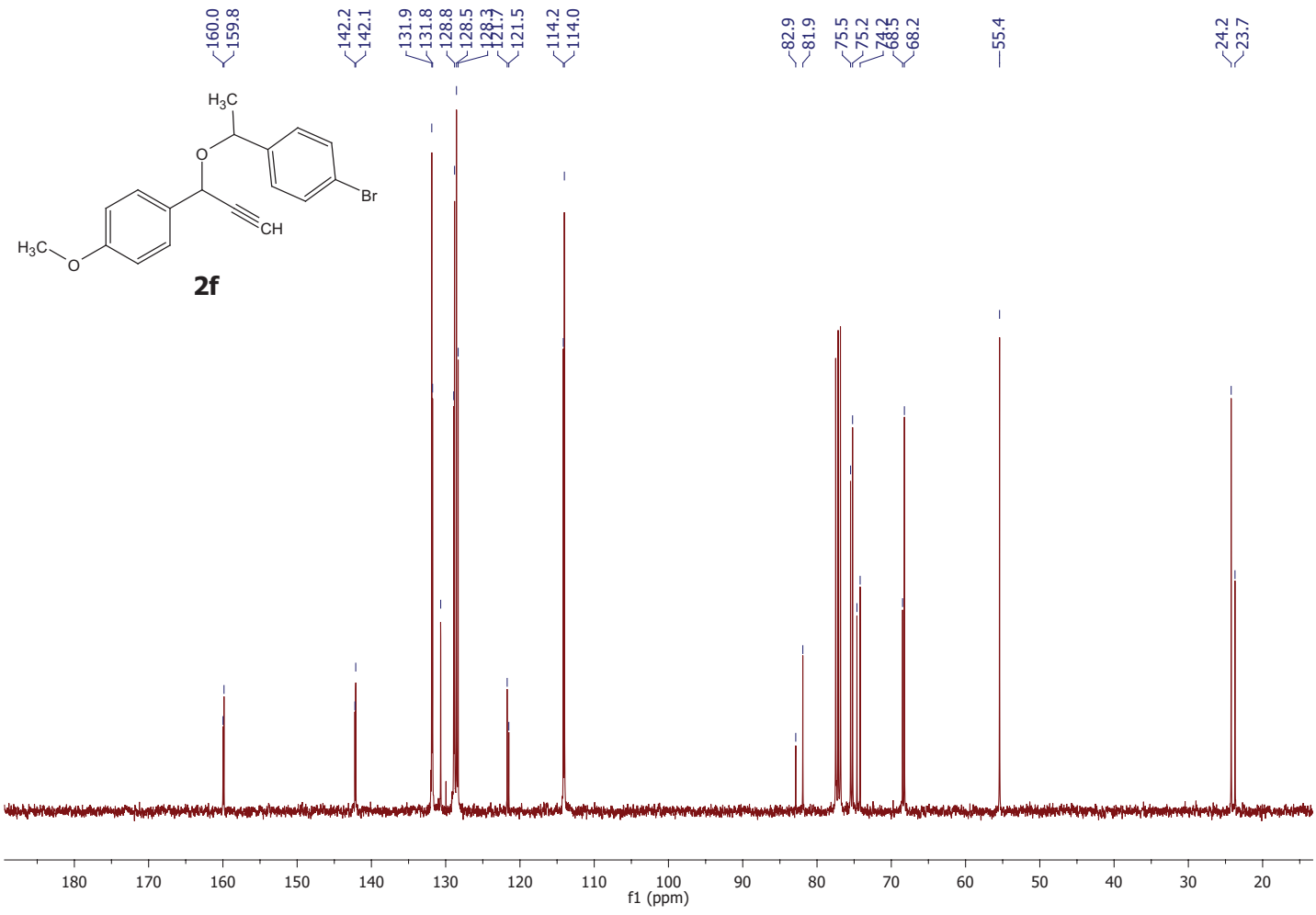
O
O
S
CH3
O
CH
H3C
O

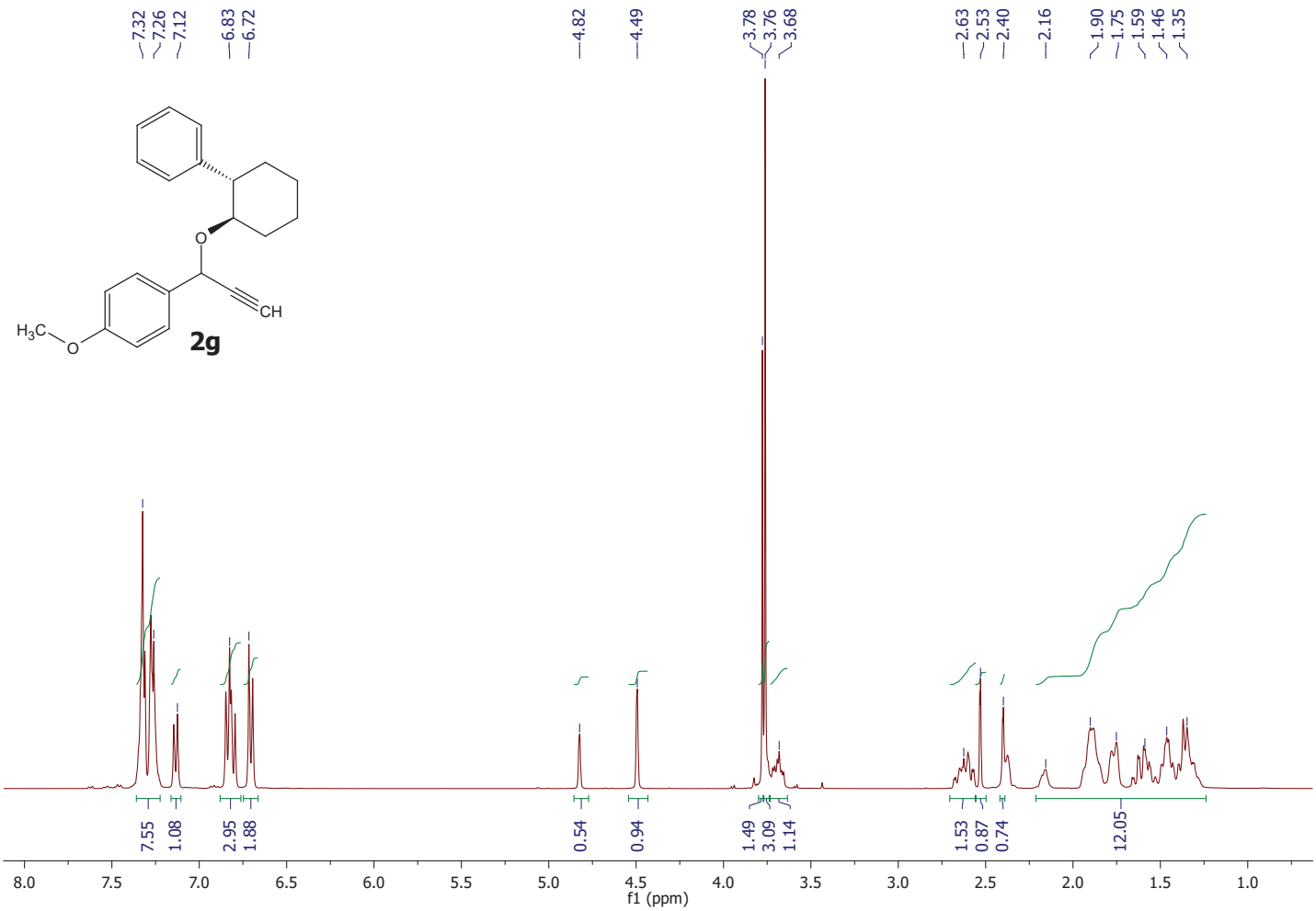
2d

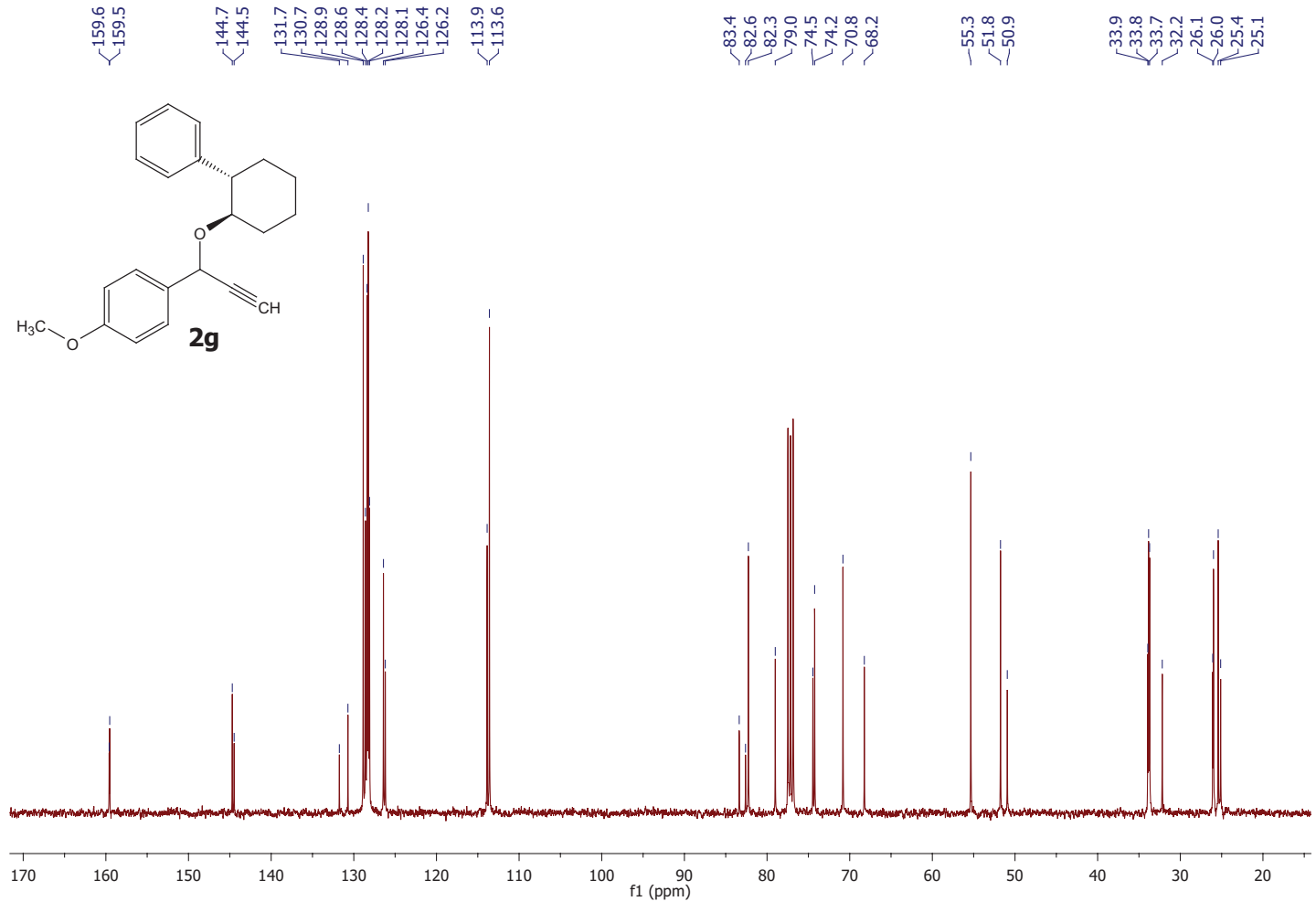

7.51
7.44
7.29
6.92
5.38
5.34
5.29
5.27
3.83
3.82
3.14
2.84
2.73
2.69
2.45
2.35
2.23
2.15
O
CH
H3C
O
2e
3.01
3.03
2.03
3.71
4.09
1.00
0.97
1.09
0.92
6.30
2.16
2.15
0.92
0.91
1.14
1.08
1.11
0.97
f1 (ppm)

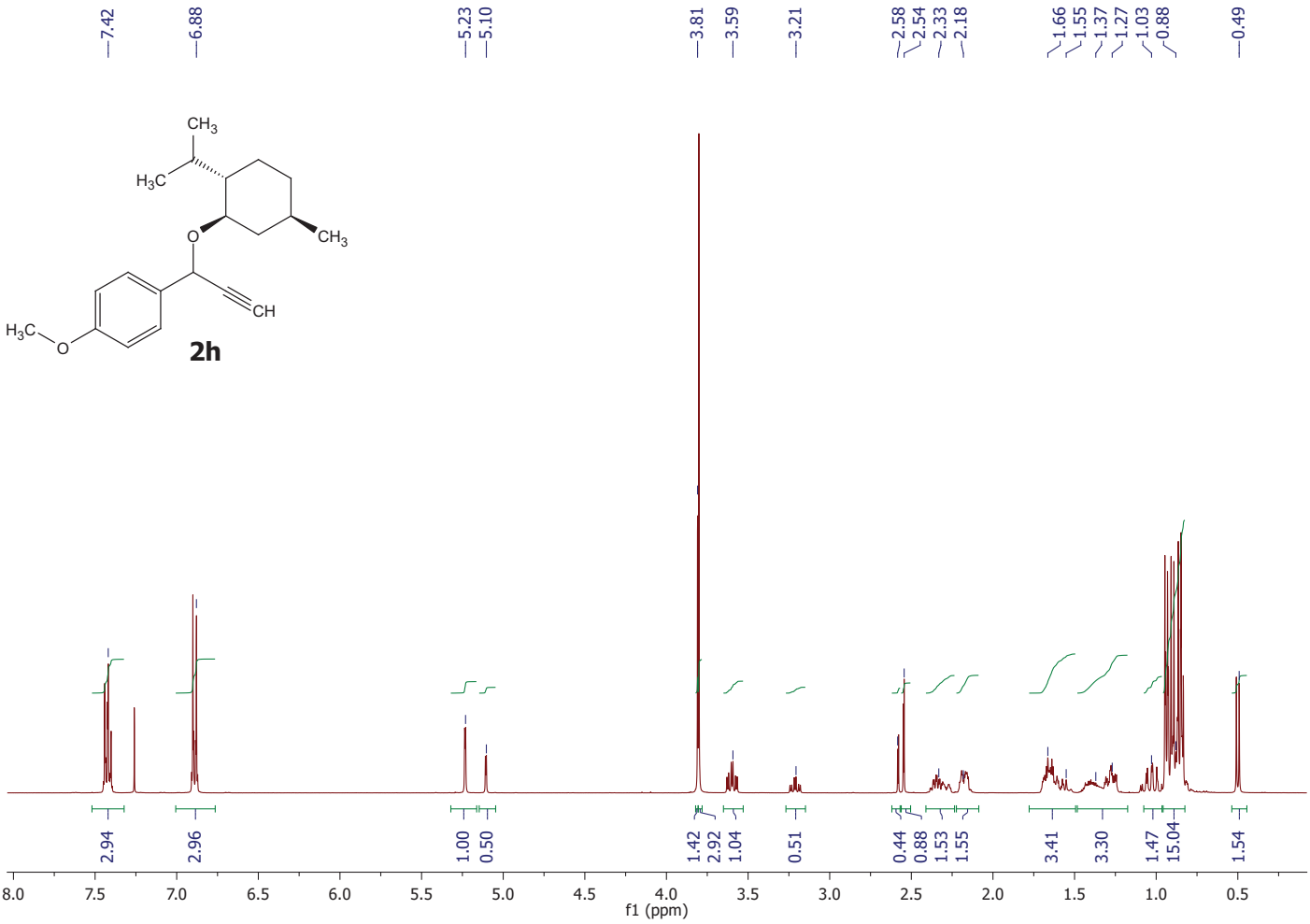

159.8
159.8
144.6
143.7
143.0
142.4
128.9
128.9
128.6
128.4
126.5
126.2
125.4
125.3
125.1
124.8
114.0
113.9
82.8
82.7
81.1
81.1
75.6
75.3
69.4
69.0
55.4
33.6
32.5
30.4
30.2
O
CH
H3C
O
2e
f1 (ppm)

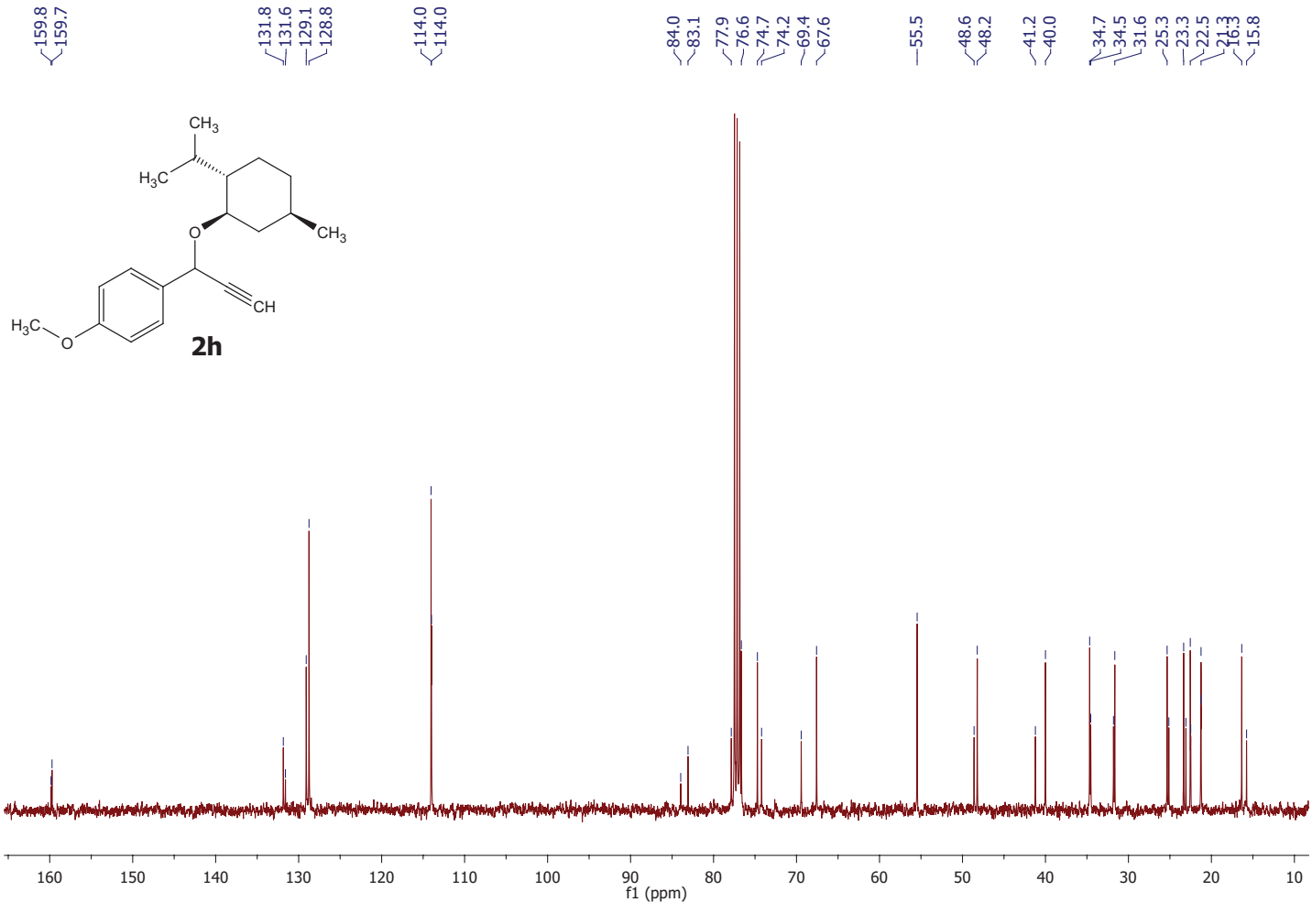

7.51
7.36
7.30
7.22
6.91
6.87
4.94
4.92
4.81
4.45
3.83
3.80
2.63
2.56
1.50
1.41
H3C
O
Br
CH
H3C
O
2f
3.22
3.24
2.08
1.19
1.29
2.05
0.96
0.67
1.00
0.61
1.90
3.15
0.93
0.53
3.15
1.85
7.8 7.6 7.4 7.2 7.0 6.6 6.2 5.8 5.4 5.0 4.6 4.2 3.8 3.4 3.0 2.6 2.2 1.8 1.4
f1 (ppm)

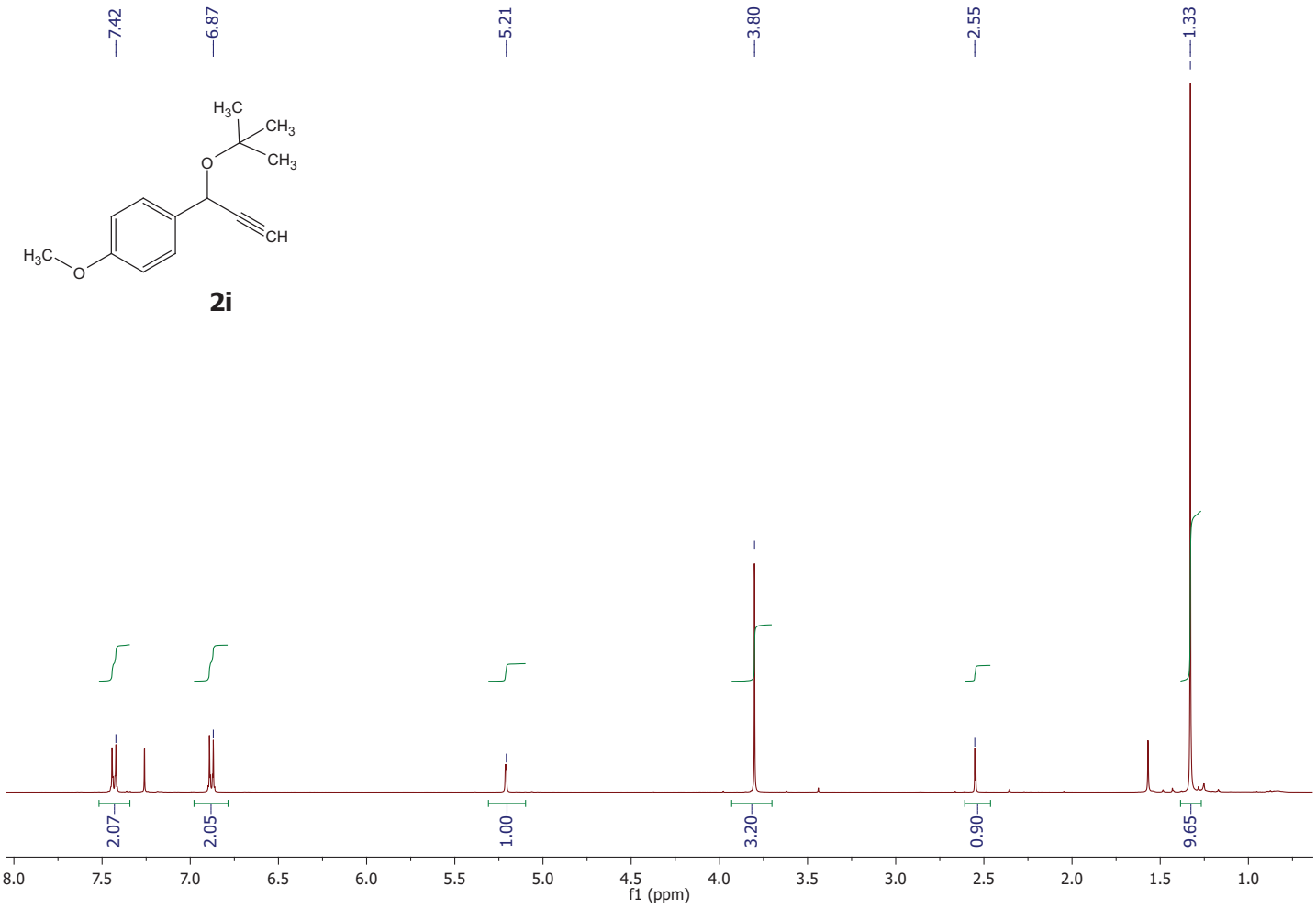

160.0
159.8
142.2
142.1
131.9
131.8
128.8
128.5
121.7
121.5
114.2
114.0
82.9
81.9
75.5
75.2
68.5
68.2
55.4
24.2
23.7
H3C
O
Br
CH
H3C
O
2f
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)


7.32
7.26
7.12
6.83
6.72
4.82
4.49
3.78
3.76
3.68
2.63
2.53
2.40
2.16
1.90
1.75
1.59
1.46
1.35
O
CH
H3C
O
2g
7.55
1.08
2.95
1.88
0.54
0.94
1.49
3.09
1.14
1.53
0.87
0.74
12.05
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
f1 (ppm)

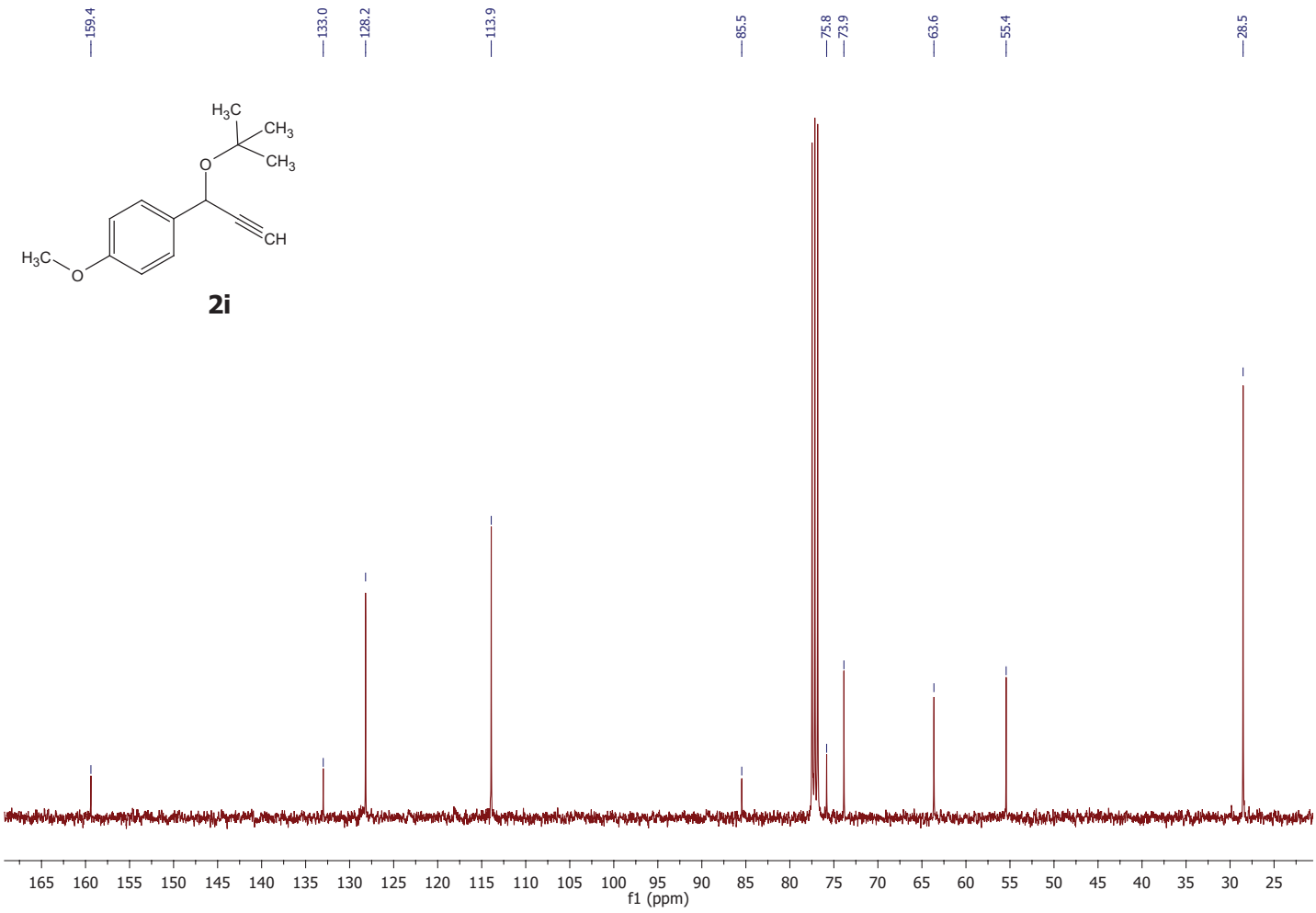

159.6
159.5
144.7
144.5
131.7
130.7
128.9
128.6
128.4
128.2
128.1
126.4
126.2
113.9
113.6
83.4
82.6
82.3
79.0
74.5
74.2
70.8
68.2
55.3
51.8
50.9
33.9
33.8
33.7
32.2
26.1
26.0
25.4
25.1
O
CH
H3C
O
2g
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


7.42
6.88
5.23
5.10
3.81
3.59
3.21
2.58
2.54
2.33
2.18
1.66
1.55
1.37
1.27
1.03
0.88
0.49
CH3
H3C
CH3
O
CH
H3C
O
2h
2.94
2.96
1.00
0.50
1.42
2.92
1.04
0.51
0.44
0.88
1.53
1.55
3.41
3.30
1.47
15.04
1.54
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

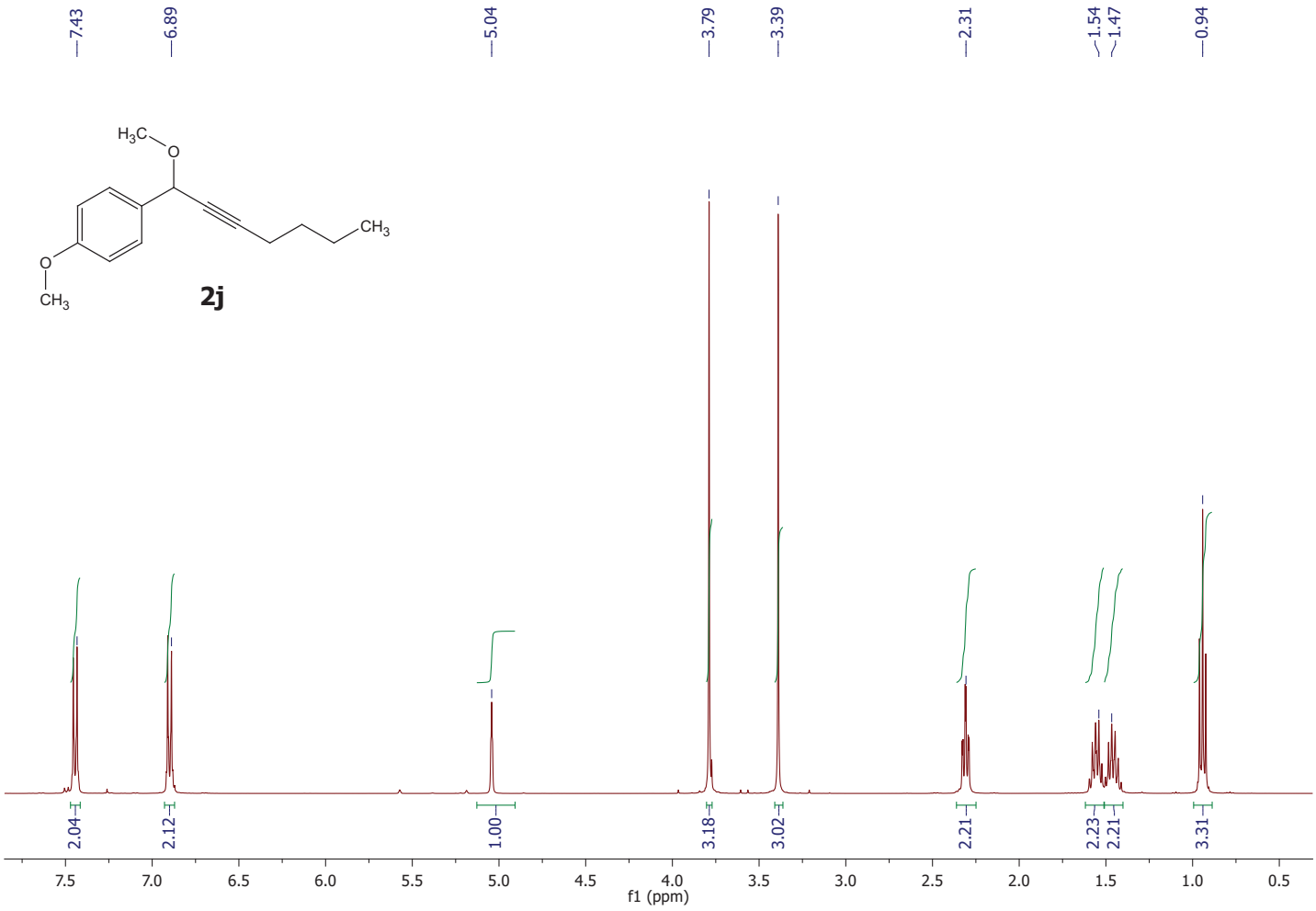

159.8
159.7
131.8
131.6
129.1
128.8
114.0
114.0
84.0
83.1
77.9
76.6
74.7
74.2
69.4
67.6
55.5
48.6
48.2
41.2
40.0
34.7
34.5
31.6
25.3
23.3
22.5
21.3
16.3
15.8
CH3
H3C
CH3
O
CH
H3C
O
2h
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.42
6.87
5.21
3.80
2.55
1.33
H3C
CH3
CH3
O
CH
H3C
O
2.07
2.05
1.00
3.20
0.90
9.65
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
f1 (ppm)

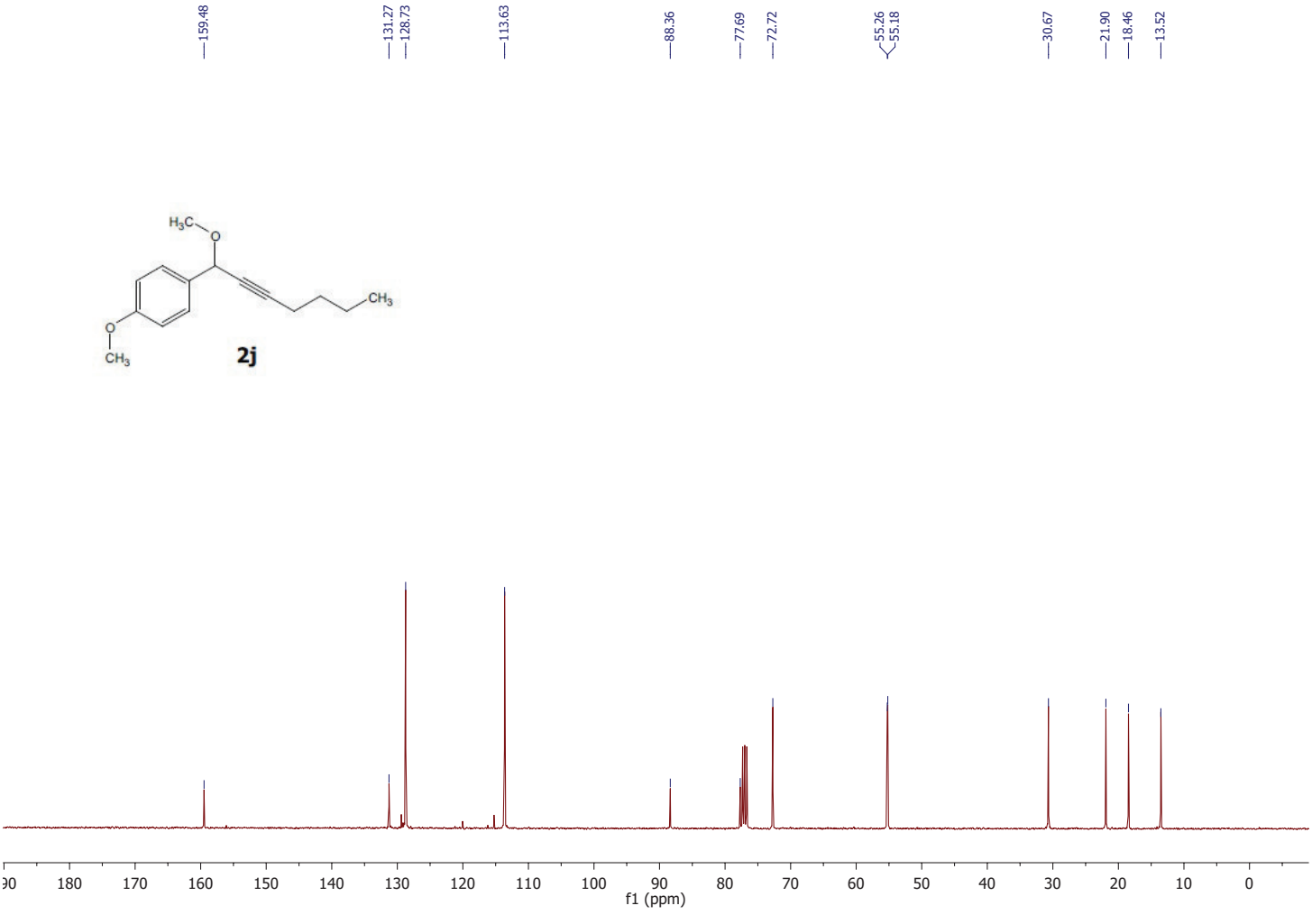
2i


159.4
133.0
128.2
113.9
85.5
75.8
73.9
63.6
55.4
28.5
H3C
CH3
CH3
O
CH
H3C
O
165
160
155
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
f1 (ppm)

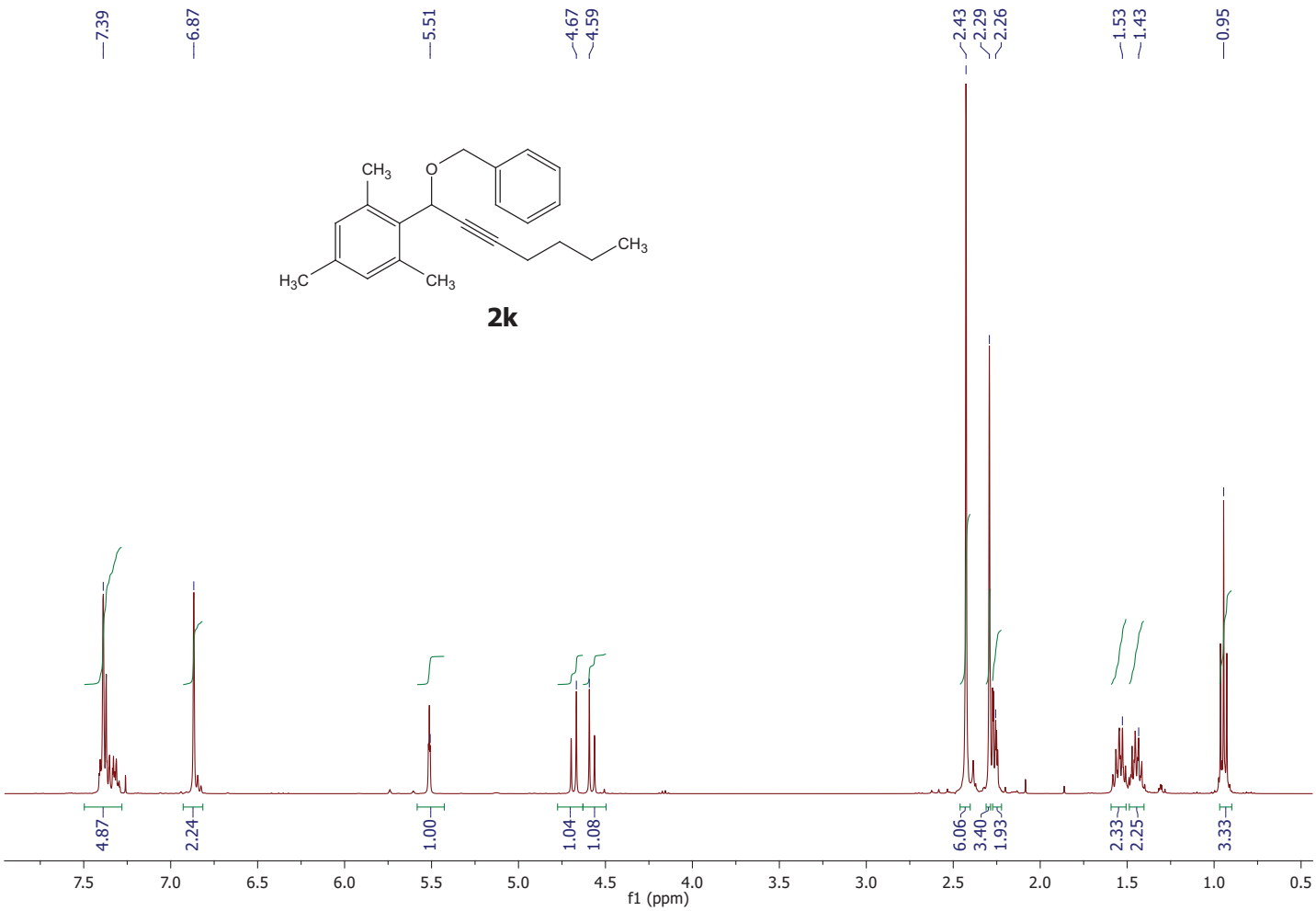
2i


7.43
6.89
5.04
3.79
3.39
2.31
1.54
1.47
0.94
H3C
O
CH3
O
CH3
2.04
2.12
1.00
3.18
3.02
2.21
2.23
2.21
3.31
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

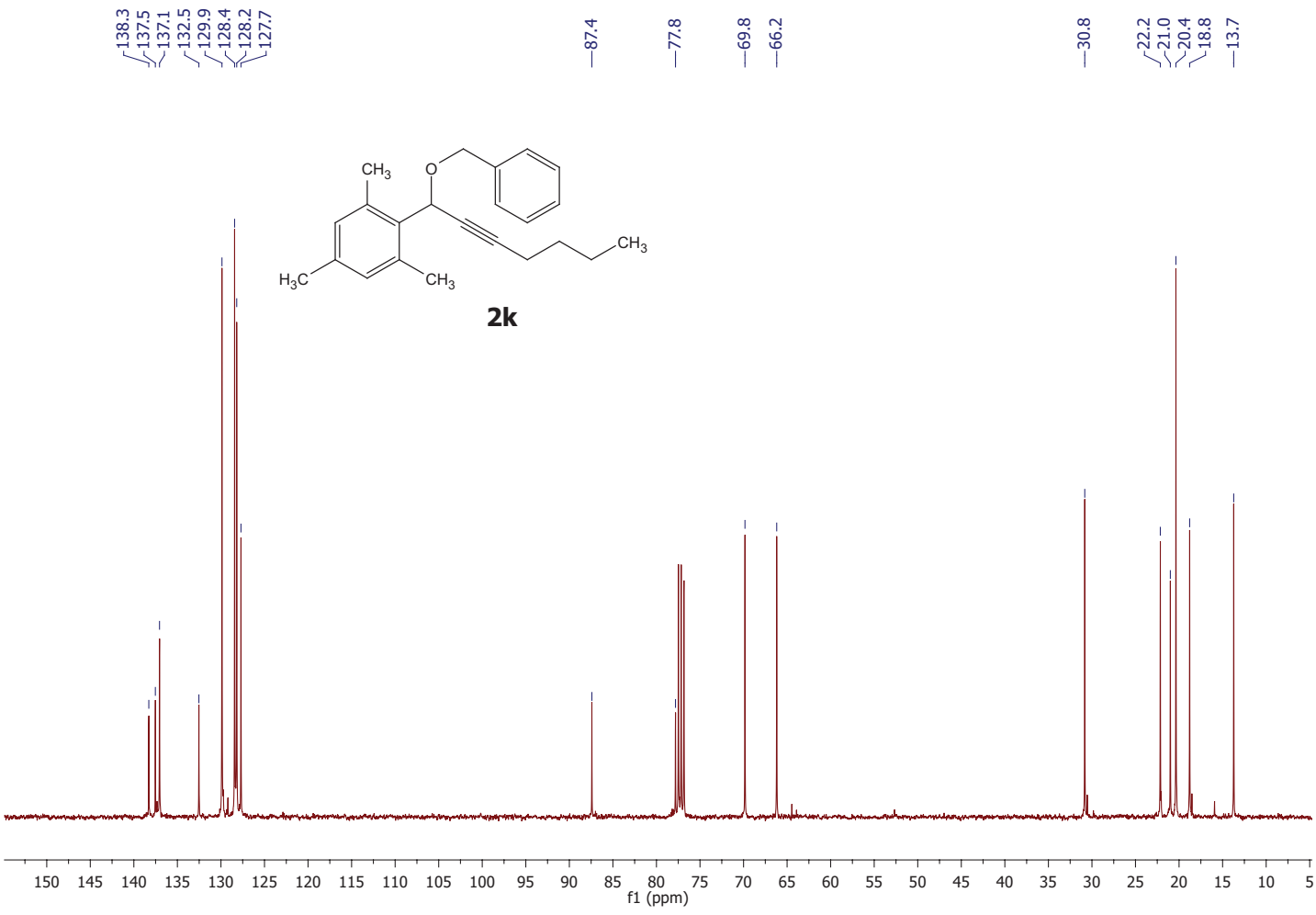
2j


159.48
131.27
128.73
113.63
88.36
77.69
72.72
55.26
55.18
30.67
21.90
18.46
13.52
H3C
O
CH3
O
CH3
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

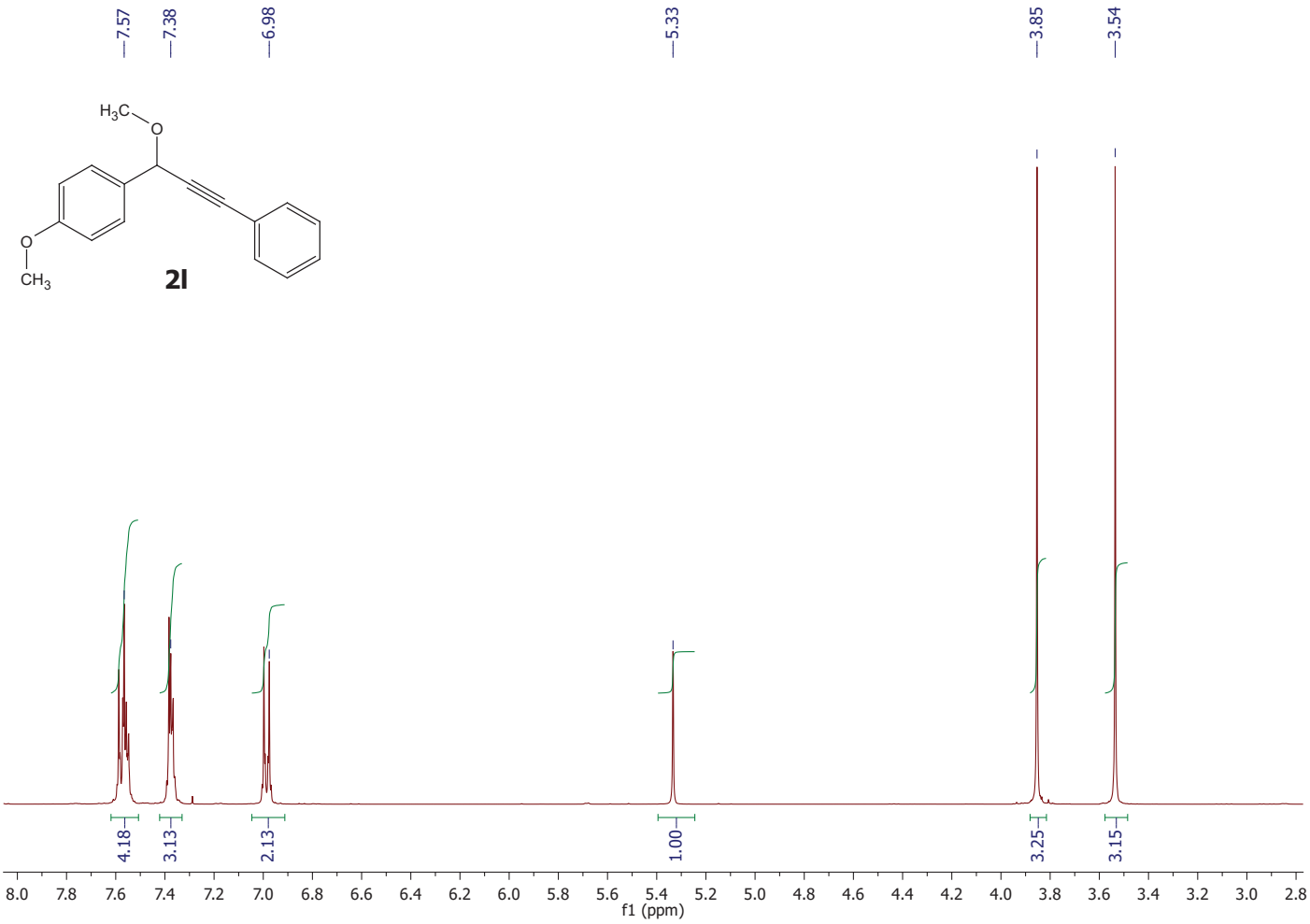
2j

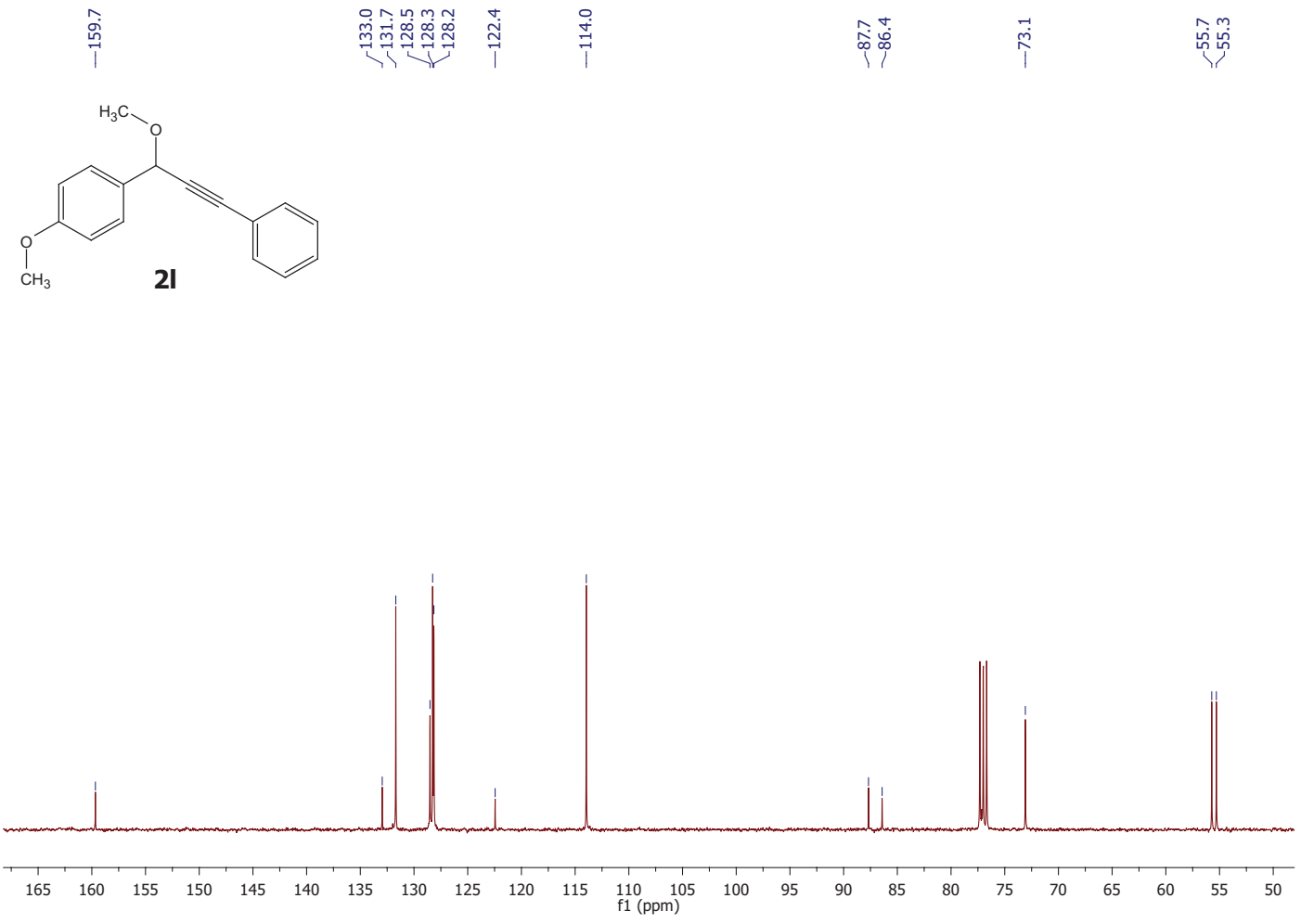

7.39
6.87
5.51
4.67
4.59
2.43
2.29
2.26
1.53
1.43
0.95
CH3
O
CH3
H3C
CH3
4.87
2.24
1.00
1.04
1.08
6.06
3.40
1.93
2.33
2.25
3.33
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

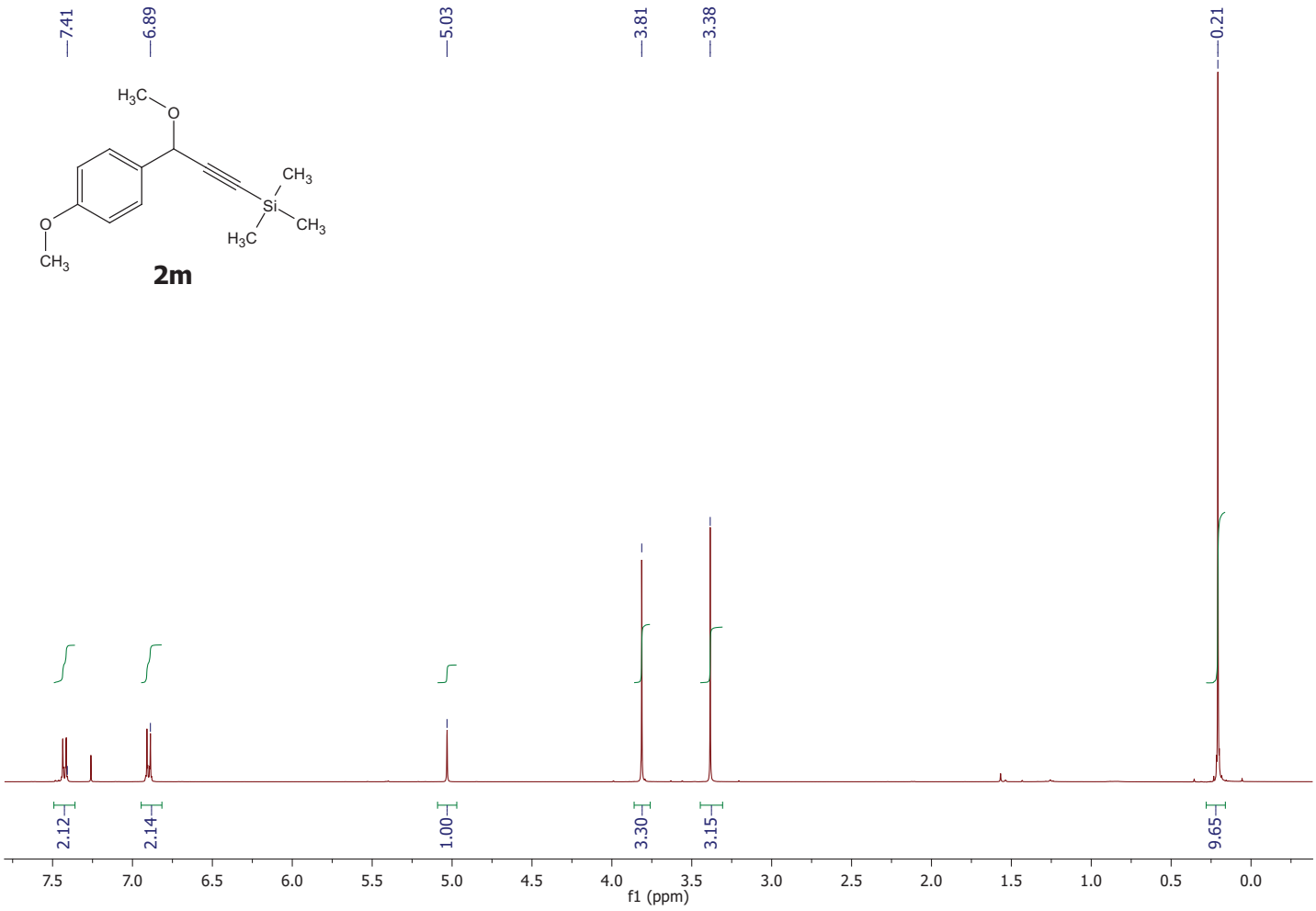
2k


138.3
137.5
137.1
132.5
129.9
128.4
128.2
127.7
87.4
77.8
69.8
66.2
30.8
22.2
21.0
20.4
18.8
13.7
CH3
O
CH3
H3C
CH3
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
f1 (ppm)

2k

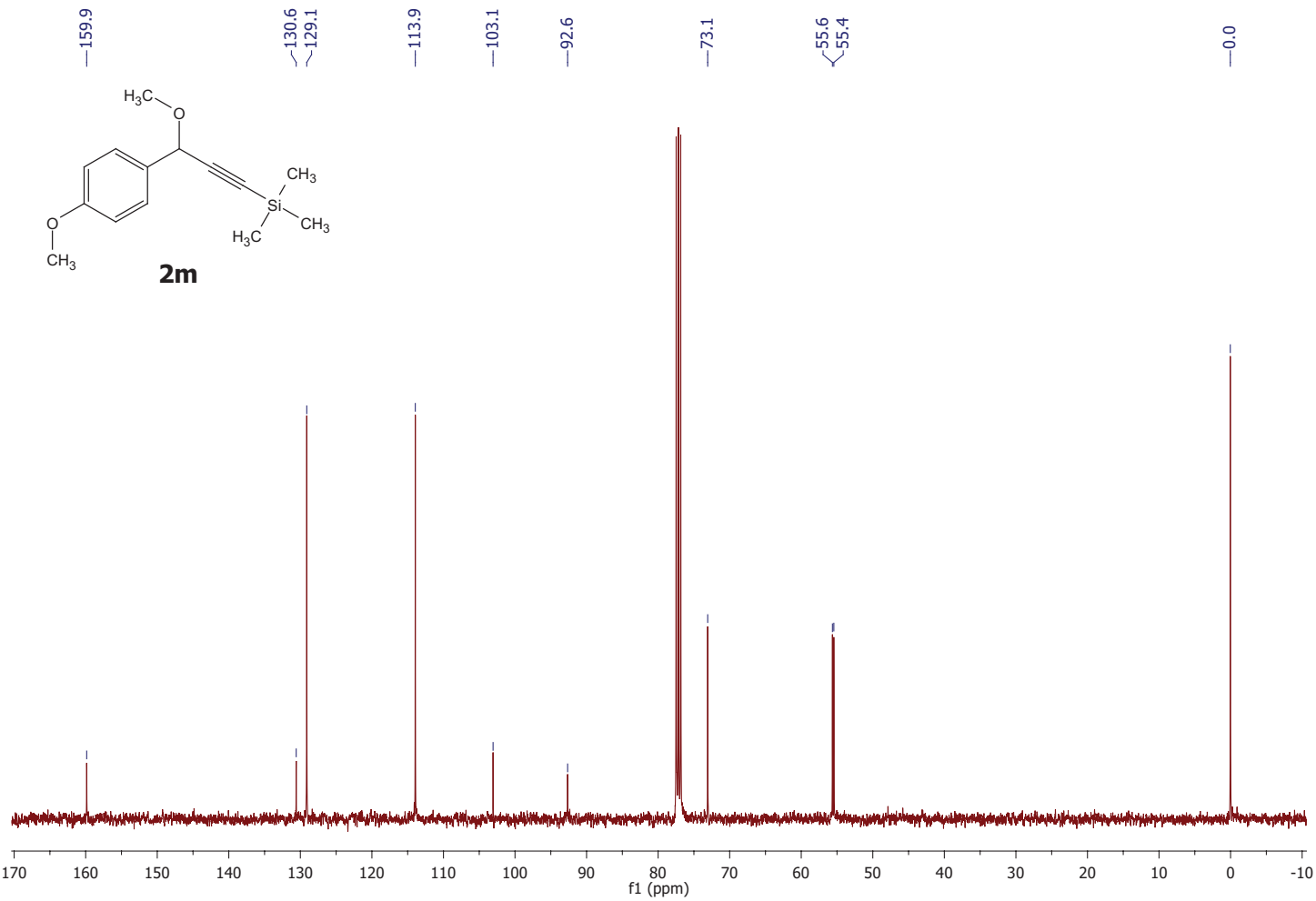

7.57
7.38
6.98
5.33
3.85
3.54
H3C
O
O
CH3
2l
4.18
3.13
2.13
1.00
3.25
3.15
8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8
f1 (ppm)


159.7
133.0
131.7
128.5
128.3
128.2
122.4
114.0
87.7
86.4
73.1
55.7
55.3
H3C
O
O
CH3
2l
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50
f1 (ppm)


7.41
6.89
5.03
3.81
3.38
0.21
2.12
2.14
1.00
3.30
3.15
9.65
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)

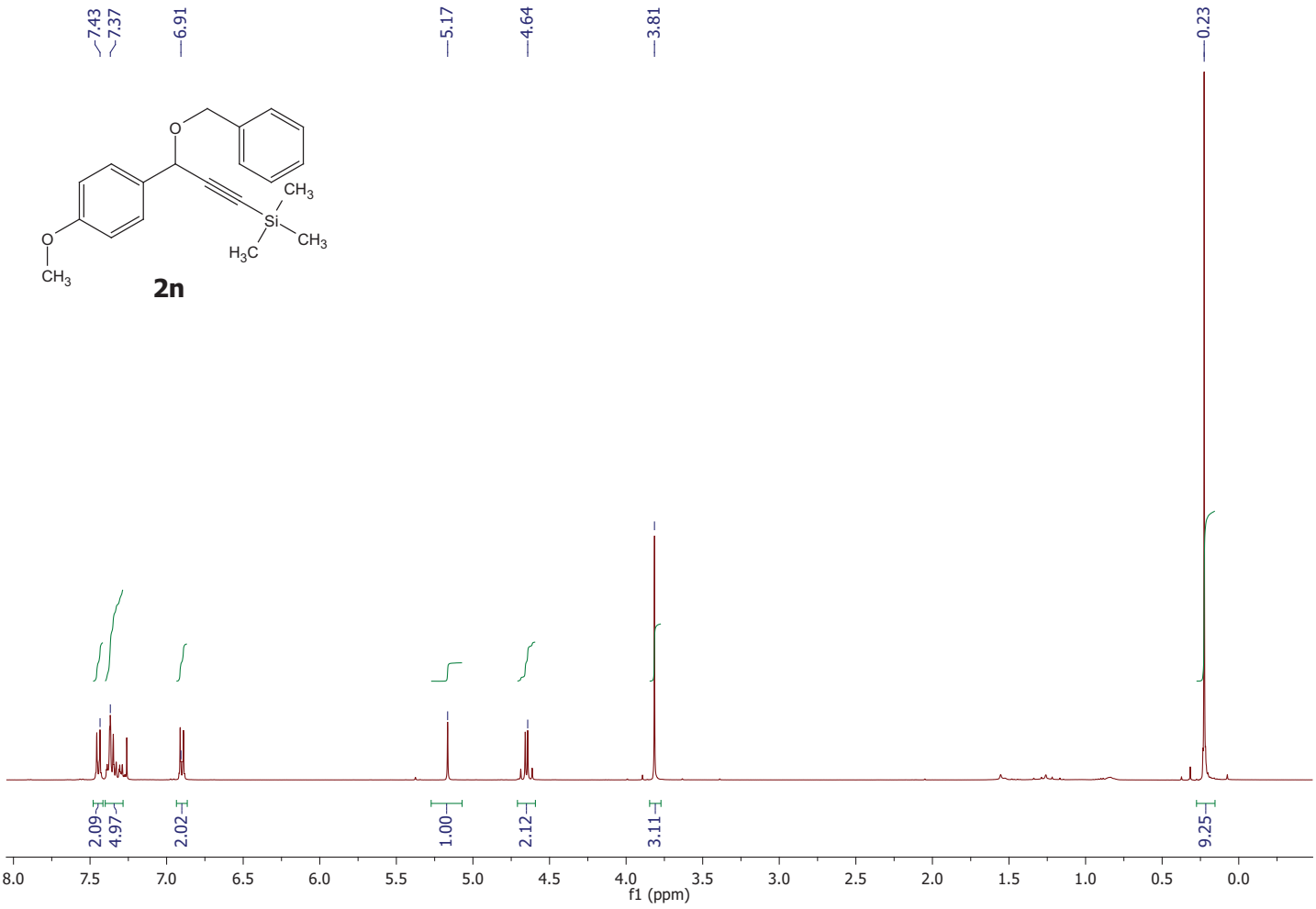
H3C
O
CH3
Si
CH3
H3C
O
CH3

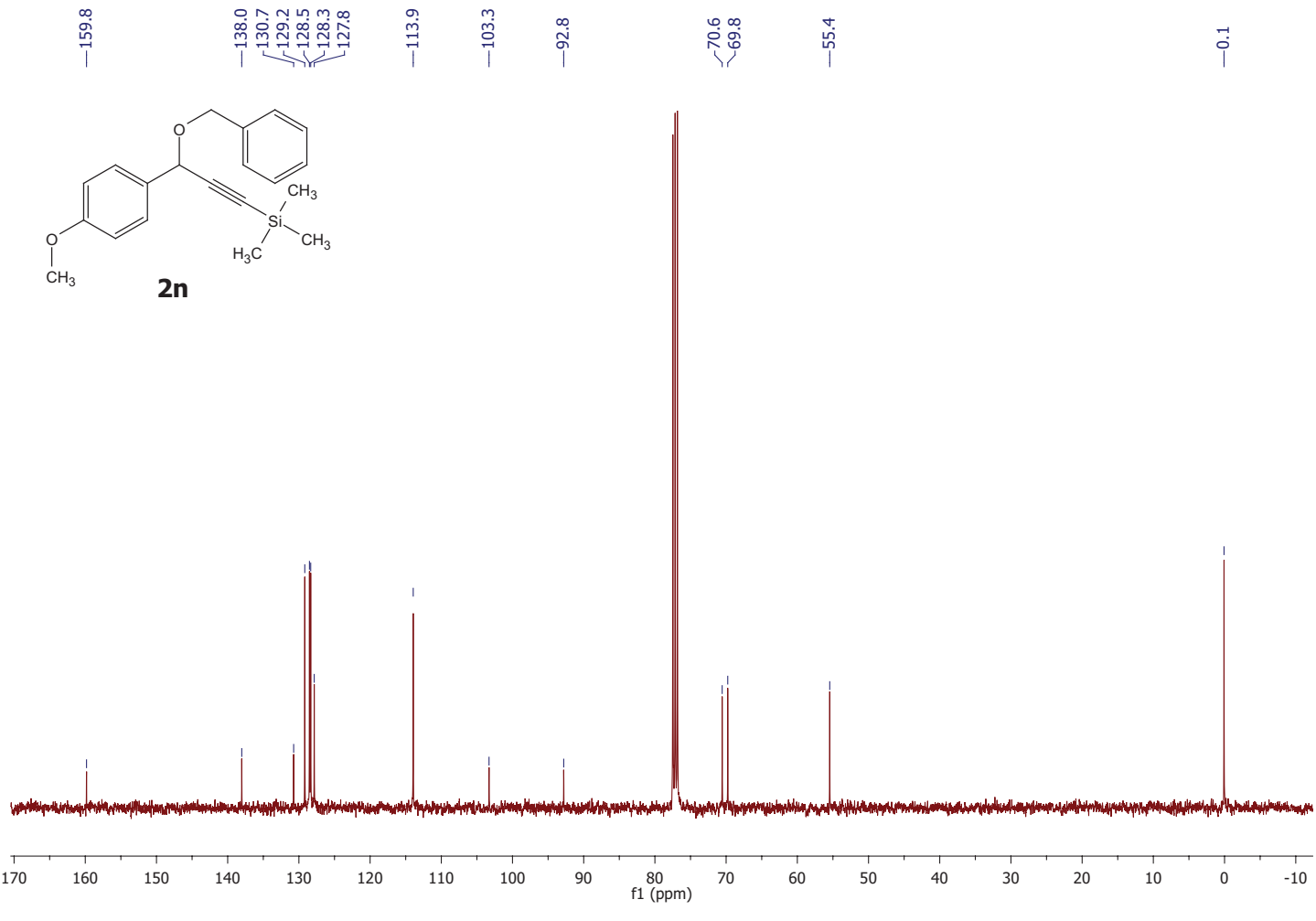
2m

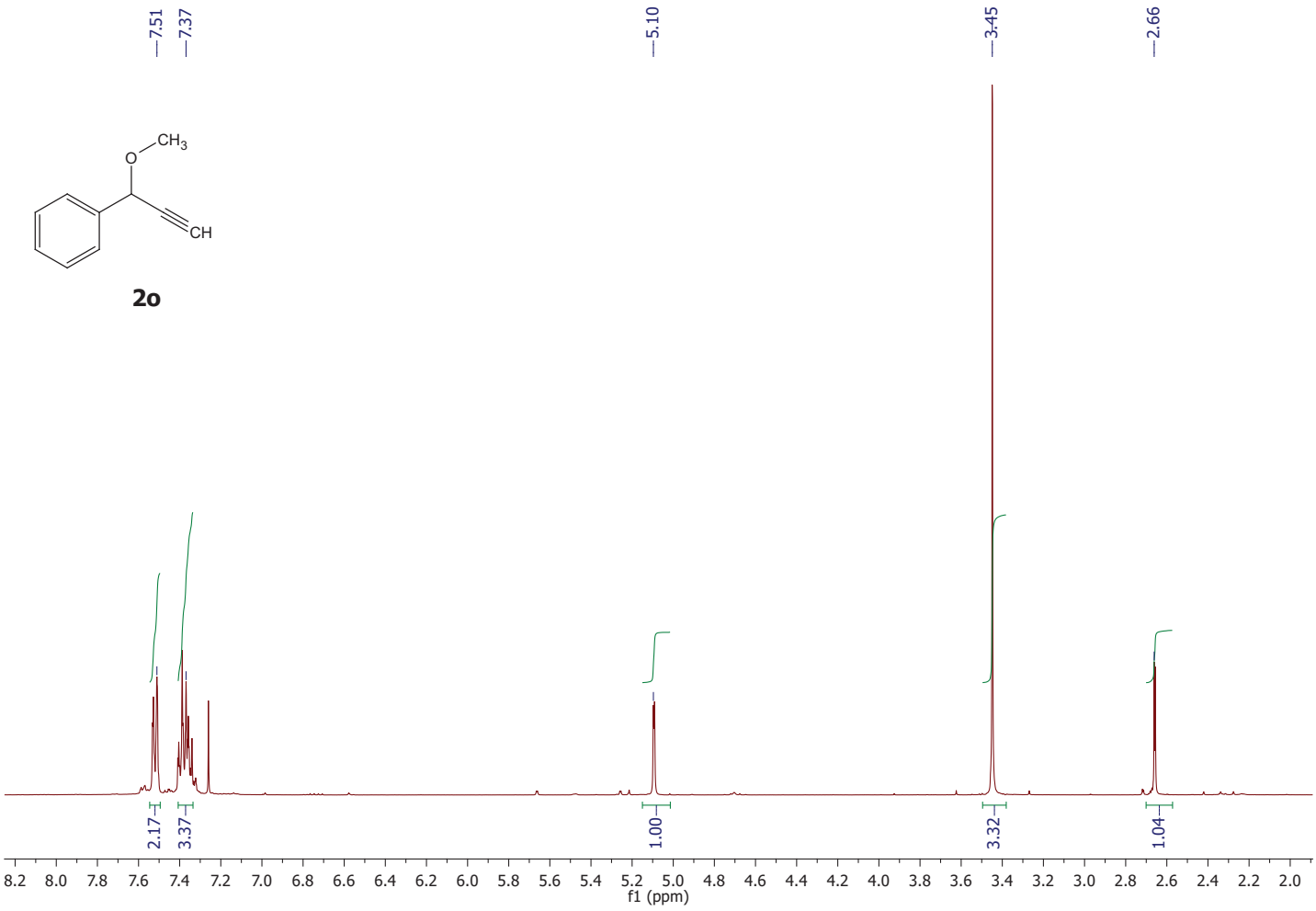

159.9
130.6
129.1
113.9
103.1
92.6
73.1
55.6
55.4
0.0
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

H3C
O
CH3
Si
CH3
H3C
O
CH3

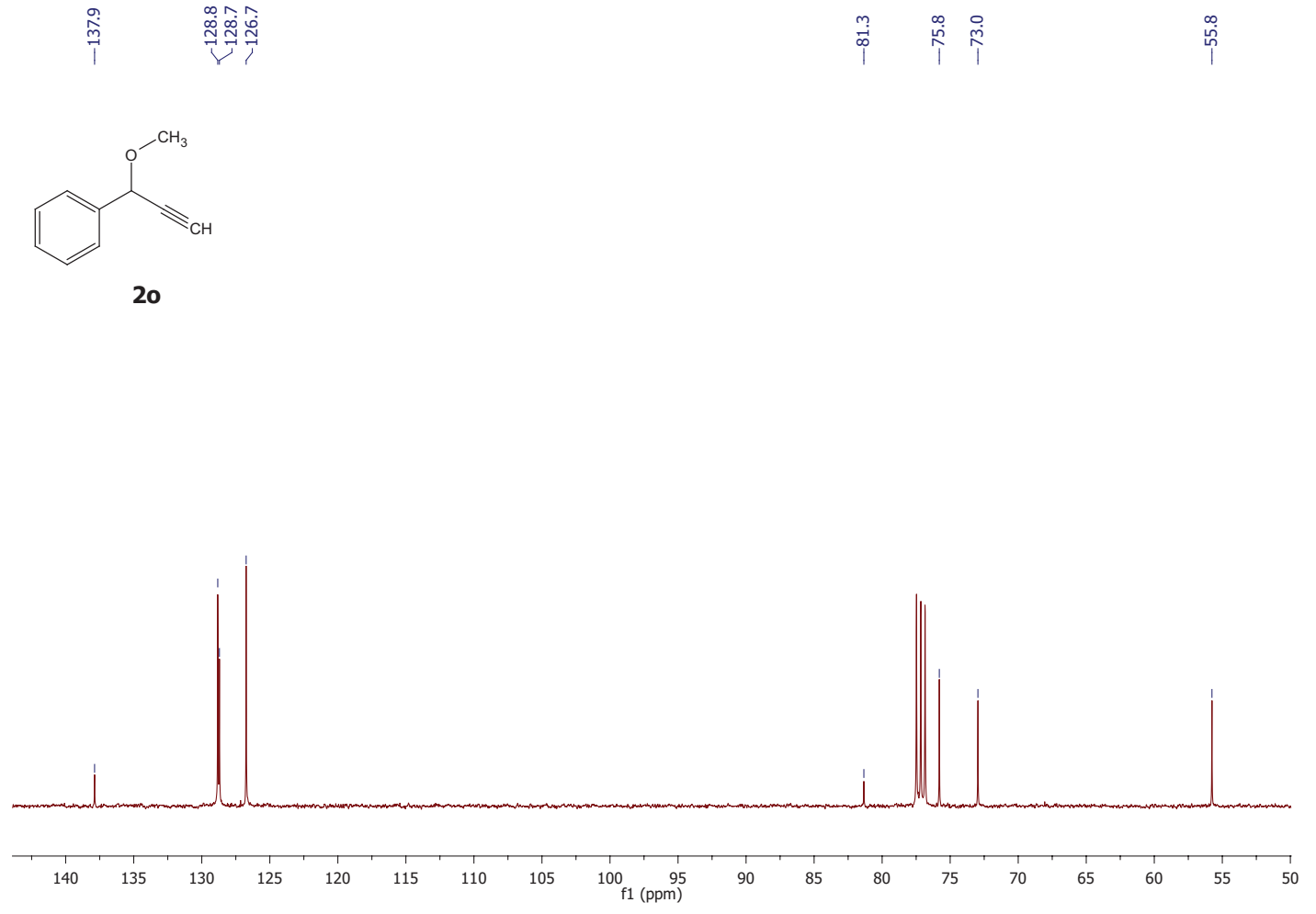
2m


7.43
7.37
6.91
5.17
4.64
3.81
0.23
O
CH3
Si
CH3
H3C
CH3
O
CH3
2n
2.09
4.97
2.02
1.00
2.12
3.11
9.25
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)

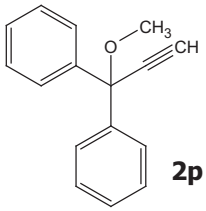

159.8
138.0
130.7
129.2
128.5
128.3
127.8
113.9
103.3
92.8
70.6
69.8
55.4
0.1
O
CH3
Si
CH3
H3C
CH3
O
CH3
2n
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.51
7.37
5.10
3.45
2.66
CH3
O
CH
2.17
3.37
1.00
3.32
1.04
f1 (ppm)

2o

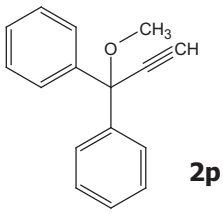

137.9
128.8
128.7
126.7
81.3
75.8
73.0
55.8
CH3
O
CH
f1 (ppm)

2o


CH3
O
CH

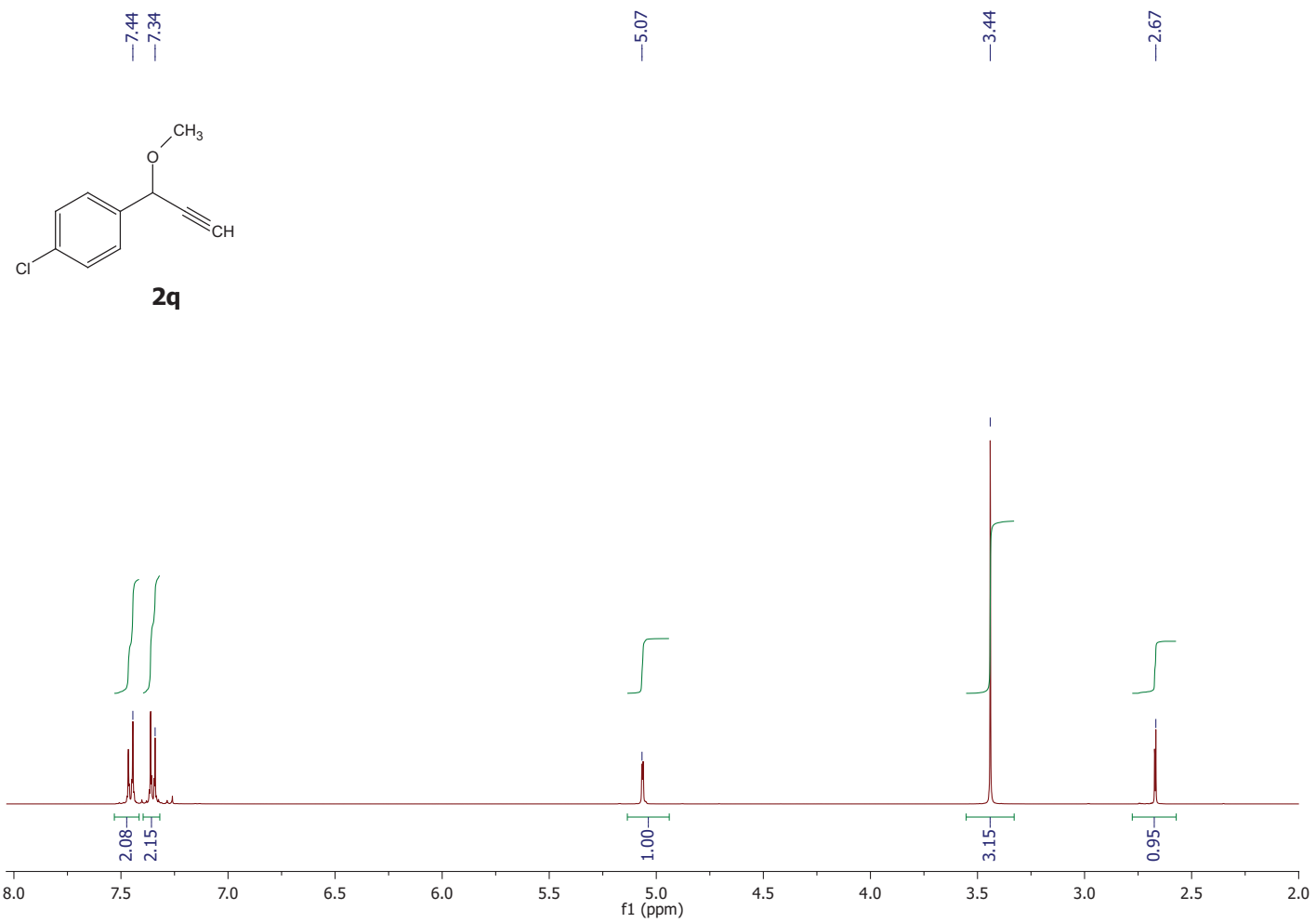
2p

7.55
7.33
7.26
7.25
3.36
2.90
3.40
7.11
3.00
1.04
7.8
7.4
7.0
6.6
6.2
5.8
5.4
5.0
4.6
4.2
3.8
3.4
3.0
2.6
f1 (ppm)

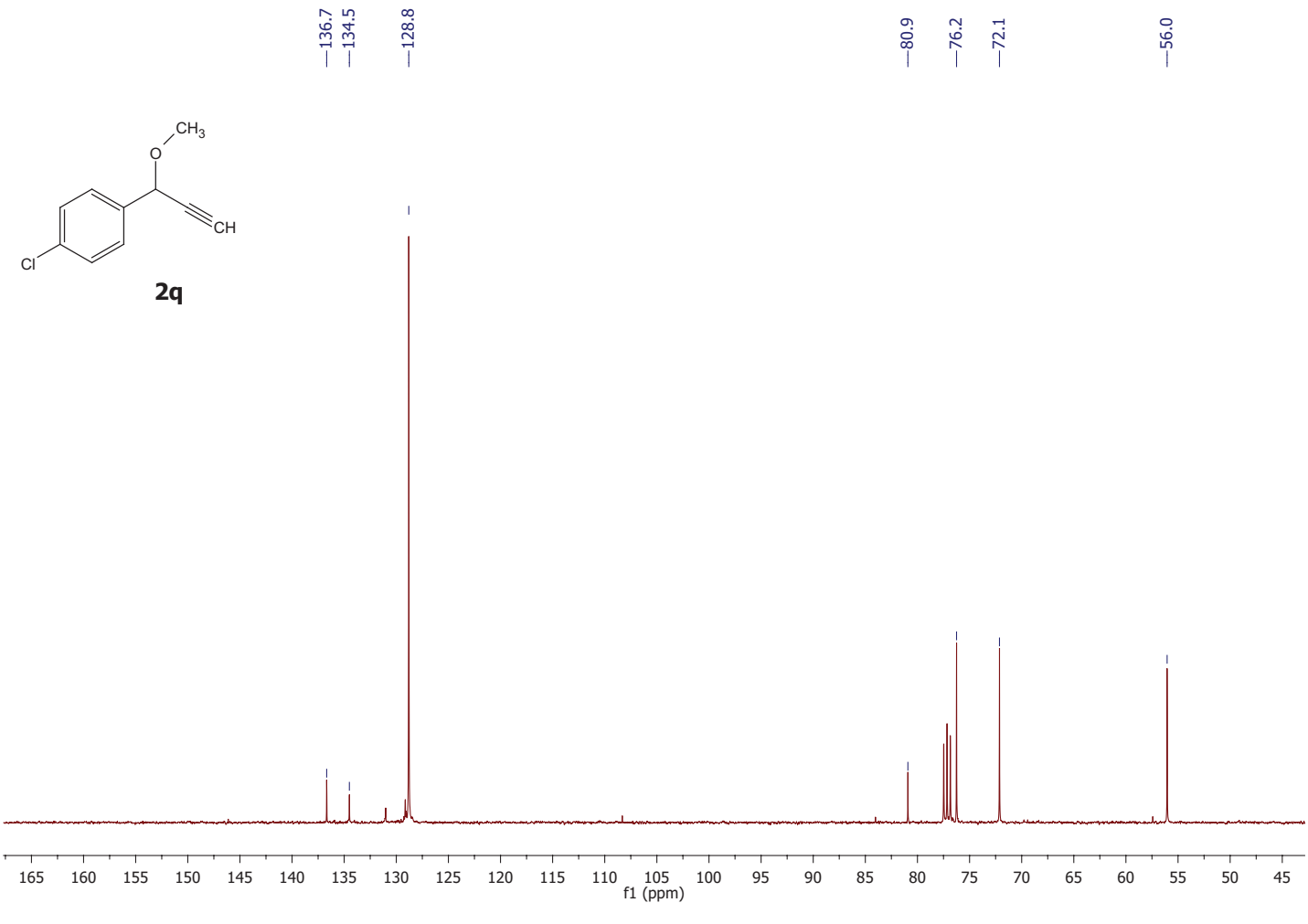

CH3
O
CH

2p

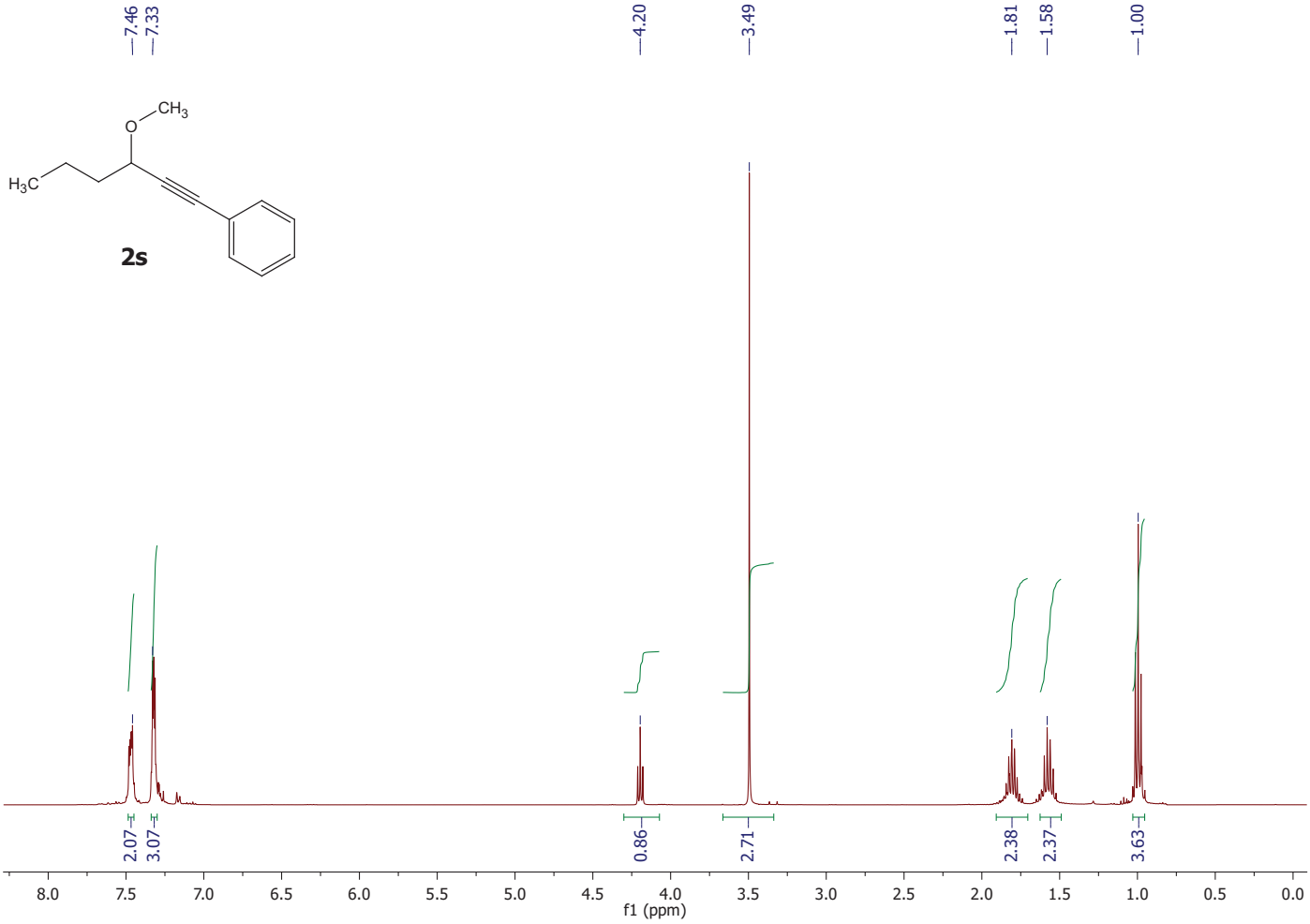
144.5
127.9
127.8
127.2
126.0
86.5
75.6
74.3
52.5
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
f1 (ppm)

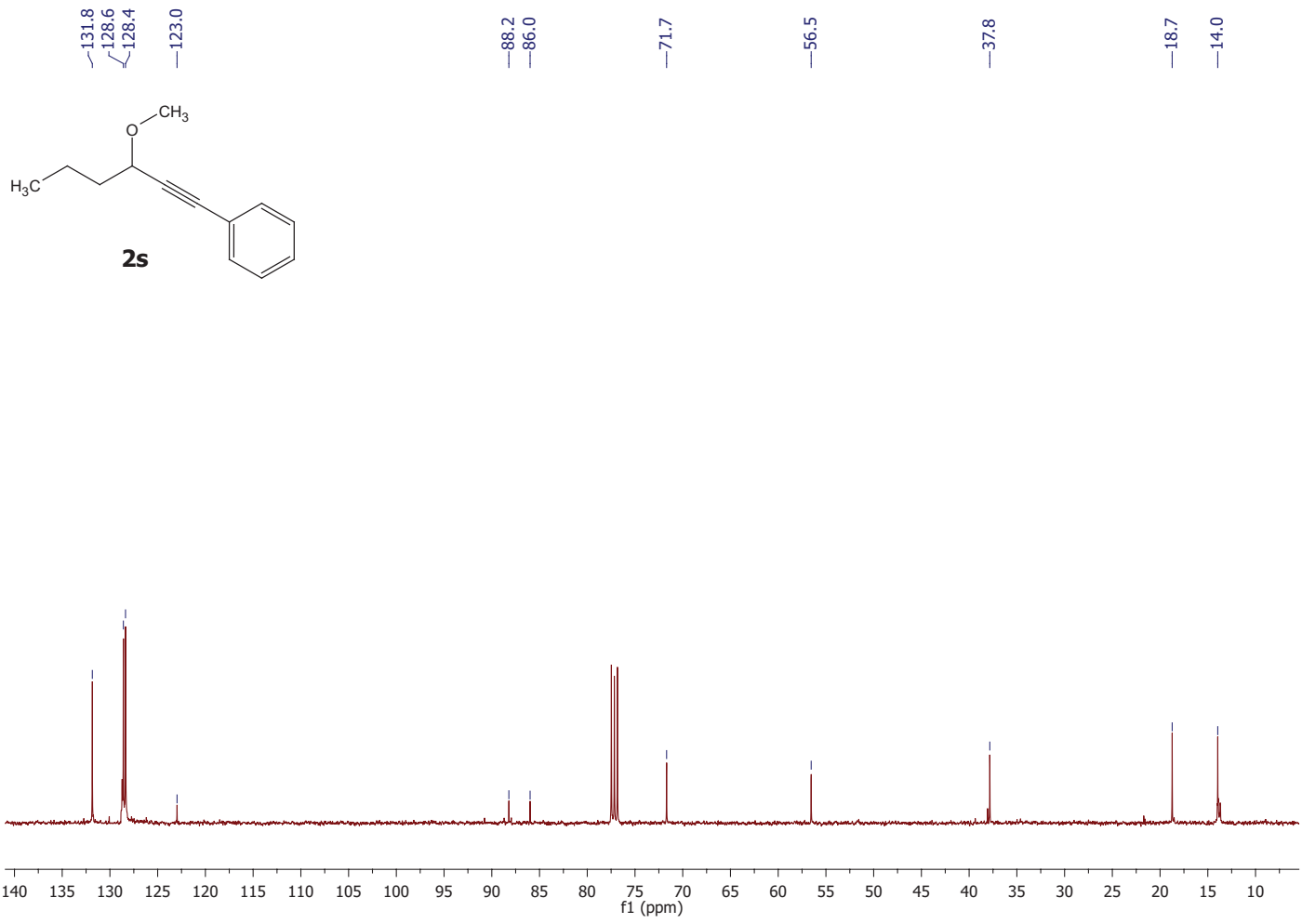

7.44
7.34
5.07
3.44
2.67
CH3
O
CH
Cl
2.08
2.15
1.00
3.15
0.95
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f1 (ppm)

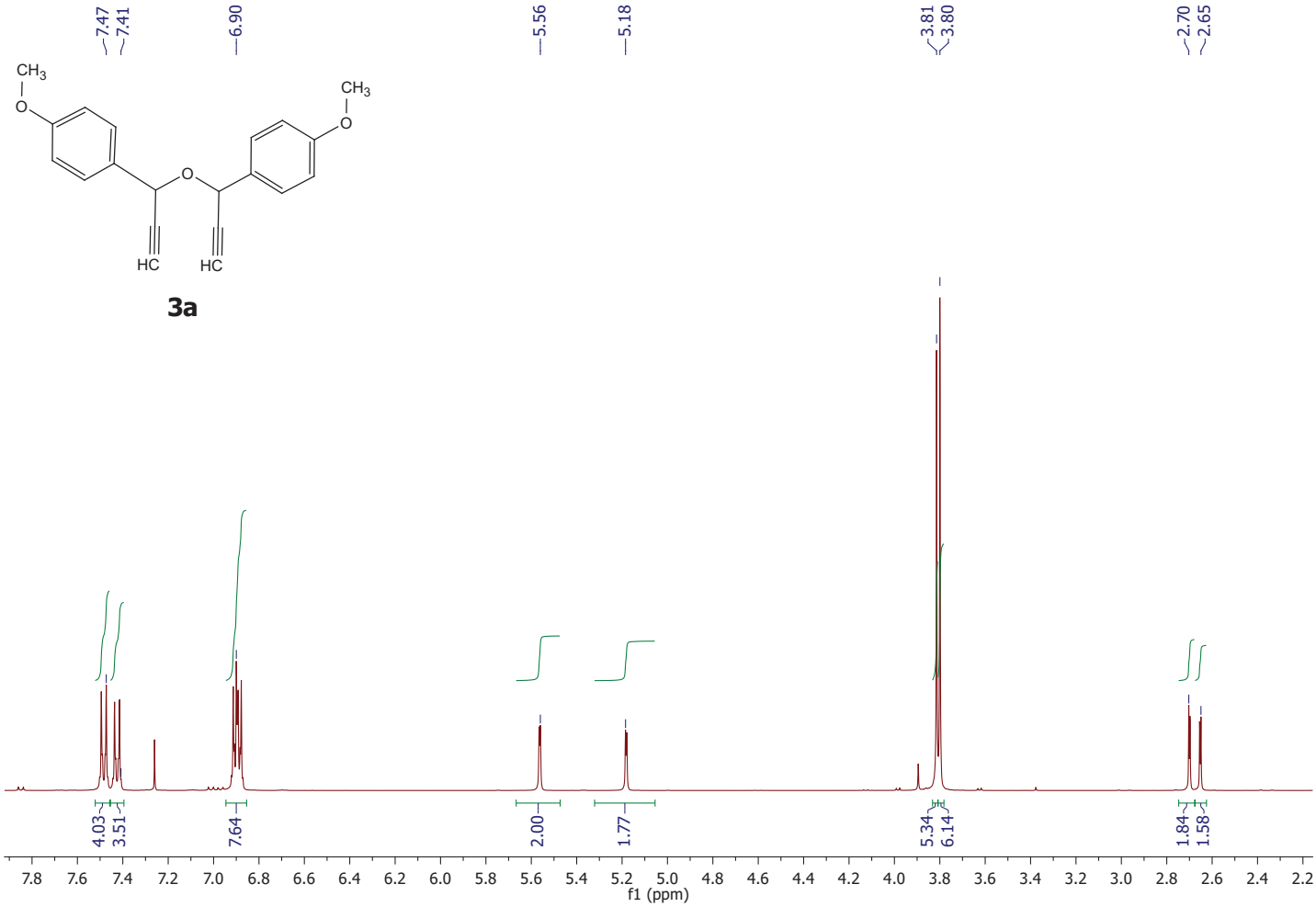
2q


136.7
134.5
128.8
80.9
76.2
72.1
56.0
CH3
O
CH
Cl
165
160
155
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
f1 (ppm)

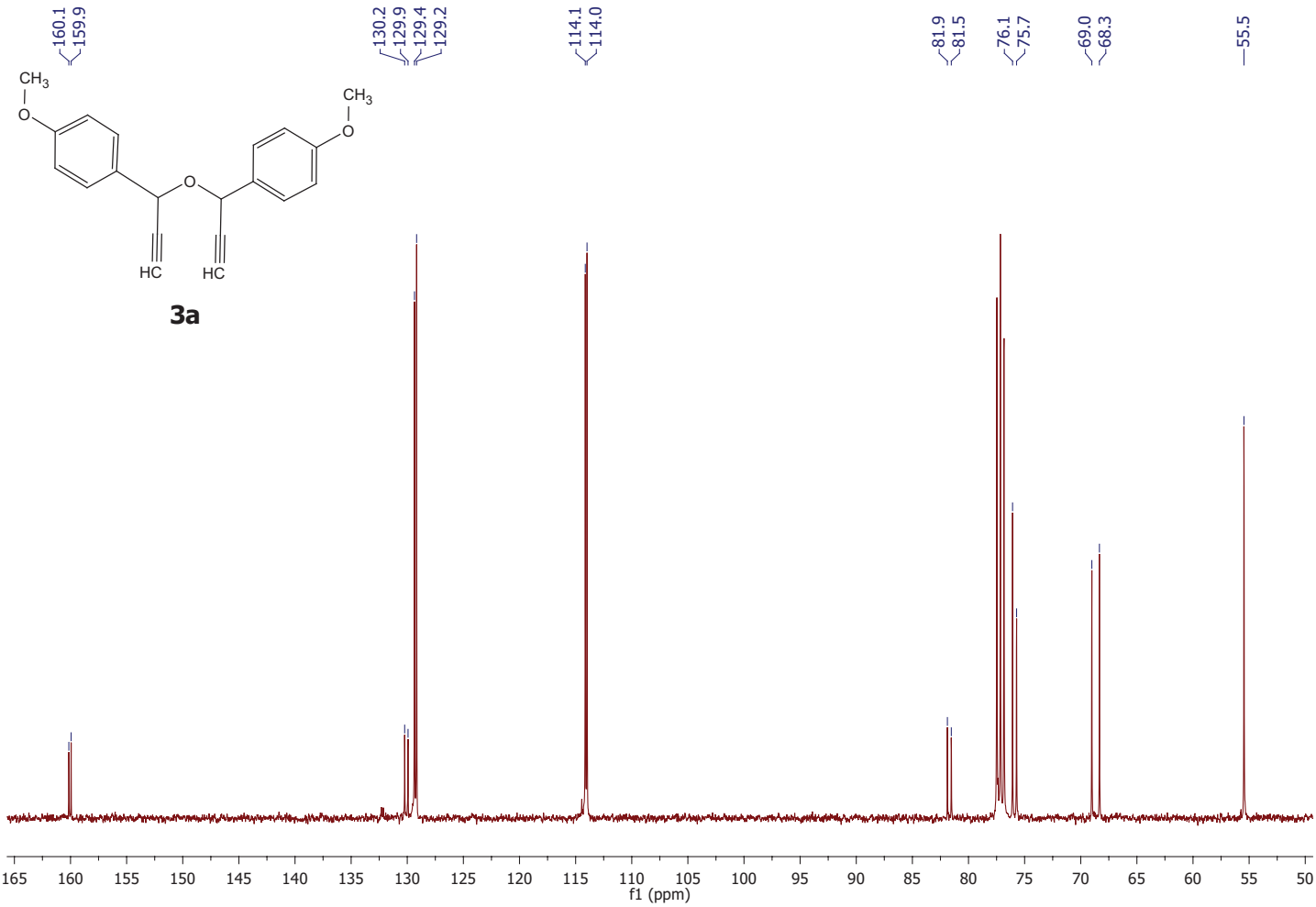
2q


7.46
7.33
4.20
3.49
1.81
1.58
1.00
O
CH3
H3C
2s
2.07
3.07
0.86
2.71
2.38
2.37
3.63
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)

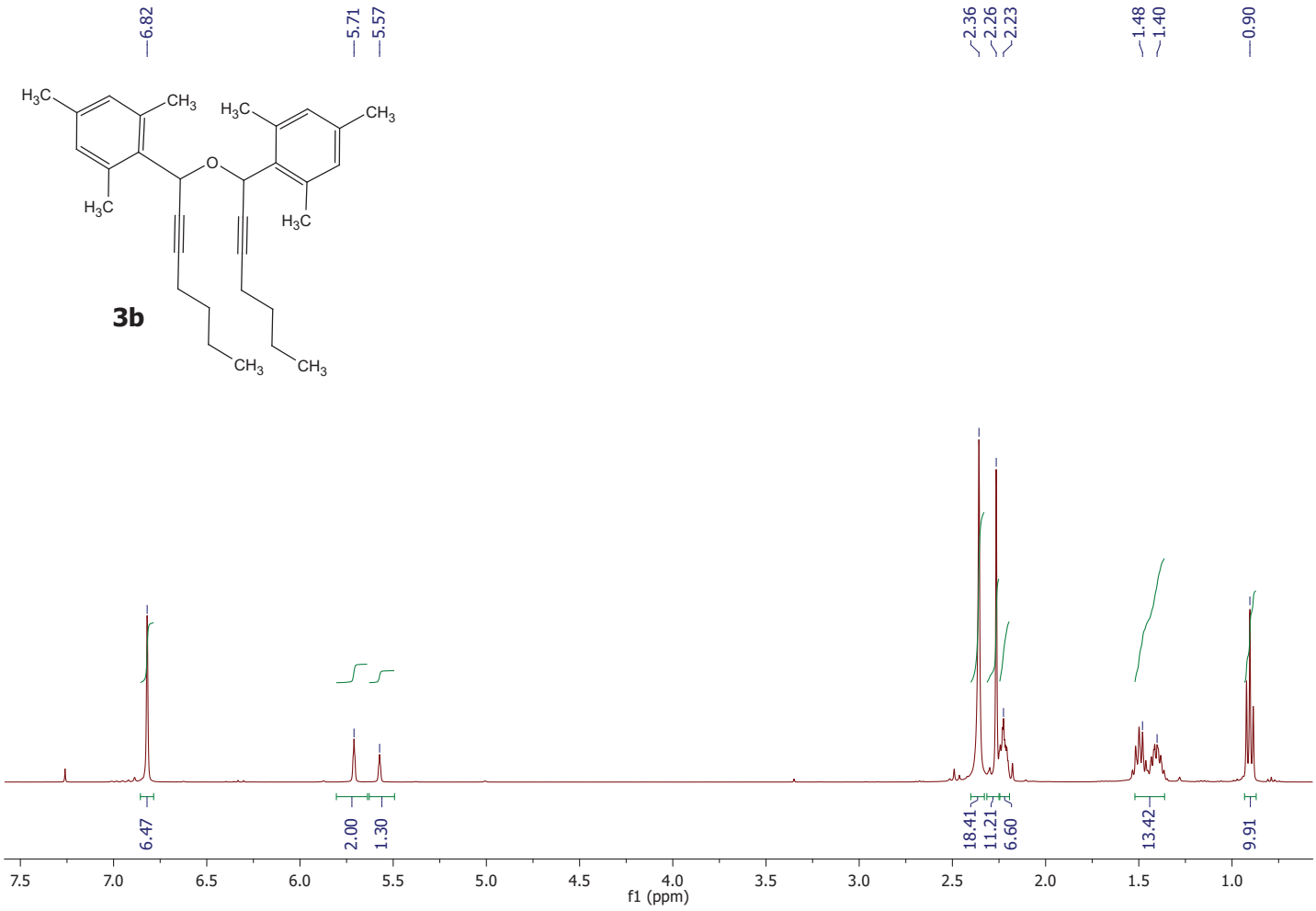

131.8
128.6
128.4
123.0
88.2
86.0
71.7
56.5
37.8
18.7
14.0
O
CH3
H3C
2s
140
135
130
125
120
115
110
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
f1 (ppm)

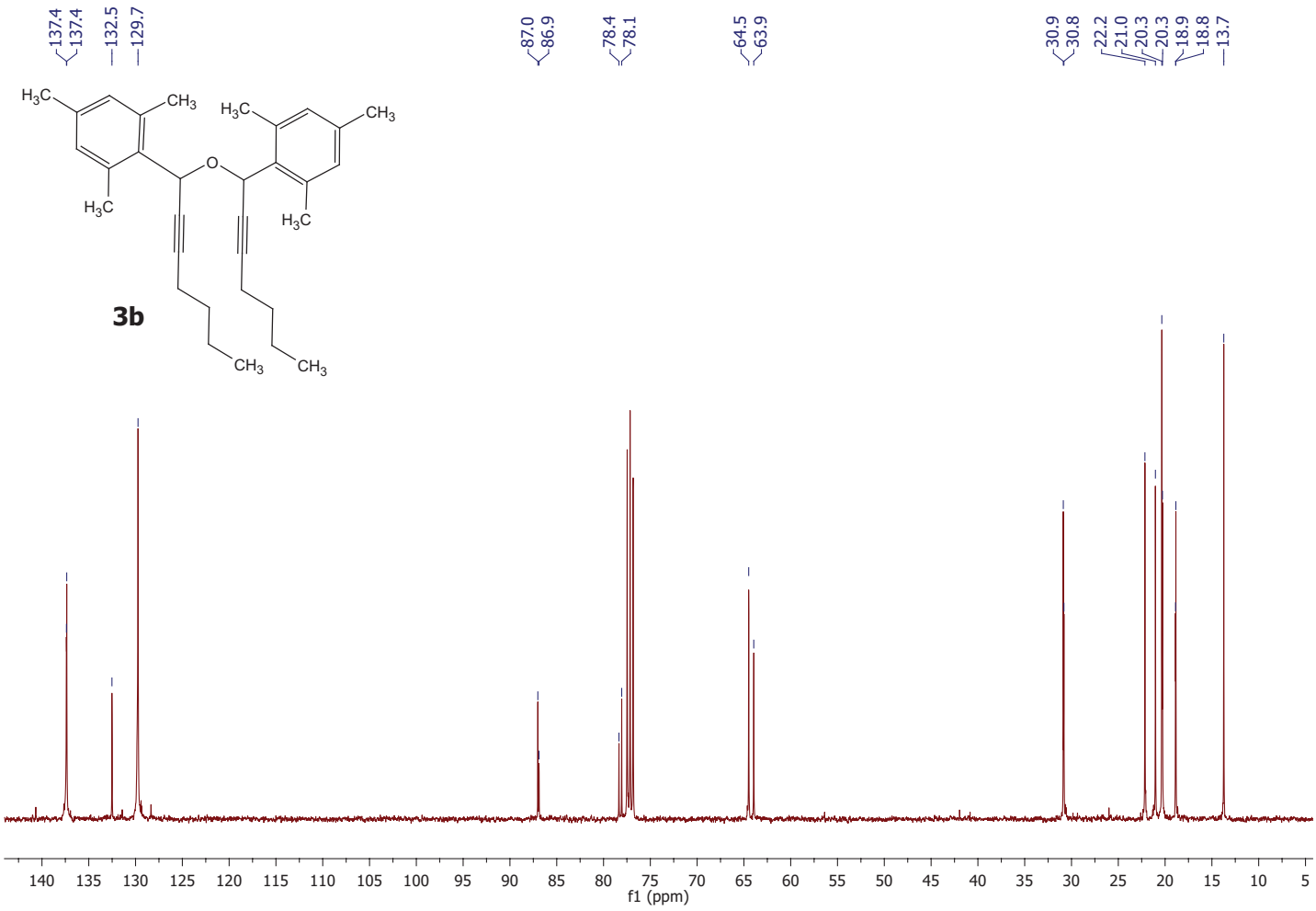

7.47
7.41
6.90
5.56
5.18
3.81
3.80
2.70
2.65
CH3
O
CH3
O
O
HC
HC
4.03
3.51
7.64
2.00
1.77
5.34
6.14
1.84
1.58
7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2
f1 (ppm)

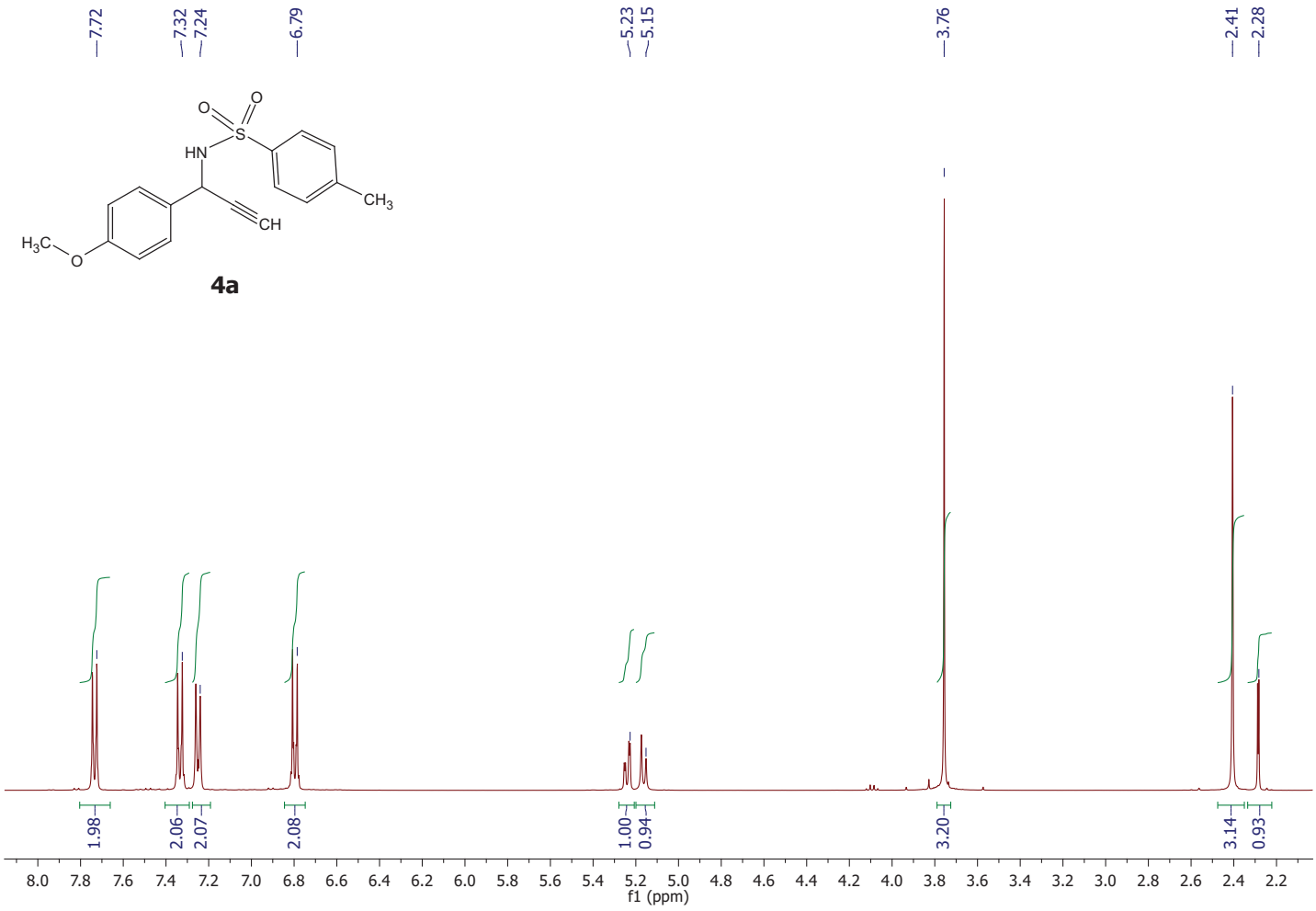
3a


160.1
159.9
130.2
129.9
129.4
129.2
114.1
114.0
81.9
81.5
76.1
75.7
69.0
68.3
55.5
CH3
O
CH3
O
O
HC
HC
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50
f1 (ppm)

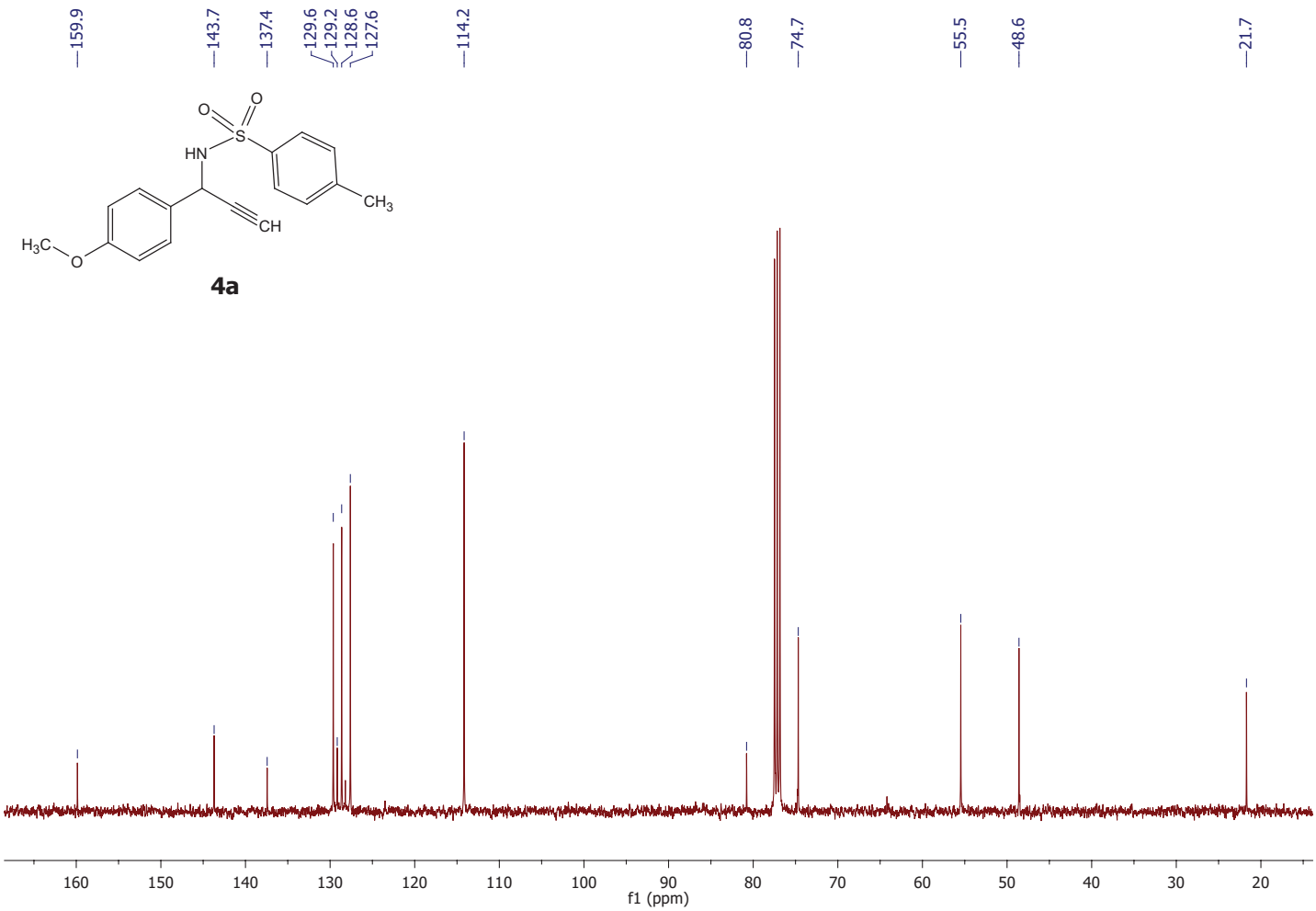
3a


6.82
5.71
5.57
2.36
2.26
2.23
1.48
1.40
0.90
3b
6.47
2.00
1.30
18.41
11.21
6.60
13.42
9.91
f1 (ppm)

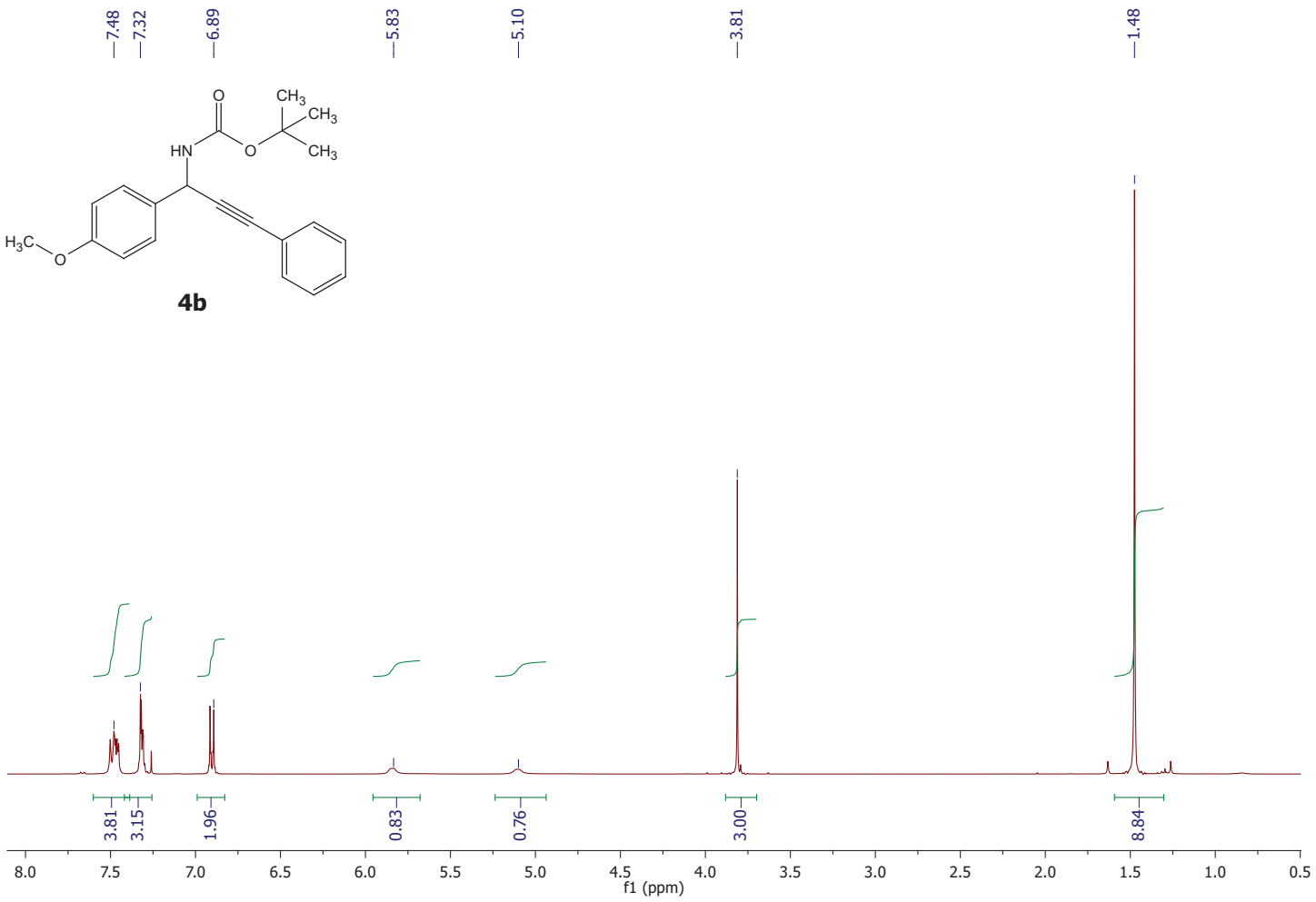

137.4
137.4
132.5
129.7
87.0
86.9
78.4
78.1
64.5
63.9
30.9
30.8
22.2
21.0
20.3
20.3
18.9
18.8
13.7
3b
f1 (ppm)

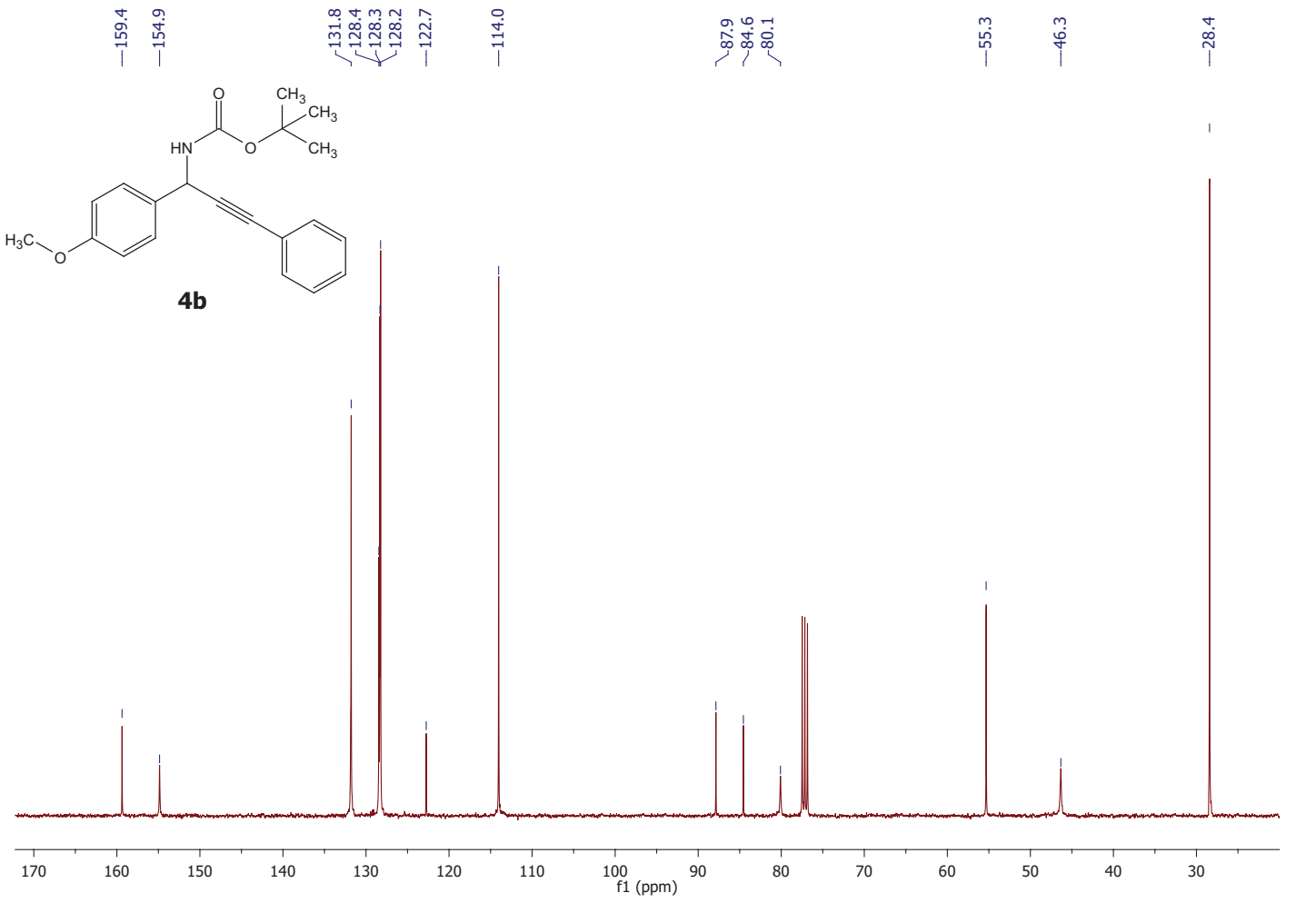

7.72
7.32
7.24
6.79
5.23
5.15
3.76
2.41
2.28
O
O
S
HN
CH3
CH
H3C
O
1.98
2.06
2.07
2.08
1.00
0.94
3.20
3.14
0.93
8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2
f1 (ppm)

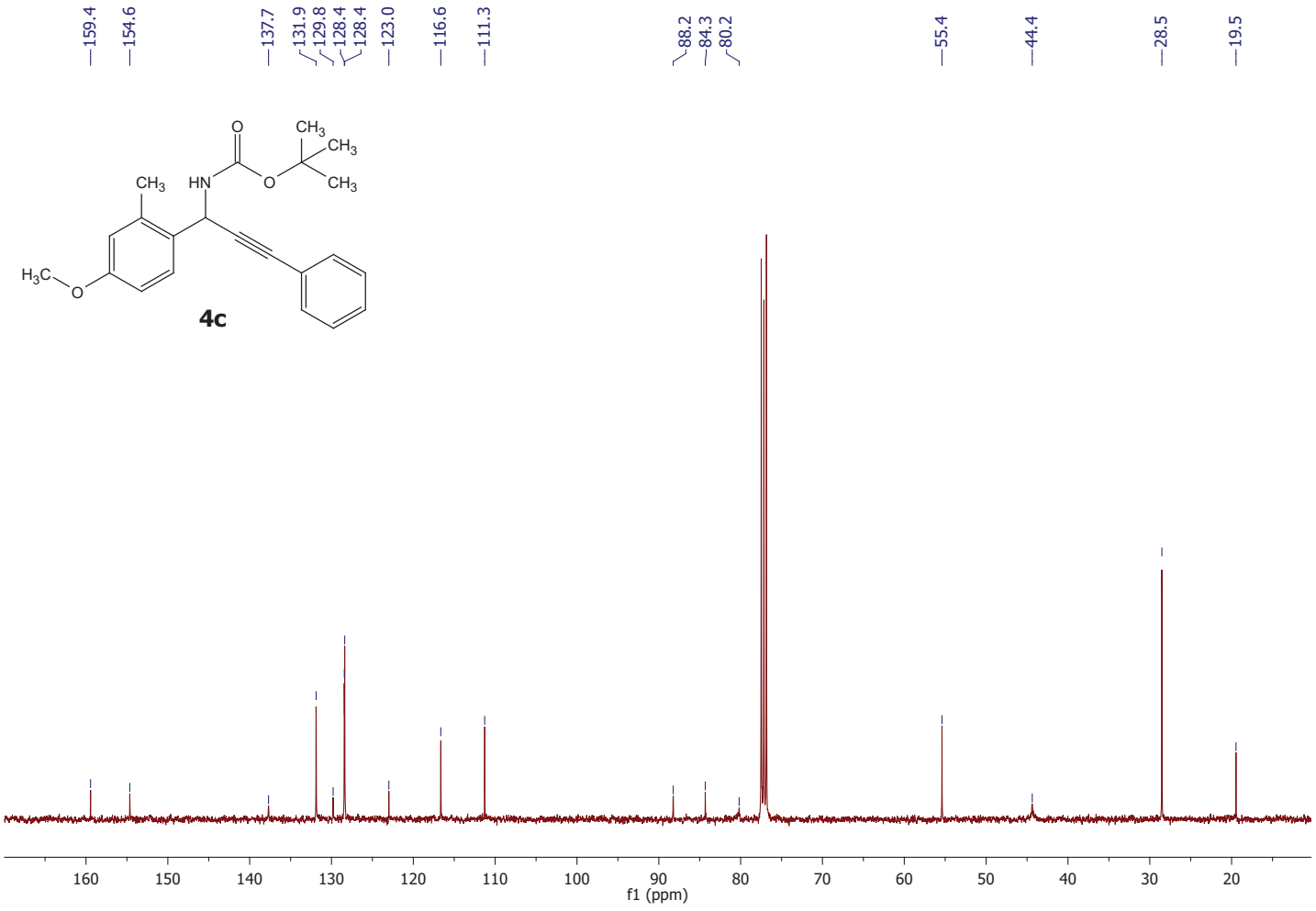
4a


159.9
143.7
137.4
129.6
129.2
128.6
127.6
114.2
80.8
74.7
55.5
48.6
21.7
O
O
S
HN
CH3
CH
H3C
O
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)

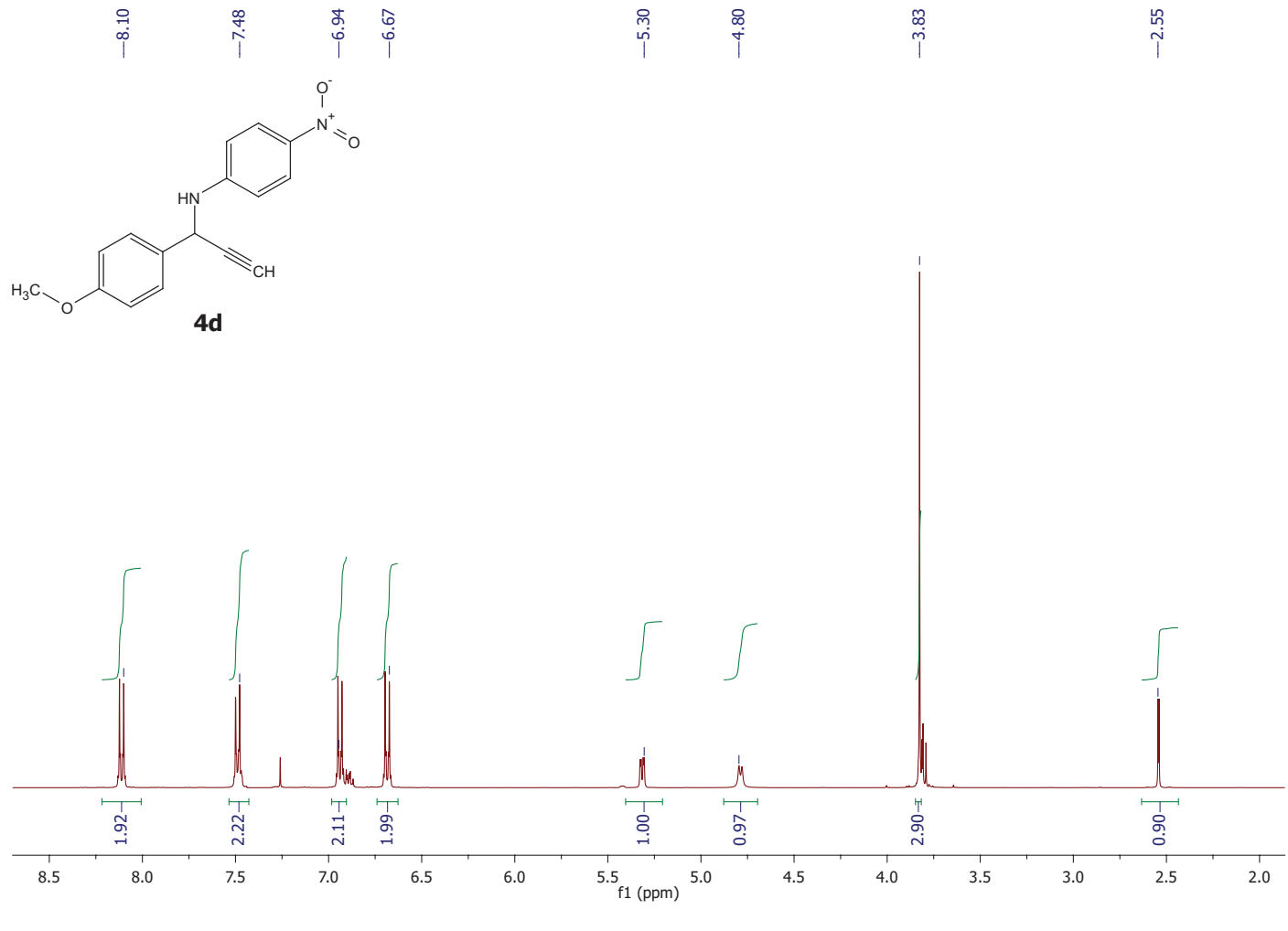
4a


7.48
7.32
6.89
5.83
5.10
3.81
1.48
O
CH3
CH3
HN
O
CH3
H3C
O
4b
3.81
3.15
1.96
0.83
0.76
3.00
8.84
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

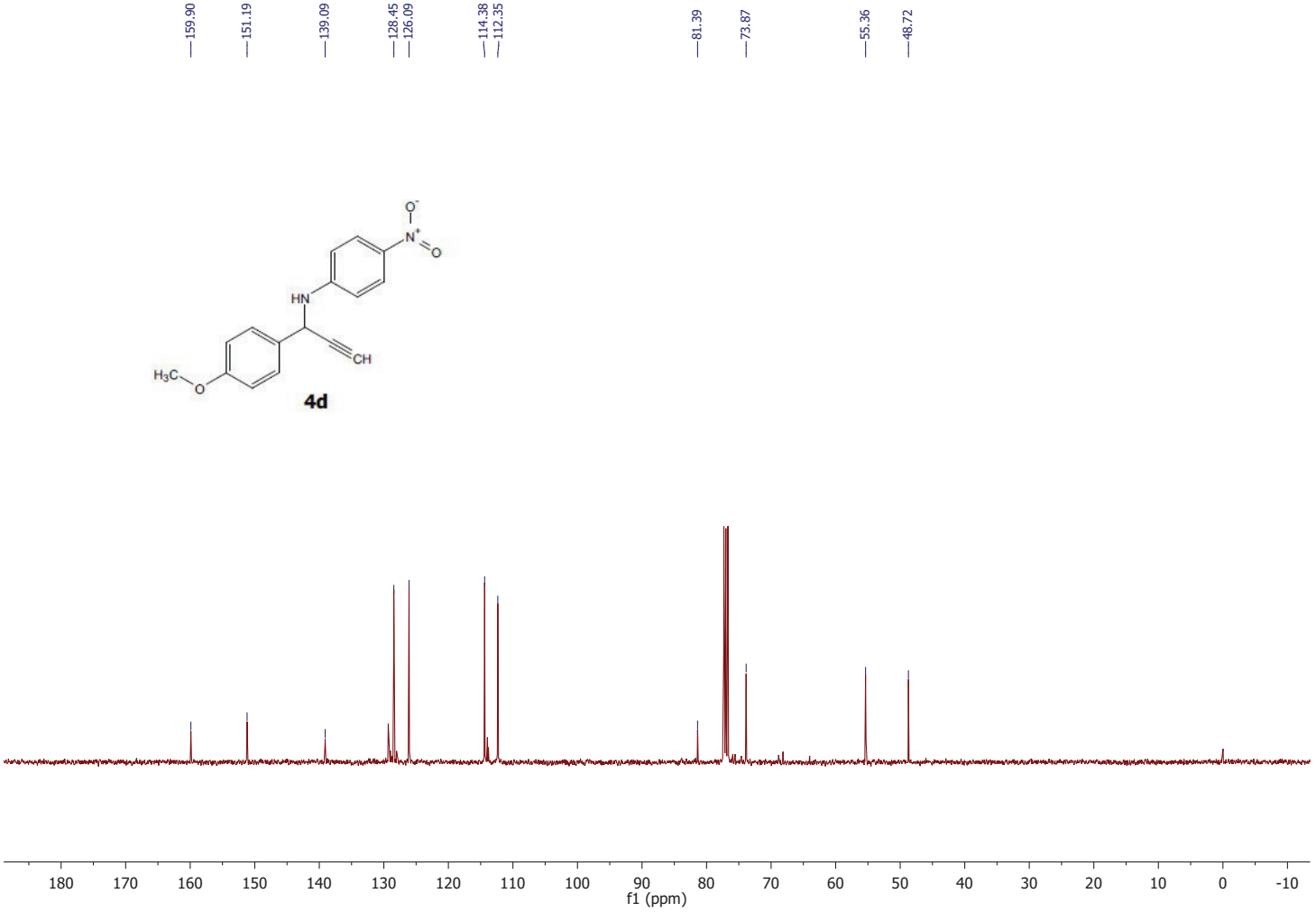

159.4
154.9
131.8
128.4
128.3
128.2
122.7
114.0
87.9
84.6
80.1
55.3
46.3
28.4
O
CH3
CH3
HN
O
CH3
H3C
O
4b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
f1 (ppm)

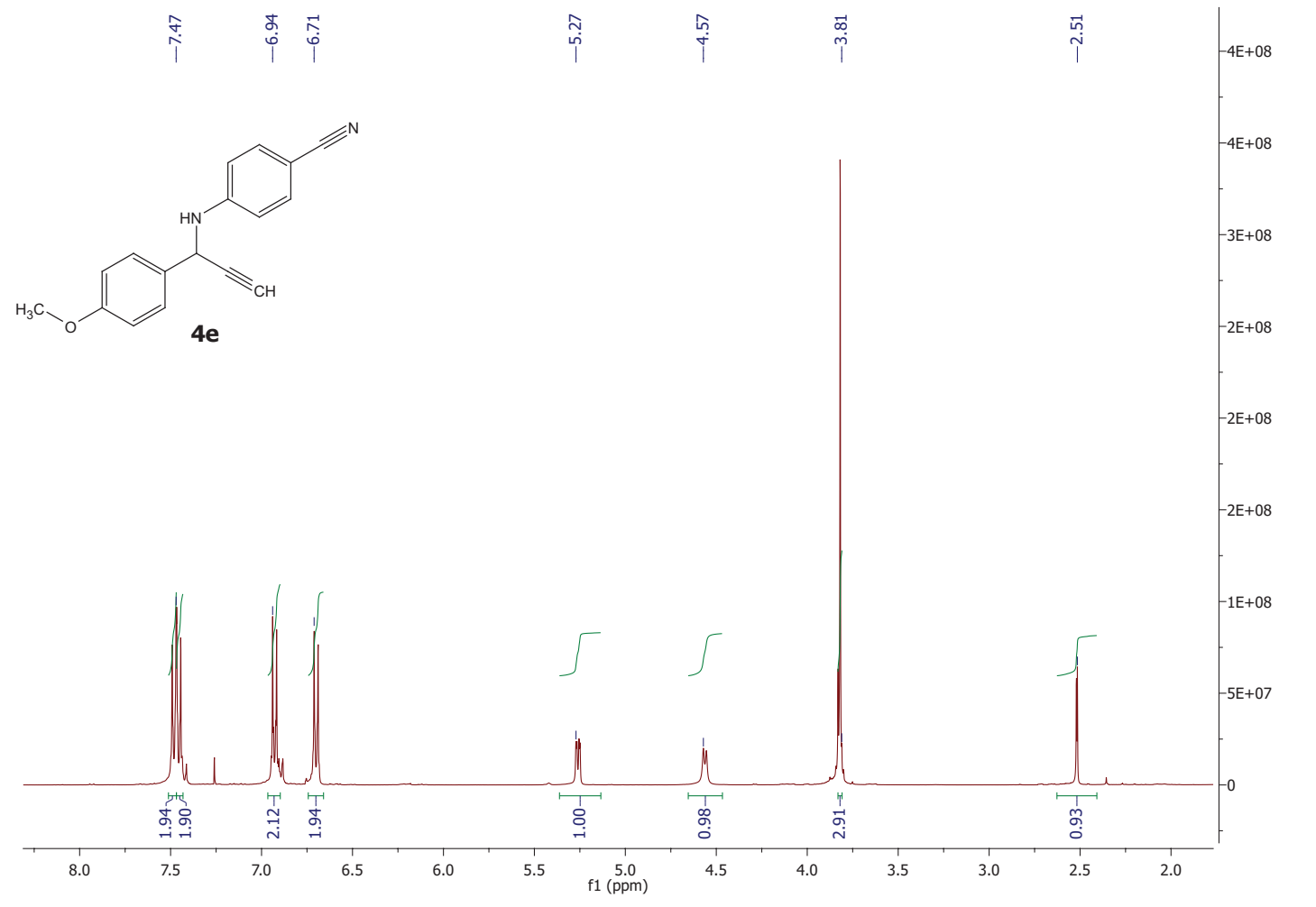

159.4
154.6
137.7
131.9
129.8
128.4
128.4
123.0
116.6
111.3
88.2
84.3
80.2
55.4
44.4
28.5
19.5
CH3
CH3
CH3
HN
O
O
CH3
H3C
O
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

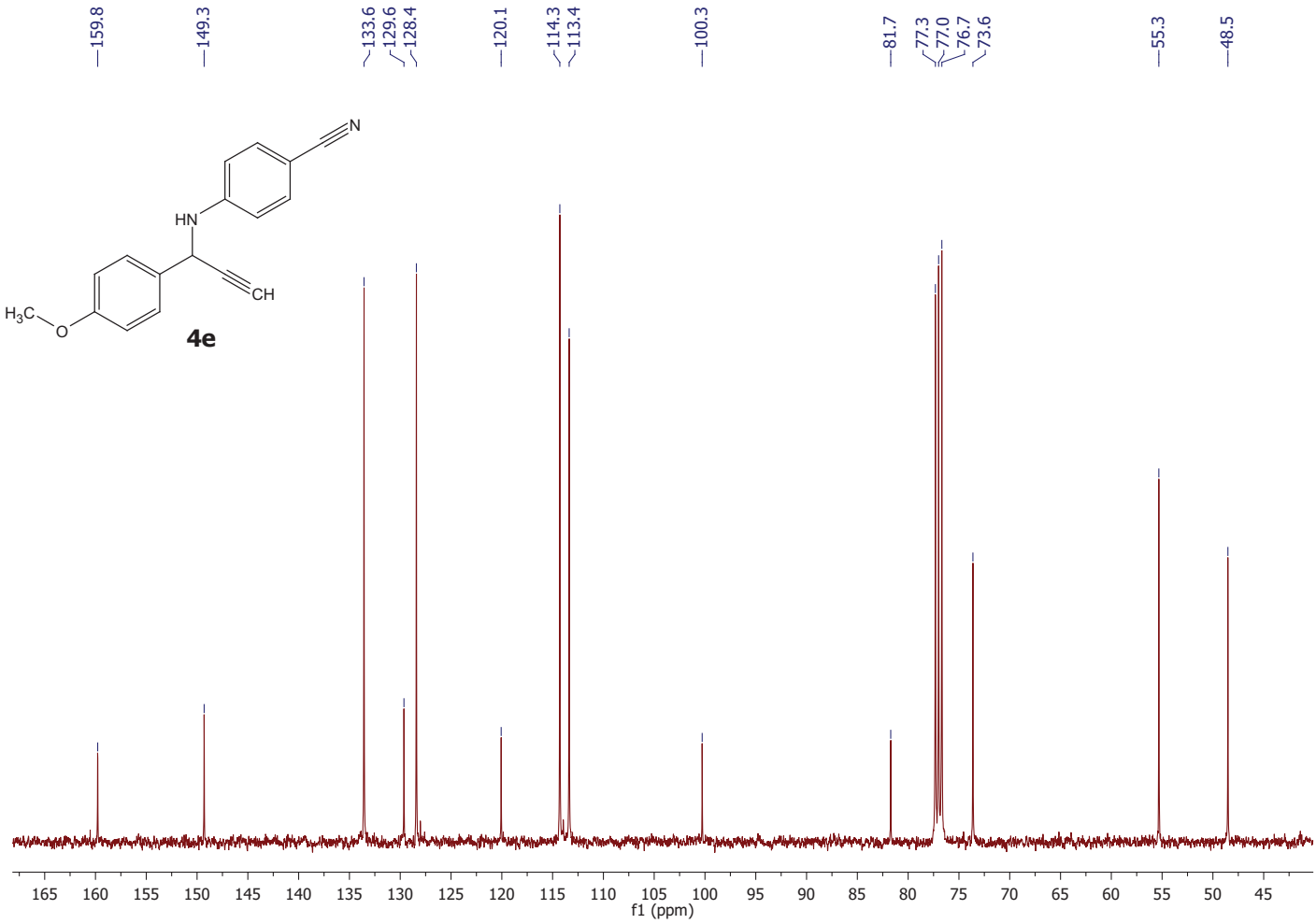
4c

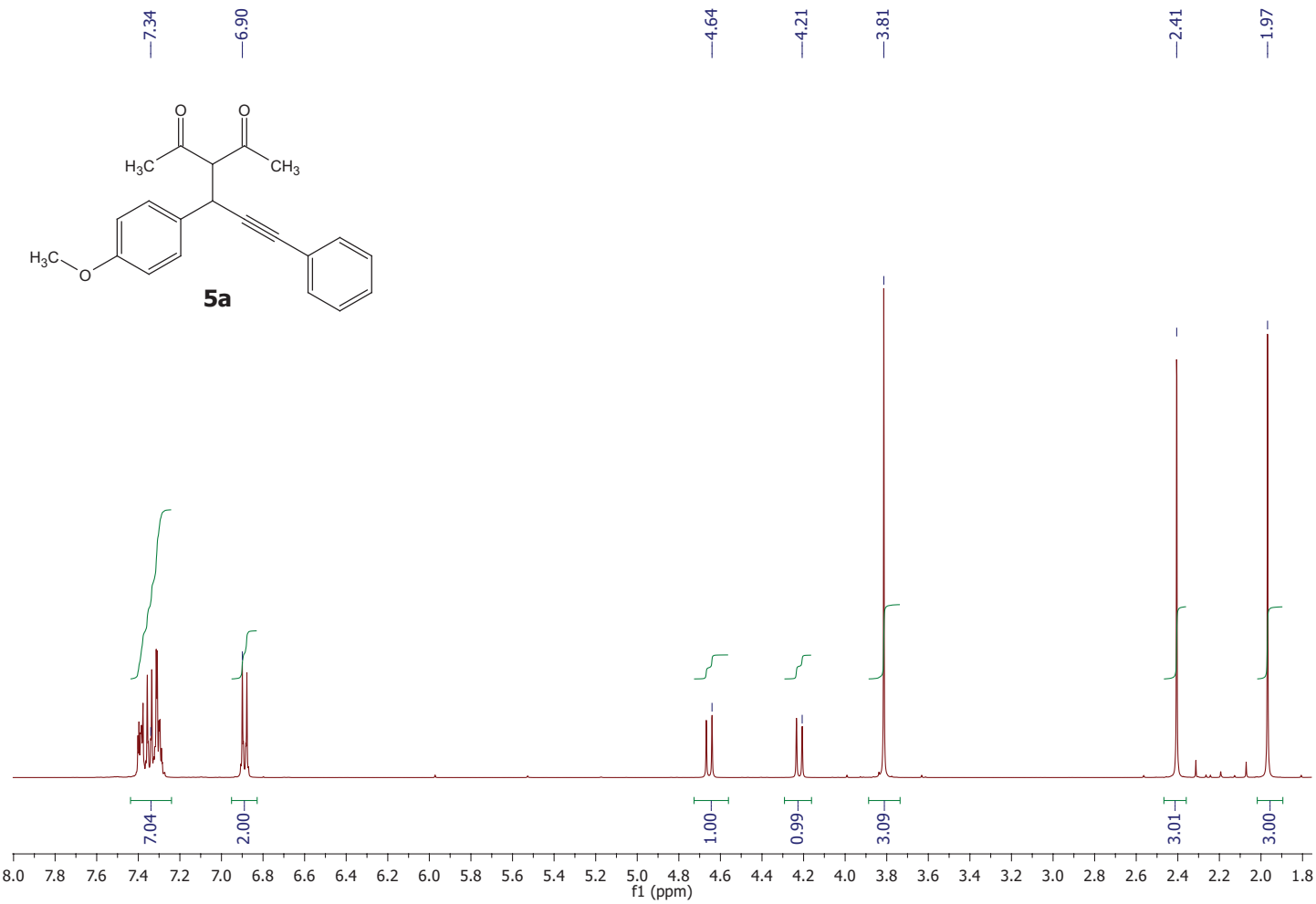

8.10
7.48
6.94
6.67
5.30
4.80
3.83
2.55
O-
N+
O
HN
CH
H3C
O
1.92
2.22
2.11
1.99
1.00
0.97
2.90
0.90
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
f1 (ppm)

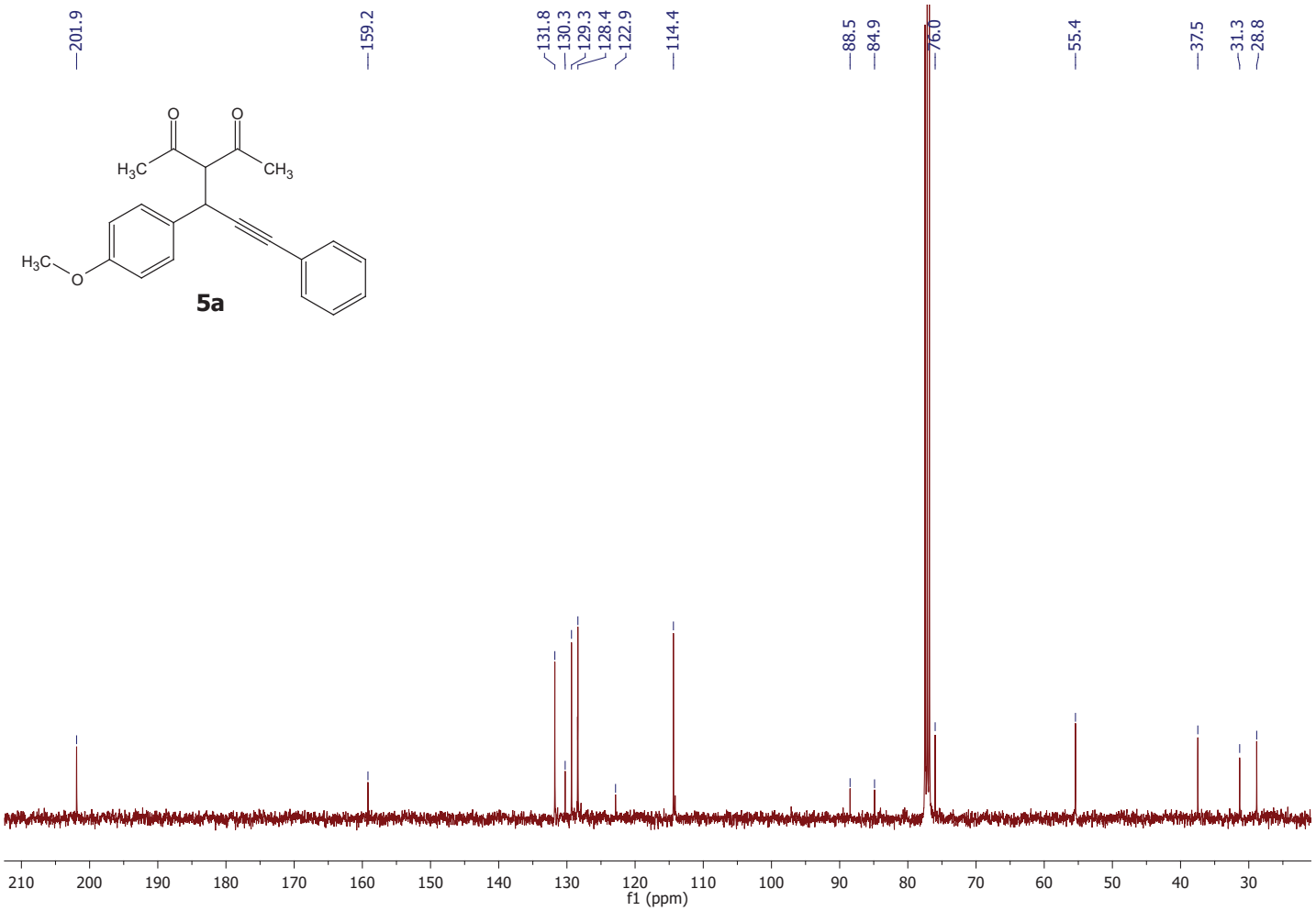
4d

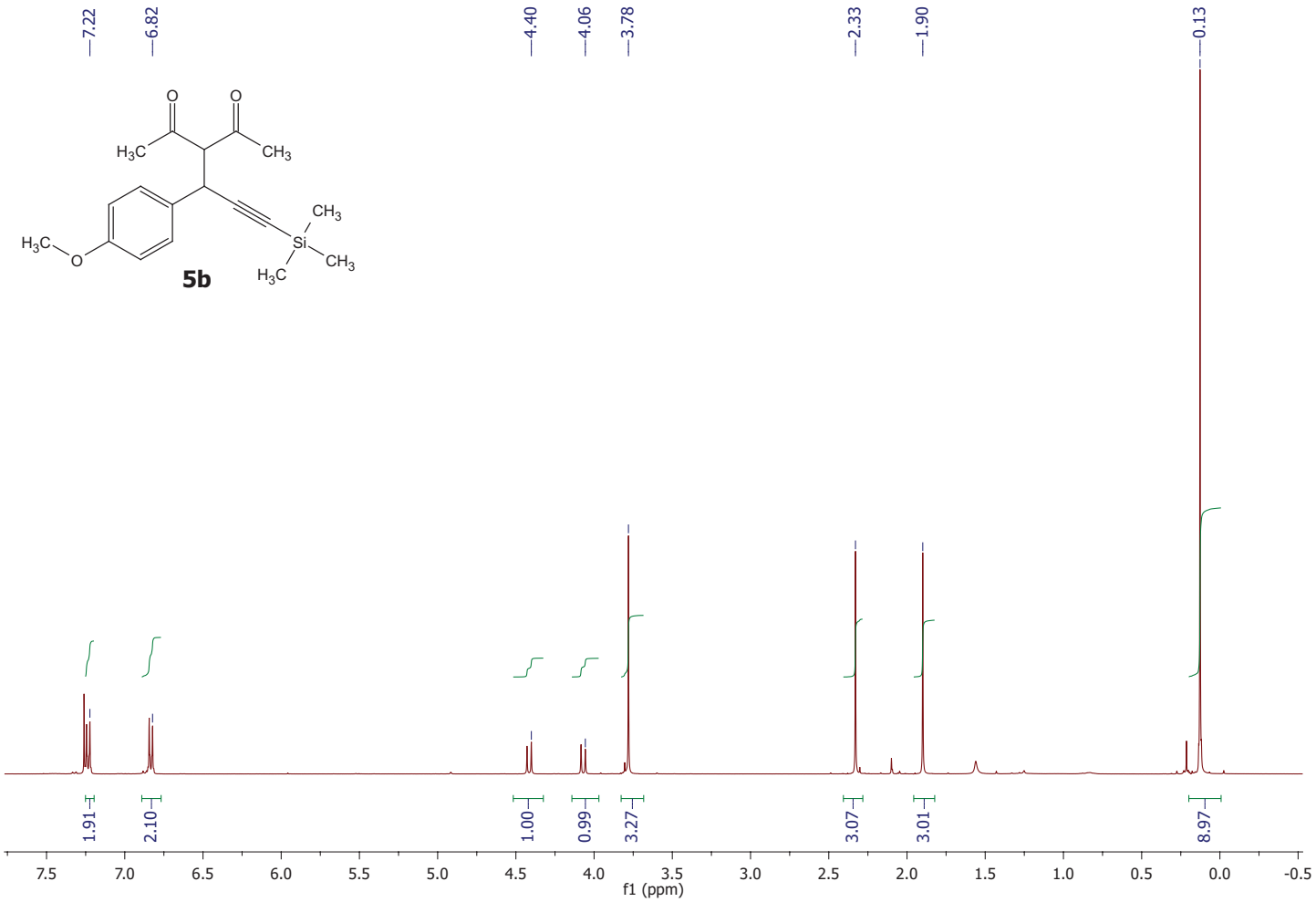

159.90
151.19
139.09
128.45
126.09
114.38
112.35
81.39
73.87
55.36
48.72
HN
CH
H_3C
O
4d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

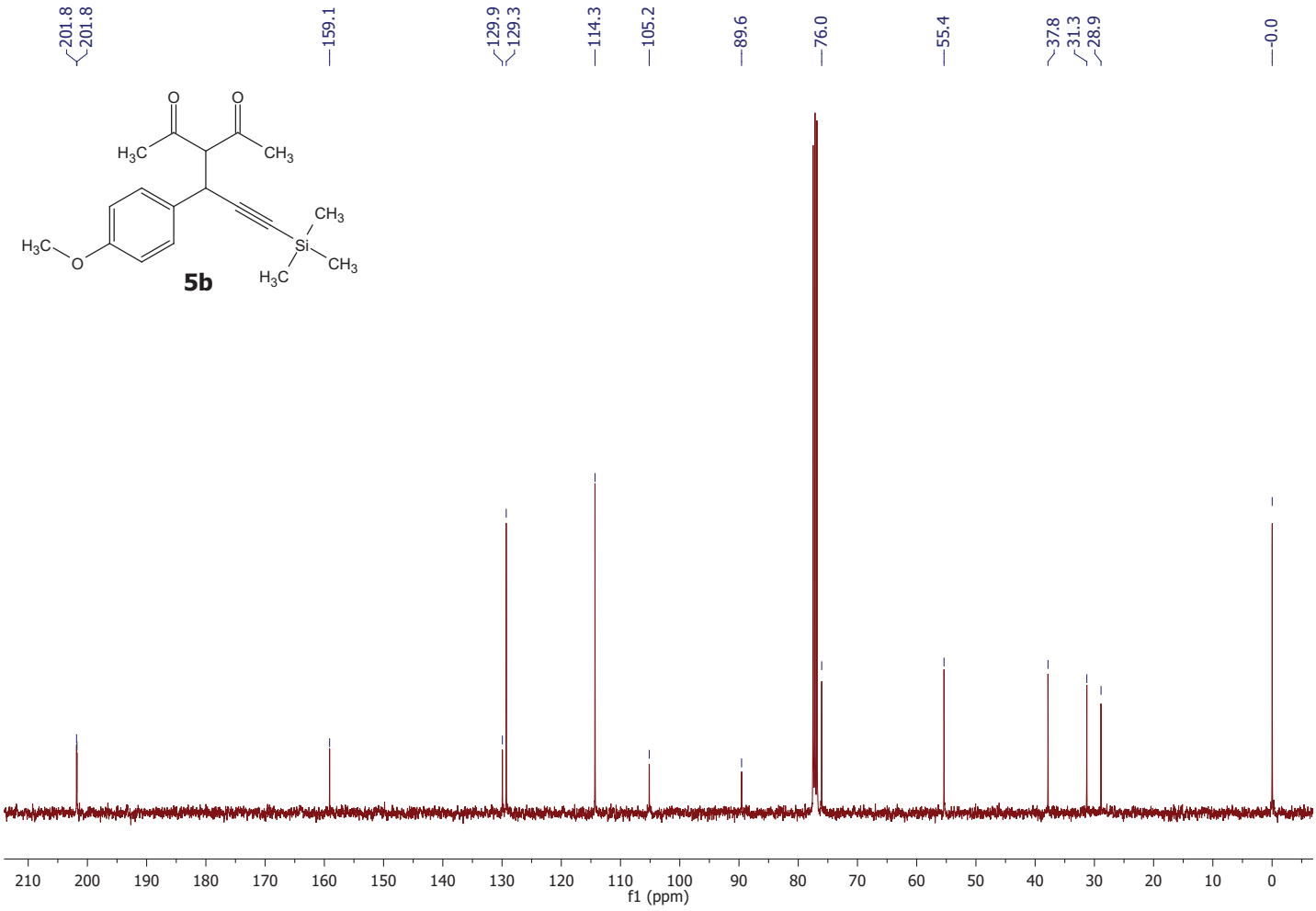

7.47
6.94
6.71
5.27
4.57
3.81
2.51
N
HN
CH
H_3C
O
4e
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
1.94
1.90
2.12
1.94
1.00
0.98
2.91
0.93
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
f1 (ppm)

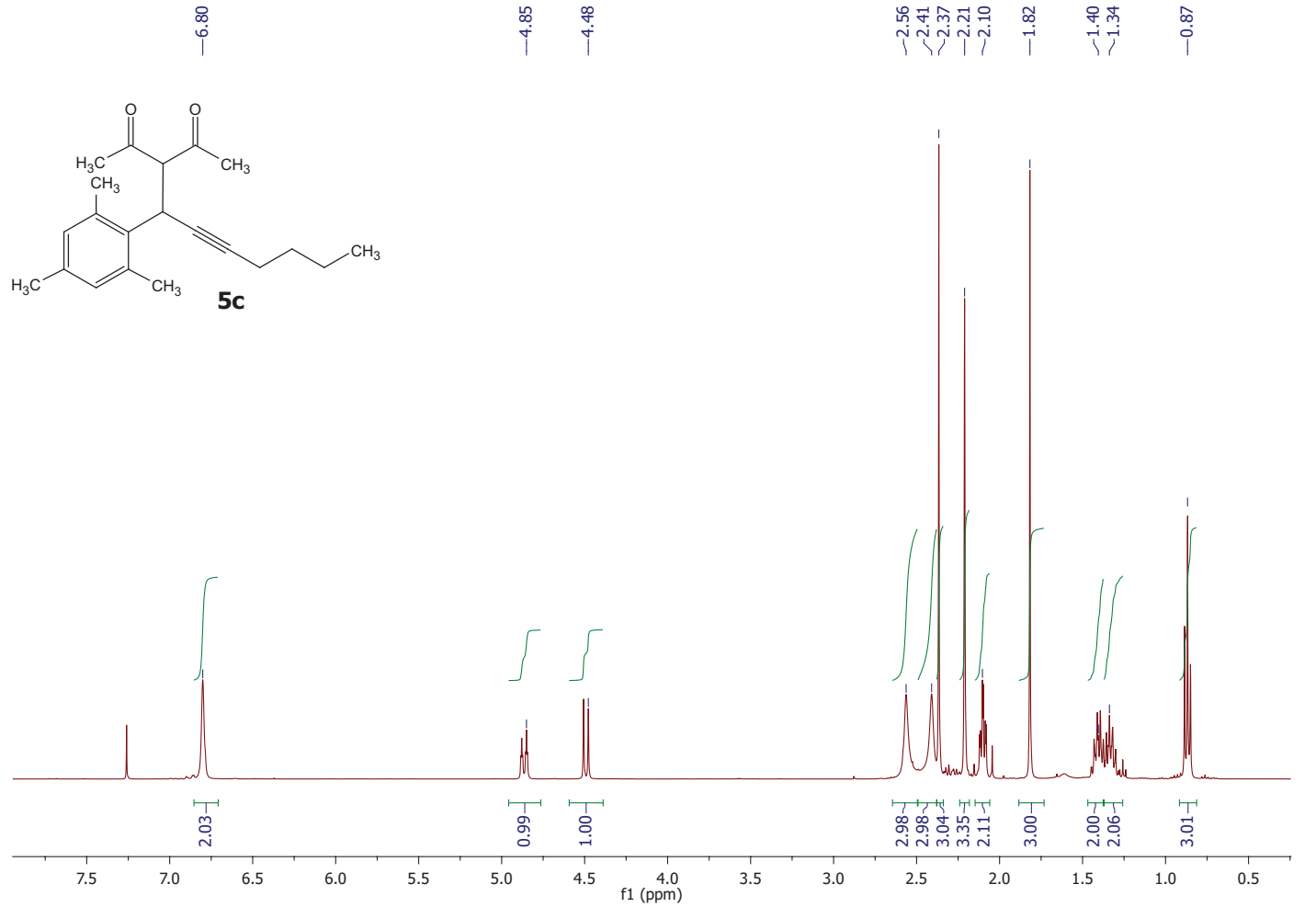

159.8
149.3
133.6
129.6
128.4
120.1
114.3
113.4
100.3
81.7
77.3
77.0
76.7
73.6
55.3
48.5
N
HN
CH
H3C
O
4e
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45
f1 (ppm)

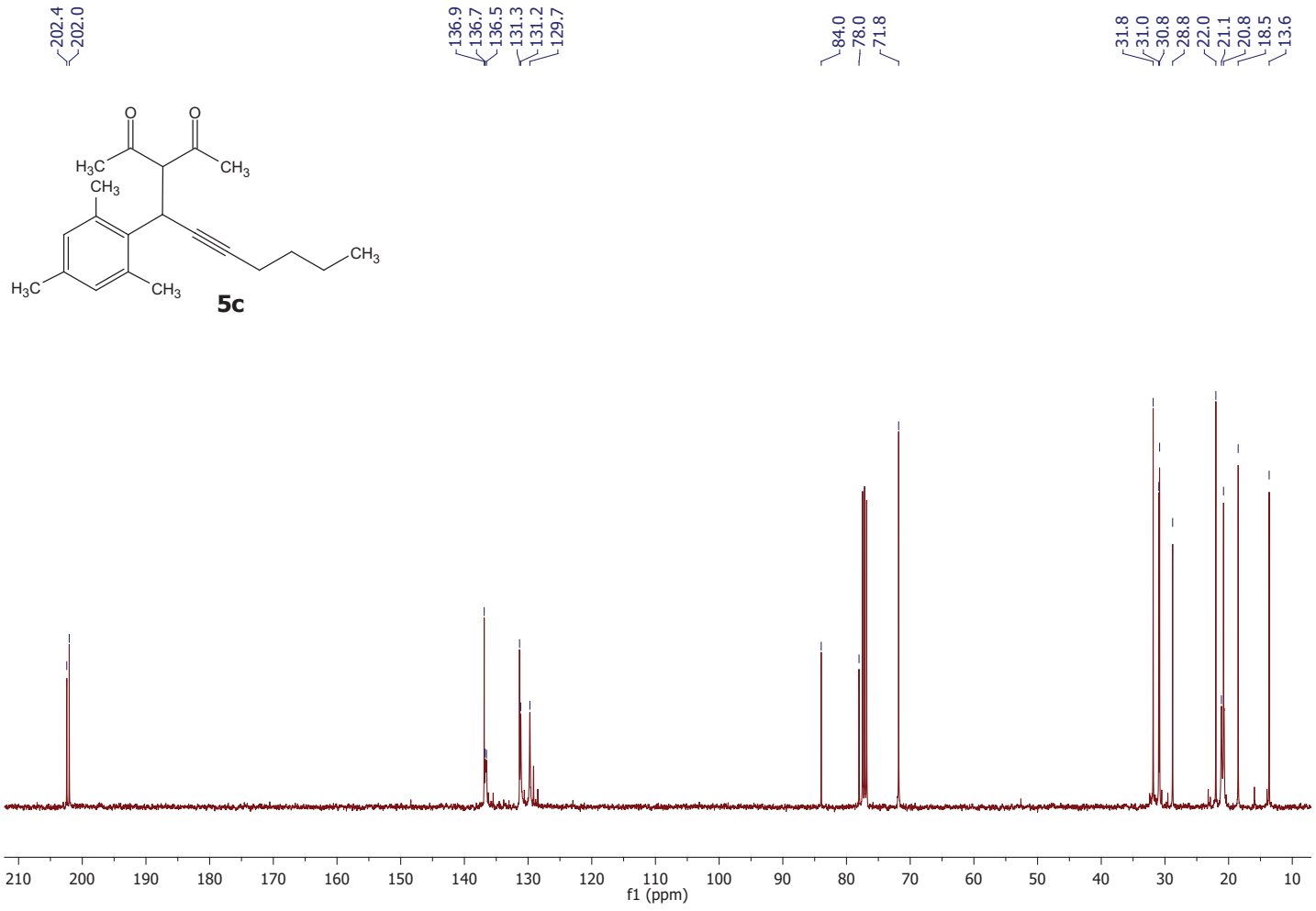

7.34
6.90
4.64
4.21
3.81
2.41
1.97
O
O
H3C
CH3
H3C
O
5a
7.04
2.00
1.00
0.99
3.09
3.01
3.00
8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8
f1 (ppm)

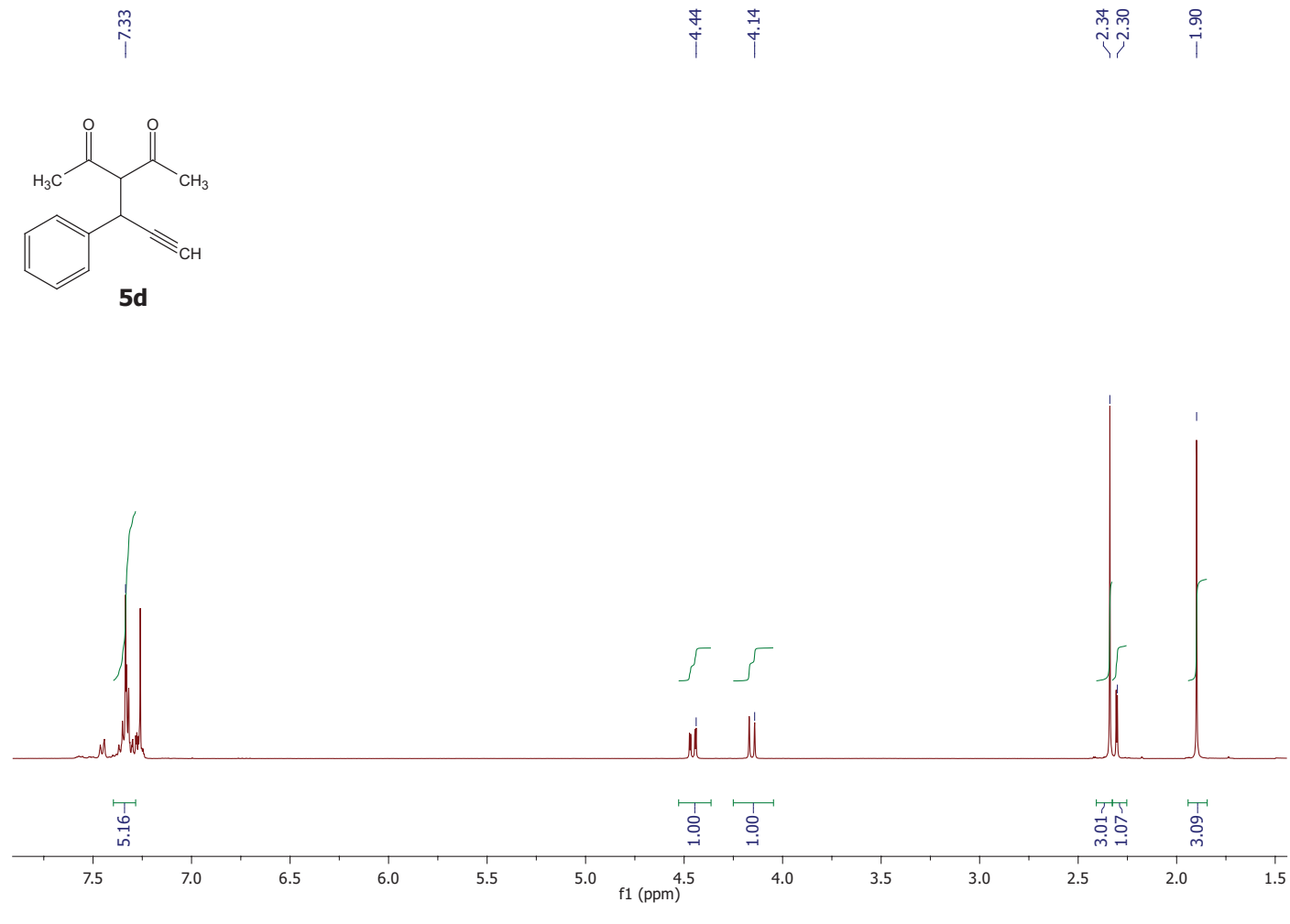

201.9
159.2
131.8
130.3
129.3
128.4
122.9
114.4
88.5
84.9
76.0
55.4
37.5
31.3
28.8
O
O
H3C
CH3
H3C
O
5a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
f1 (ppm)

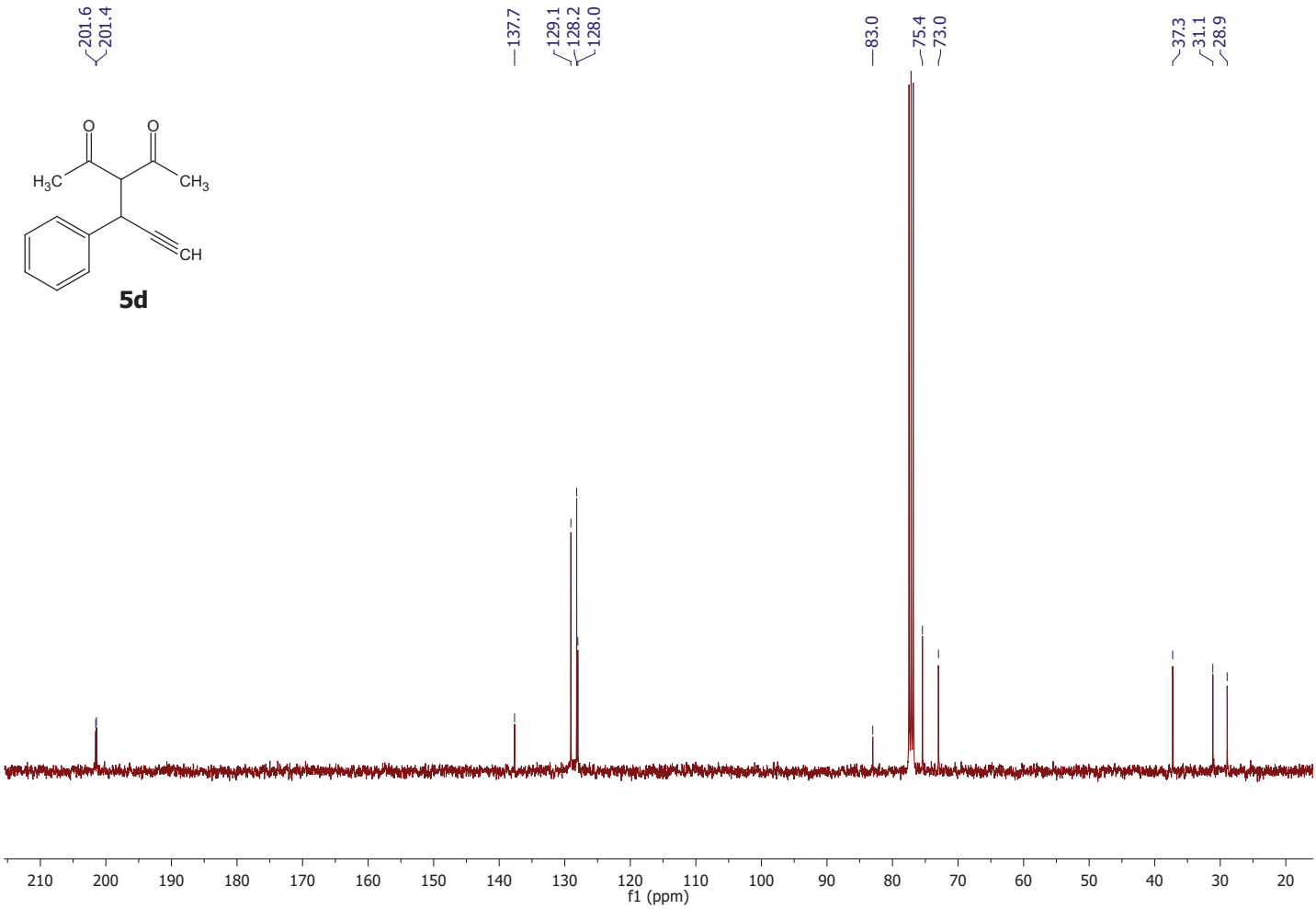

7.22
6.82
4.40
4.06
3.78
2.33
1.90
0.13
O
O
H3C
CH3
H3C
O
CH3
Si
CH3
H3C
5b
1.91
2.10
1.00
0.99
3.27
3.07
3.01
8.97
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


201.8
201.8
159.1
129.9
129.3
114.3
105.2
89.6
76.0
55.4
37.8
31.3
28.9
0.0
O
O
H3C
CH3
CH3
H3C
O
Si
CH3
H3C
5b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

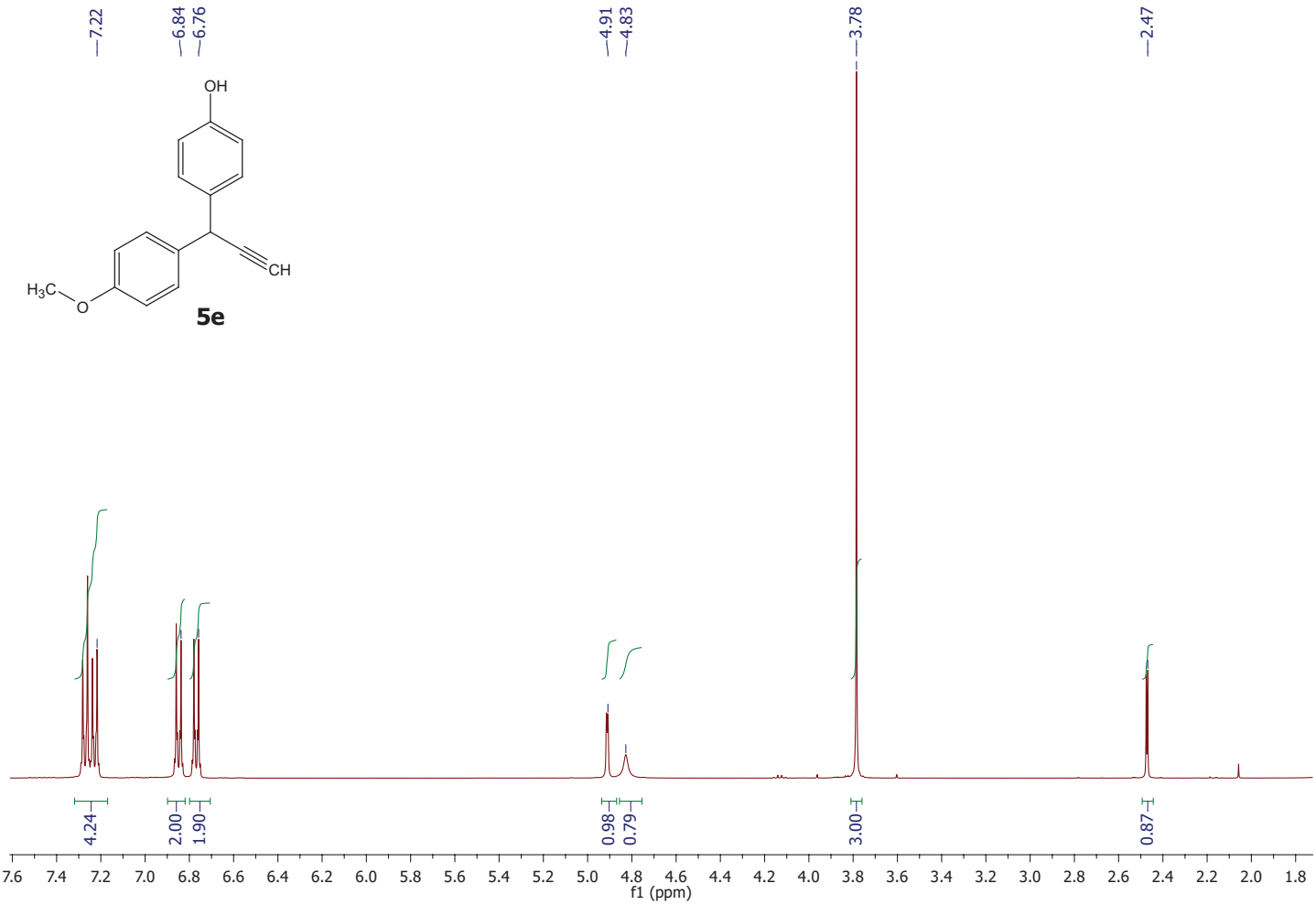

6.80
4.85
4.48
2.56
2.41
2.37
2.21
2.10
1.82
1.40
1.34
0.87
O
O
H3C
CH3
CH3
CH3
H3C
CH3
5c
2.03
0.99
1.00
2.98
2.98
3.04
3.35
2.11
3.00
2.00
2.06
3.01
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


202.4
202.0
136.9
136.7
136.5
131.3
131.2
129.7
84.0
78.0
71.8
31.8
31.0
30.8
28.8
22.0
21.1
20.8
18.5
13.6
O
O
H3C
CH3
CH3
CH3
H3C
CH3
5c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

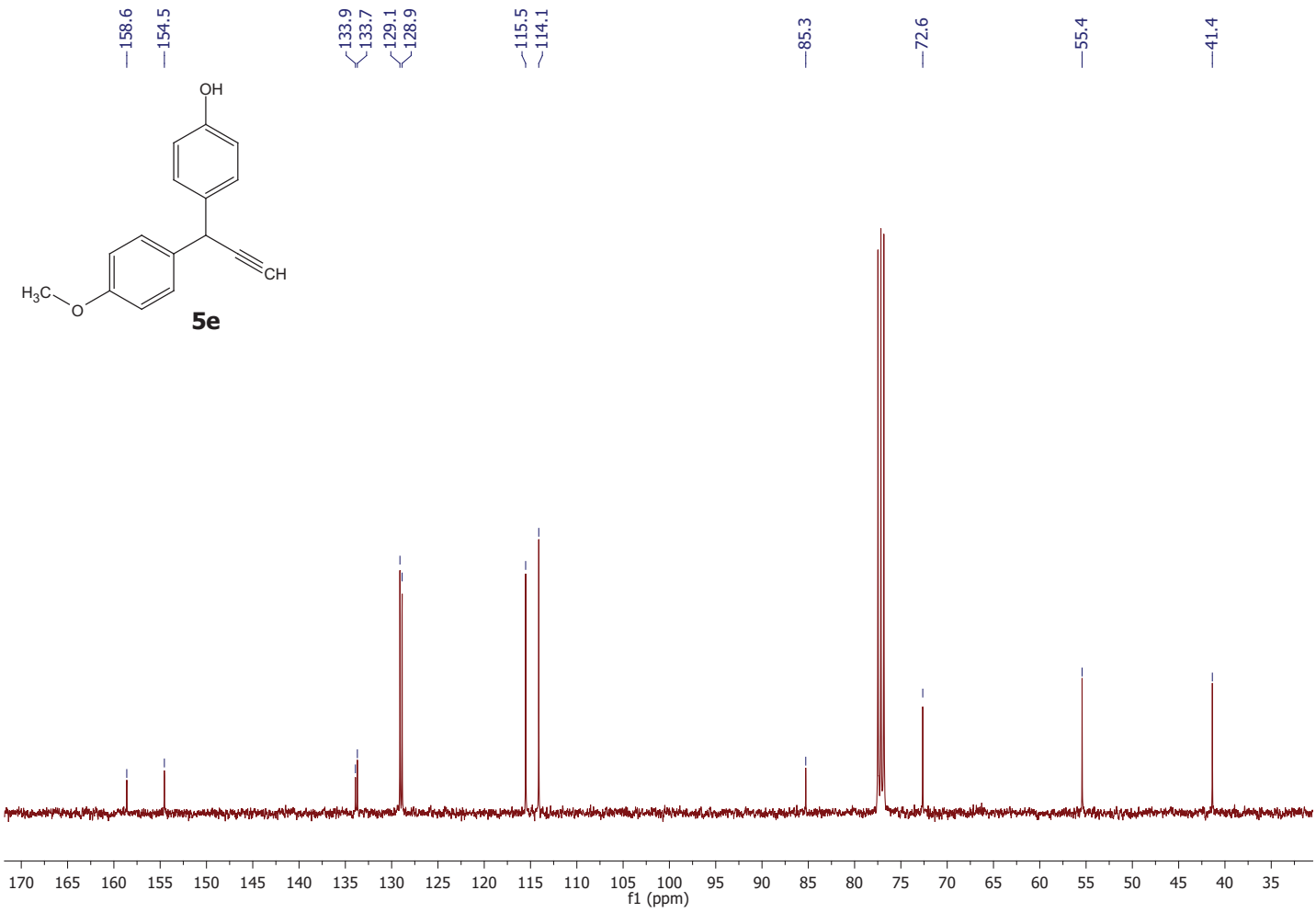

7.33
4.44
4.14
2.34
2.30
1.90
O
O
H3C
CH3
CH
5d
5.16
1.00
1.00
3.01
1.07
3.09
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5
f1 (ppm)

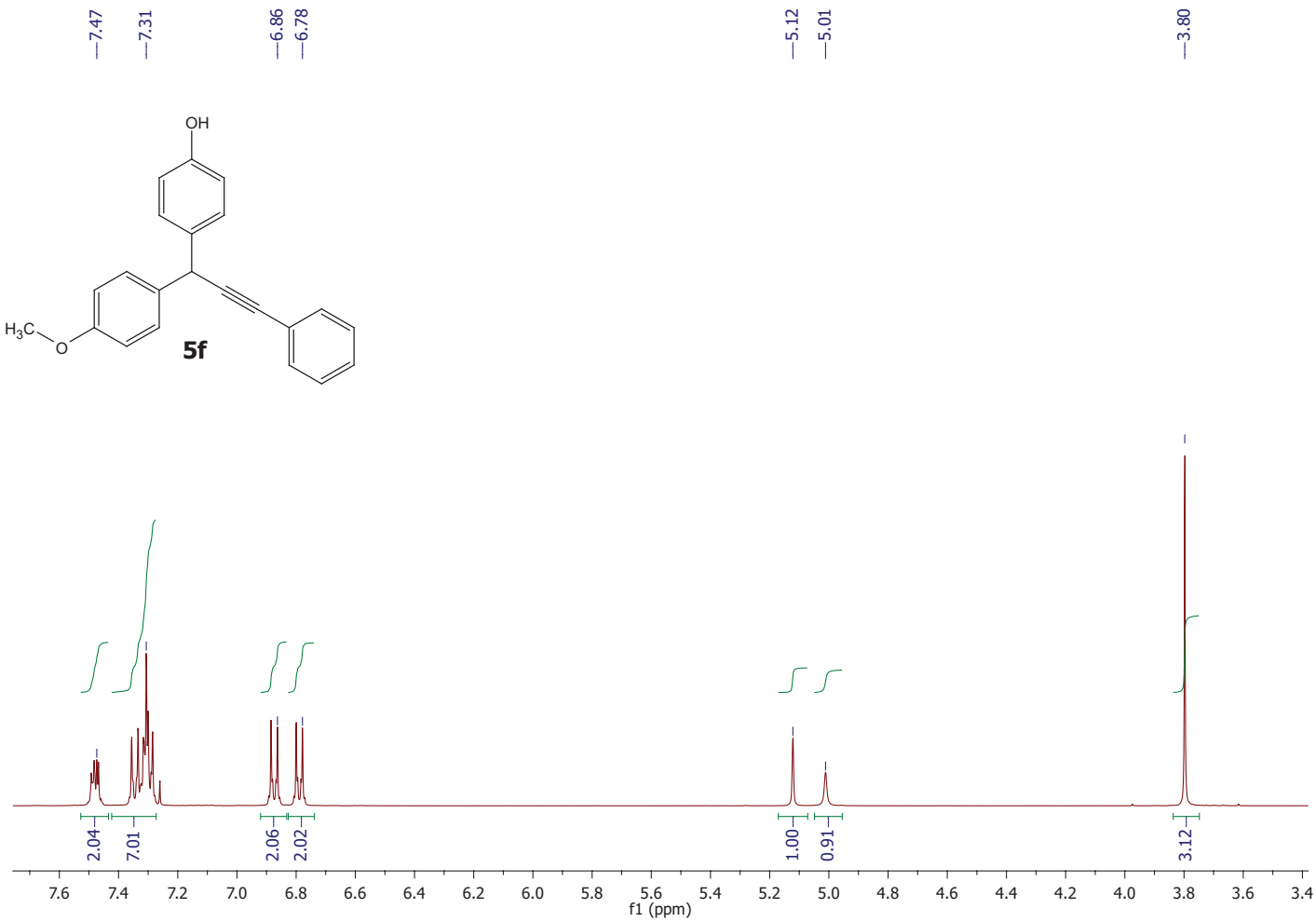

201.6
201.4
137.7
129.1
128.2
128.0
83.0
75.4
73.0
37.3
31.1
28.9
O
O
H3C
CH3
CH
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)

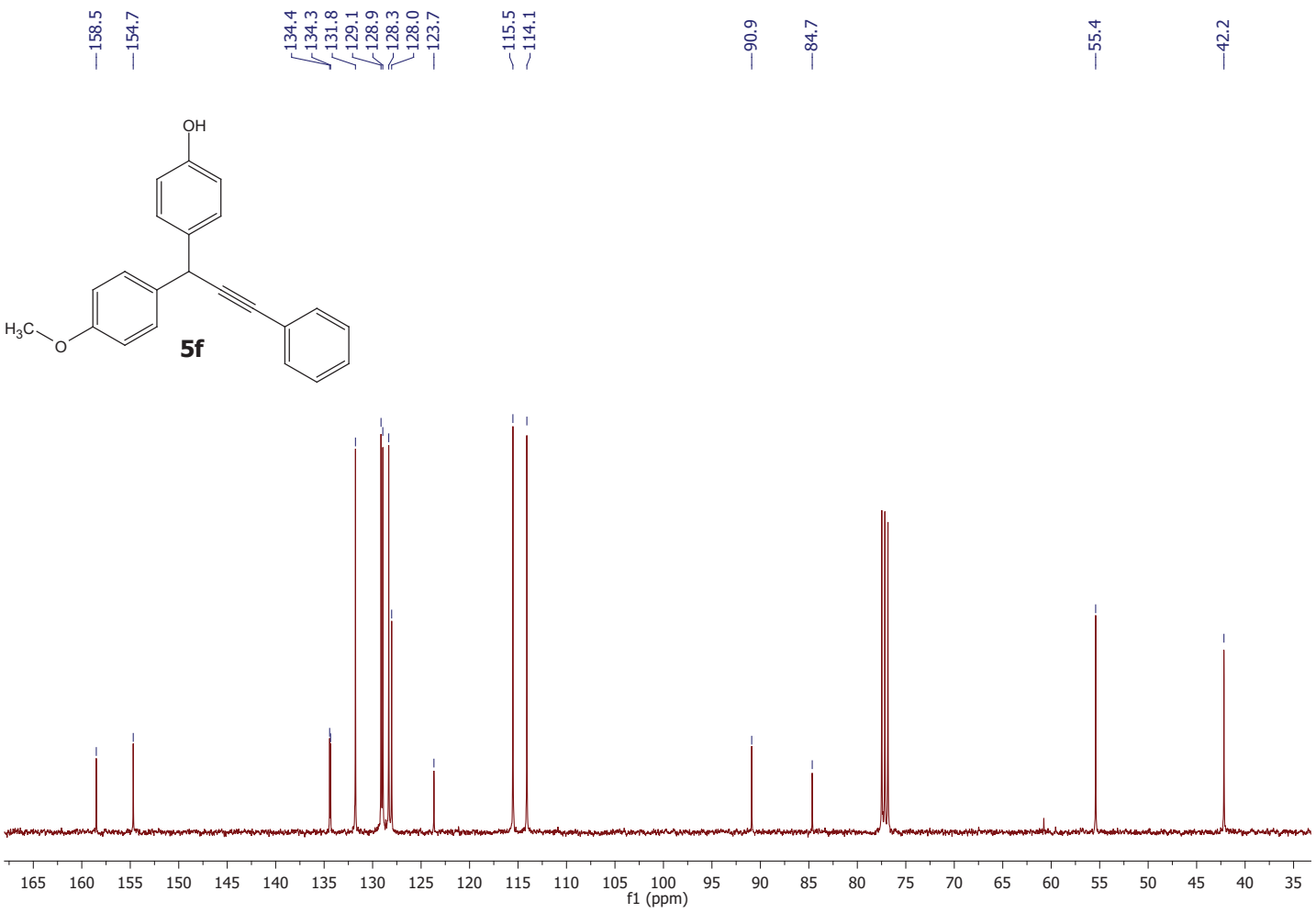
5d

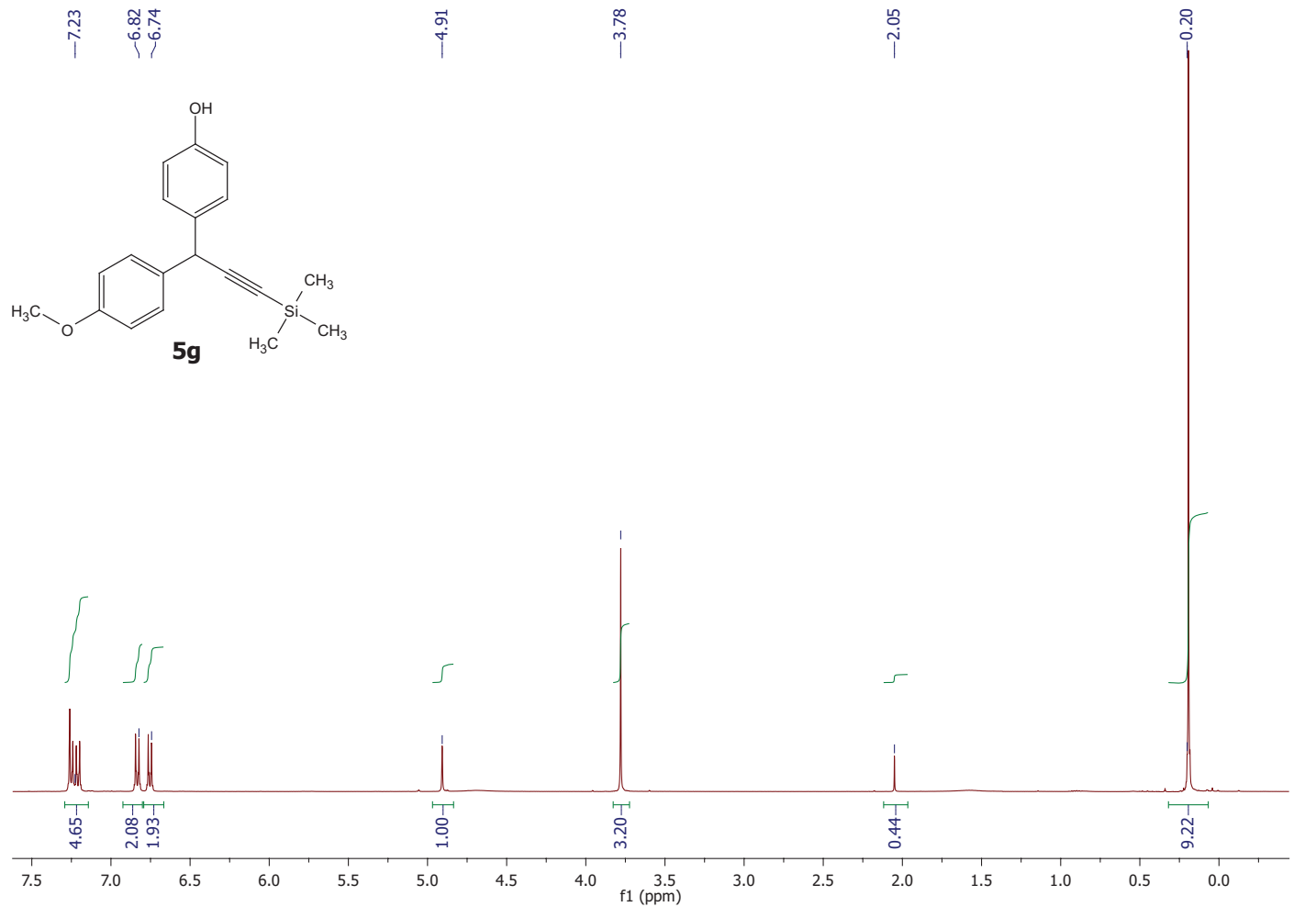

7.22
6.84
6.76
4.91
4.83
3.78
2.47
OH
CH
H3C
O
4.24
2.00
1.90
0.98
0.79
3.00
0.87
7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8
f1 (ppm)

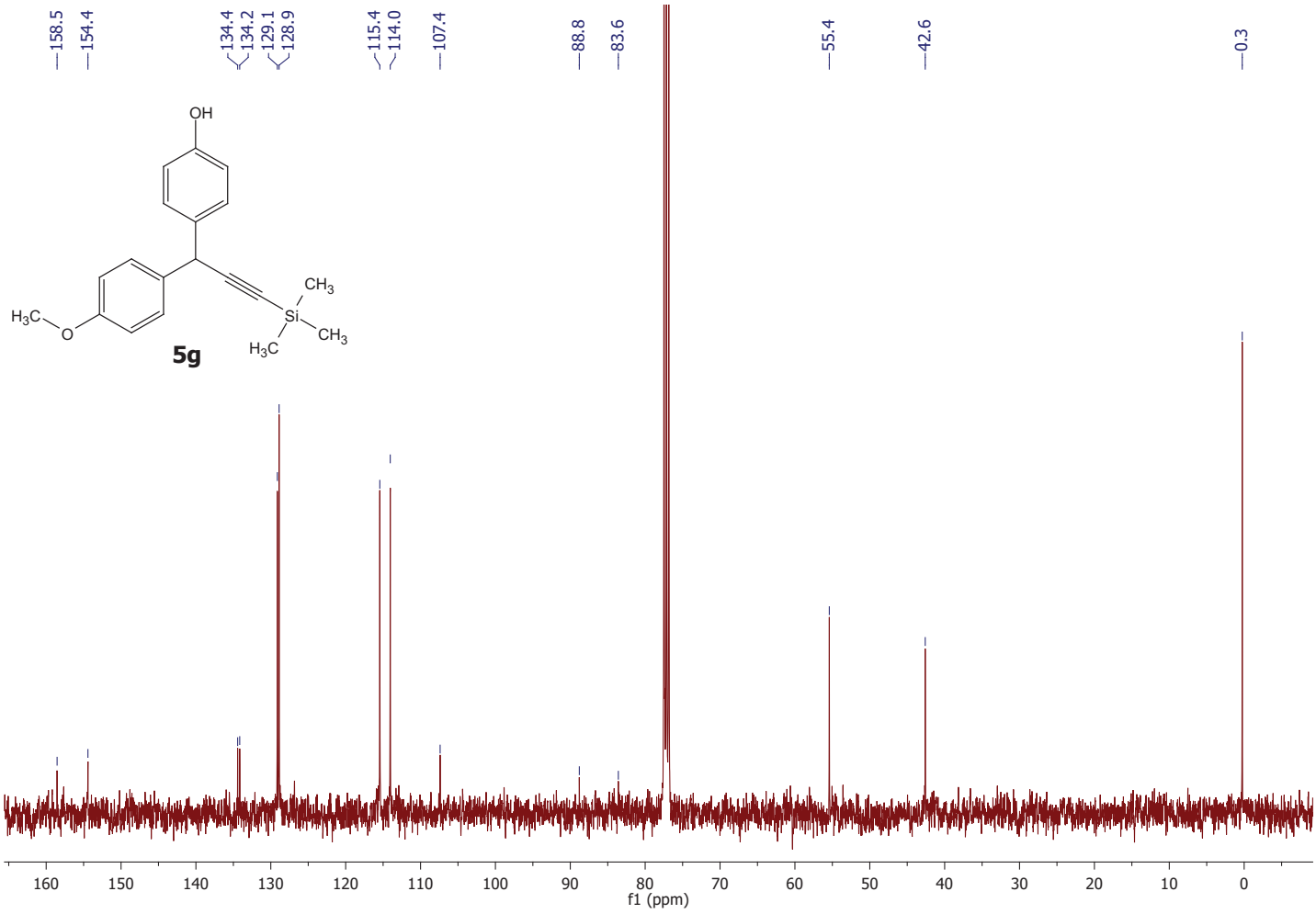
5e

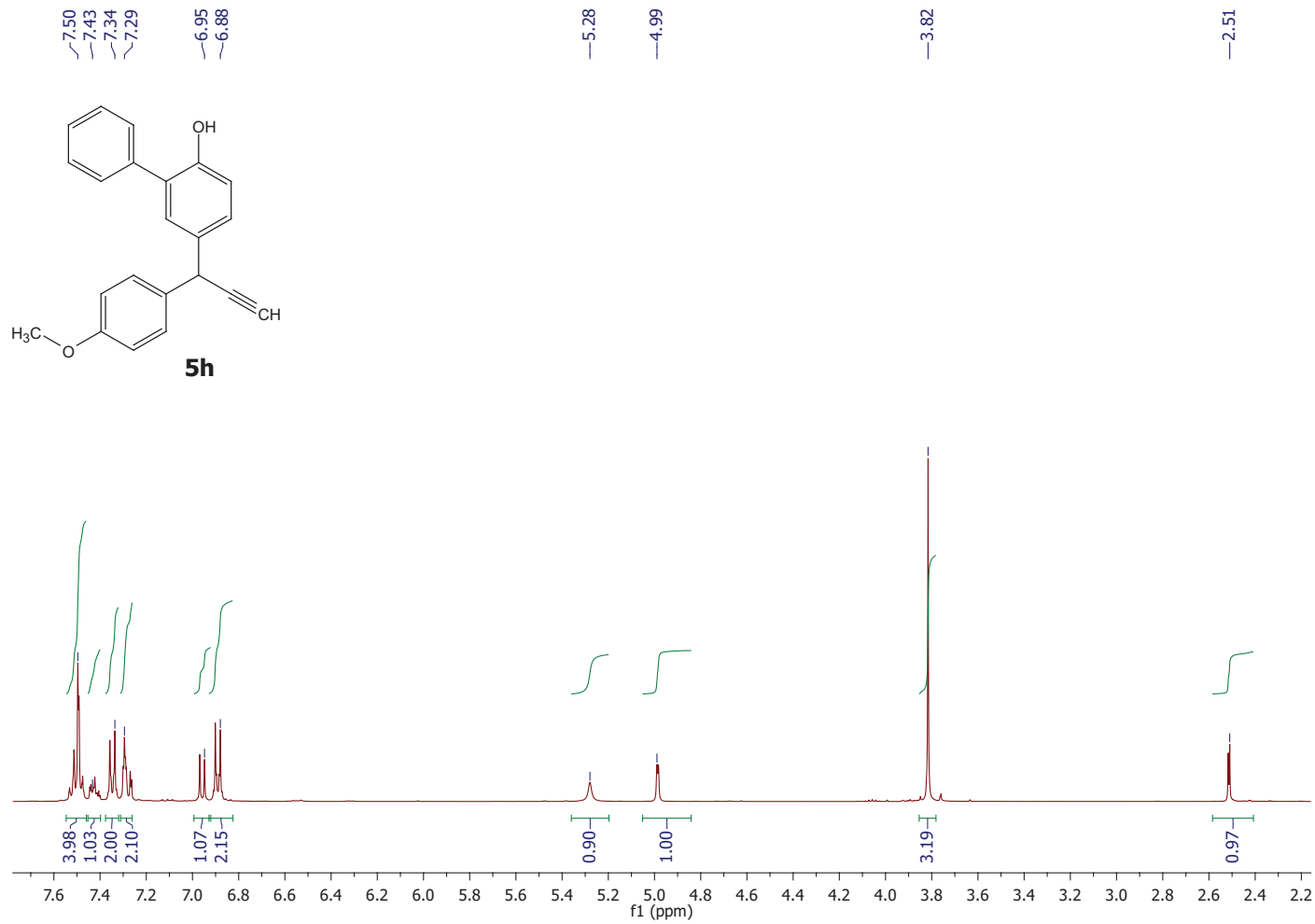

158.6
154.5
133.9
133.7
129.1
128.9
115.5
114.1
85.3
72.6
55.4
41.4
OH
CH
H3C
O
5e
170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35
f1 (ppm)

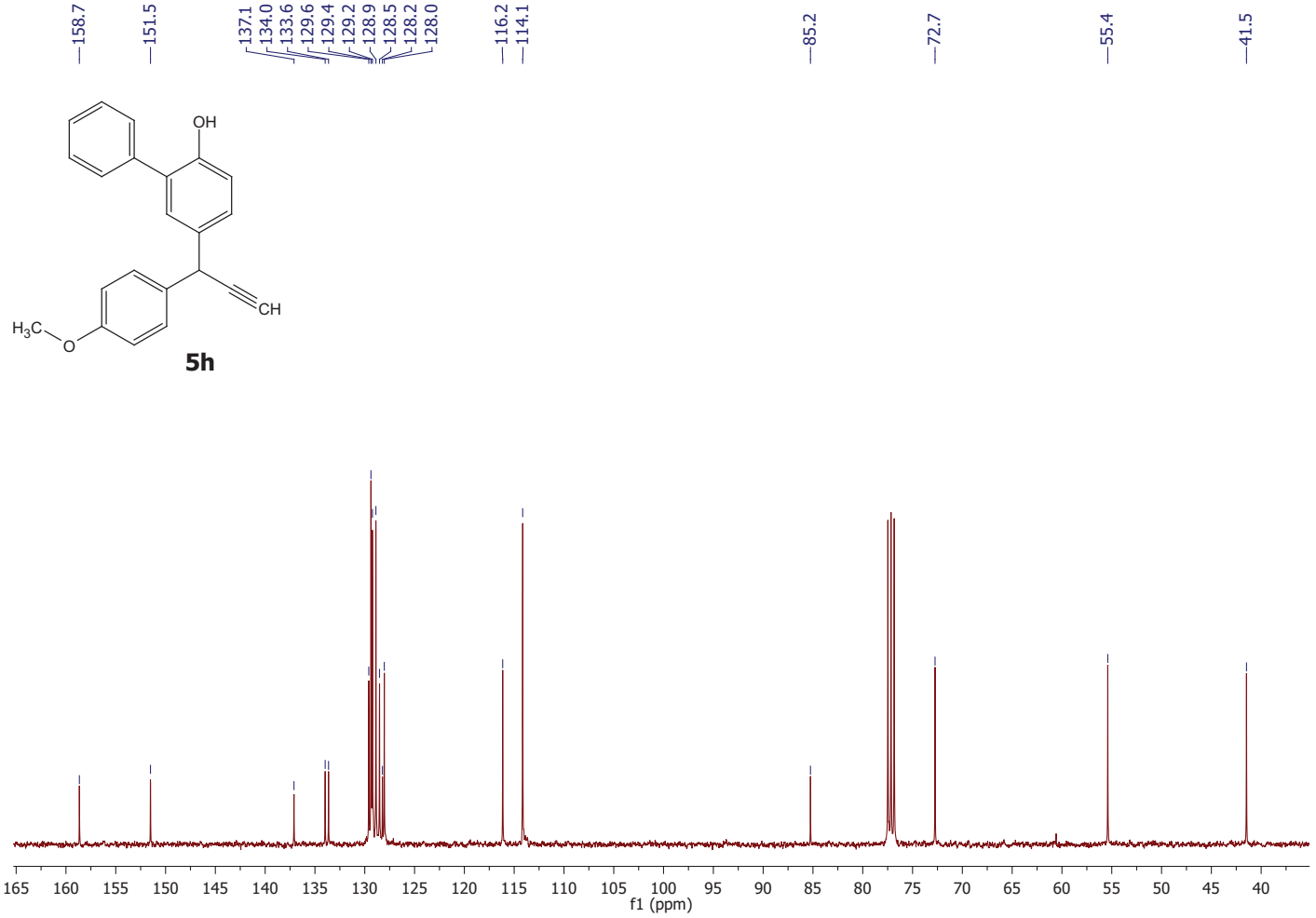

7.47
7.31
6.86
6.78
5.12
5.01
3.80
OH
H3C
O
5f
2.04
7.01
2.06
2.02
1.00
0.91
3.12
7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4
f1 (ppm)


158.5
154.7
134.4
134.3
131.8
129.1
128.9
128.3
128.0
123.7
115.5
114.1
90.9
84.7
55.4
42.2
OH
H3C
O
5f
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35
f1 (ppm)

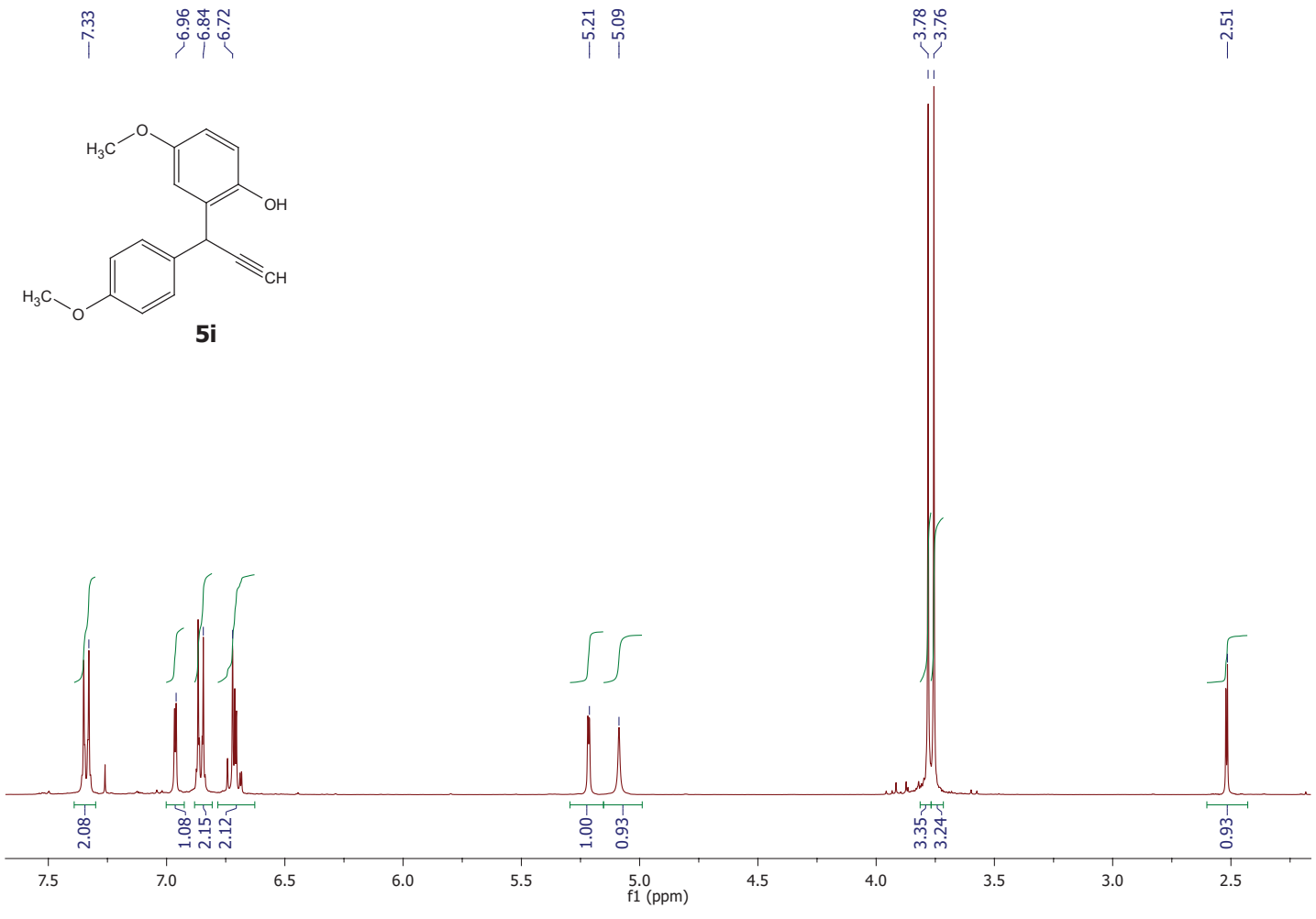

7.23
6.82
6.74
4.91
3.78
2.05
0.20
OH
H3C
O
CH3
Si
CH3
H3C
5g
4.65
2.08
1.93
1.00
3.20
0.44
9.22
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


158.5
154.4
134.4
134.2
129.1
128.9
115.4
114.0
107.4
88.8
83.6
55.4
42.6
0.3
OH
H3C
O
CH3
Si
CH3
H3C
5g
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

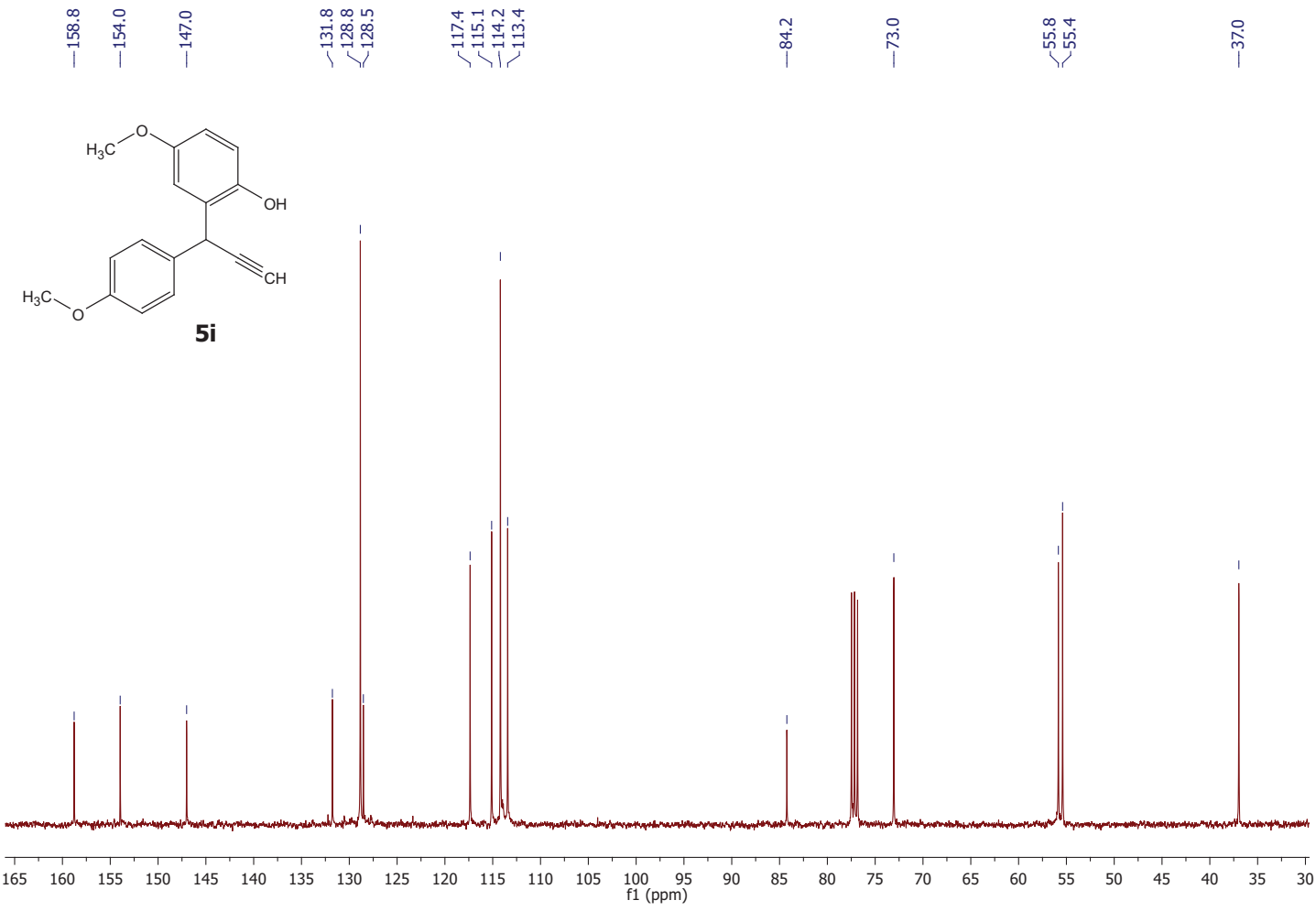

7.50
7.43
7.34
7.29
6.95
6.88
5.28
4.99
3.82
2.51
OH
H3C
O
CH
5h
3.98
1.03
2.00
2.10
1.07
2.15
0.90
1.00
3.19
0.97
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
6.0
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
f1 (ppm)

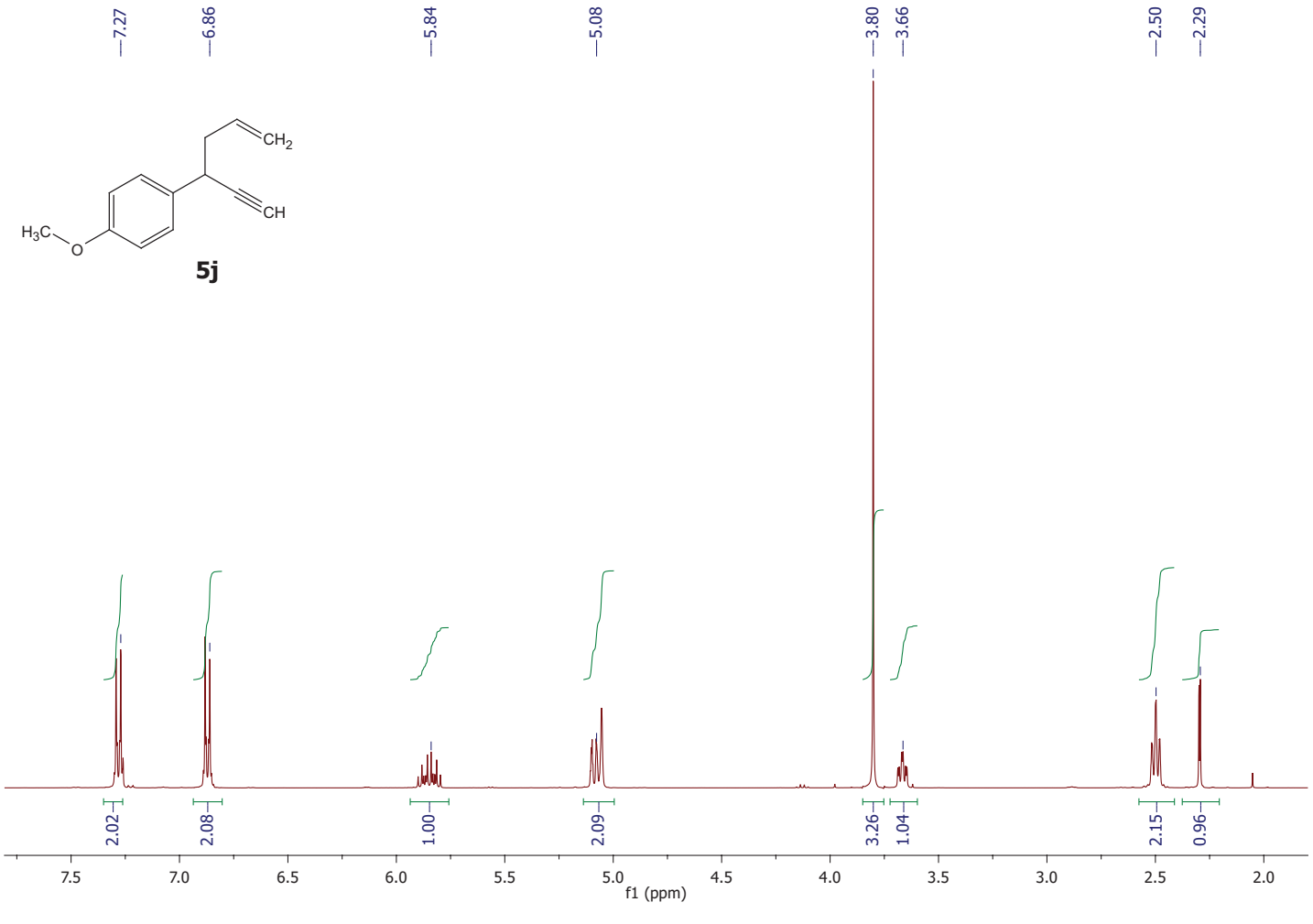

158.7
151.5
137.1
134.0
133.6
129.6
129.4
129.2
128.9
128.5
128.2
128.0
116.2
114.1
85.2
72.7
55.4
41.5
OH
CH
H3C
O
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40
f1 (ppm)

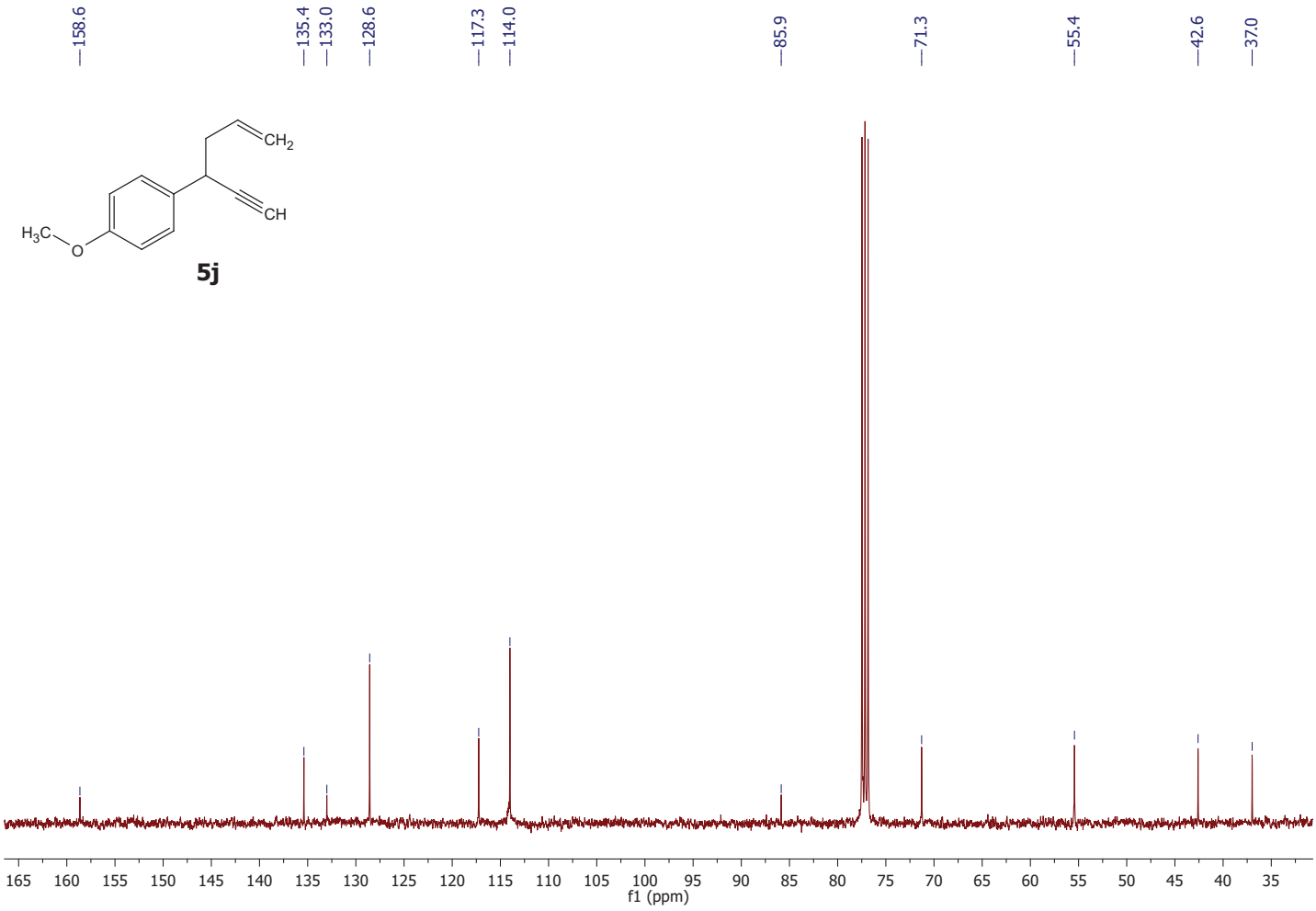
5h


7.33
6.96
6.84
6.72
5.21
5.09
3.78
3.76
2.51
H3C
O
OH
CH
2.08
1.08
2.15
2.12
1.00
0.93
3.35
3.24
0.93
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5
f1 (ppm)

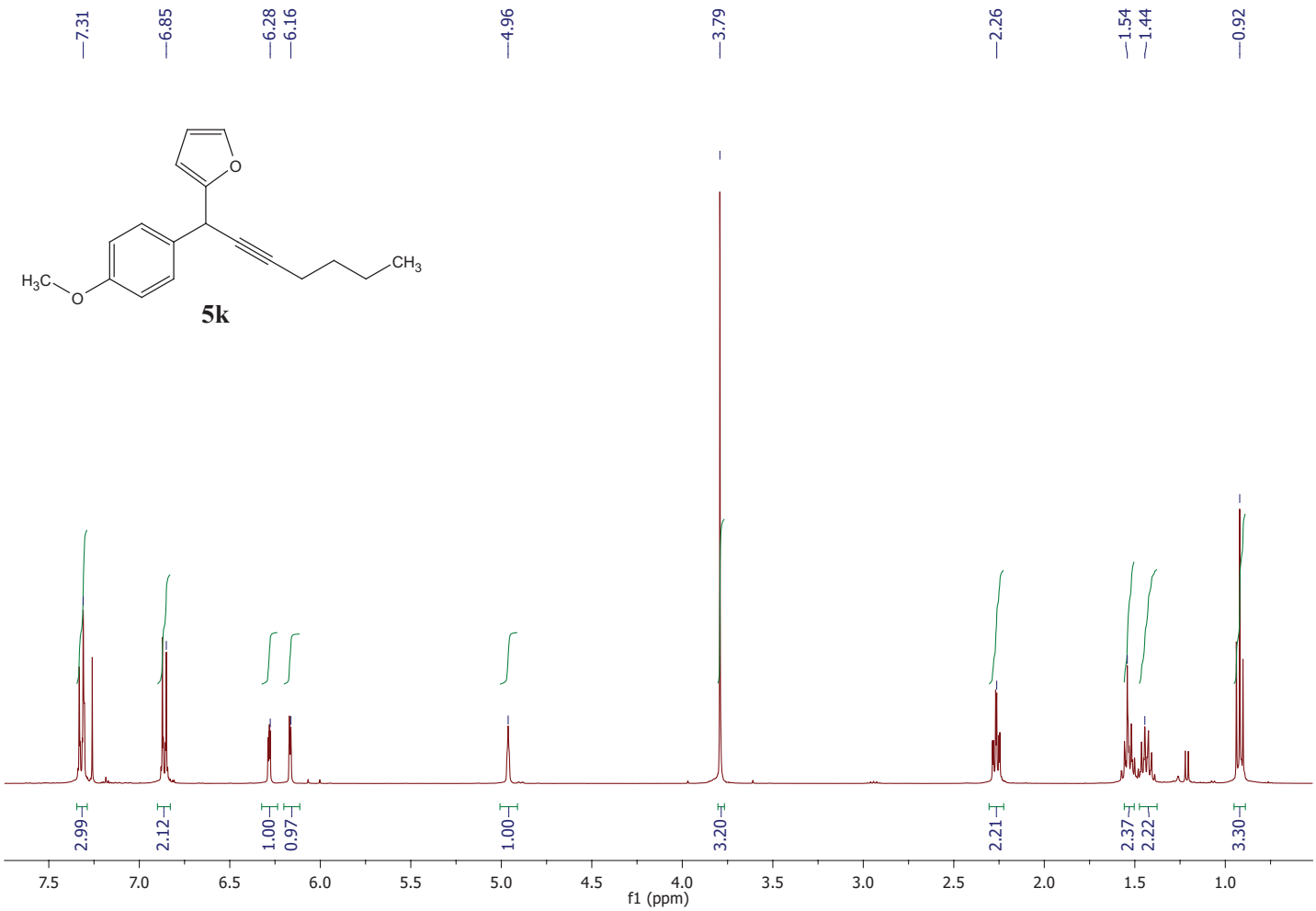
5i


158.8
154.0
147.0
131.8
128.8
128.5
117.4
115.1
114.2
113.4
84.2
73.0
55.8
55.4
37.0
OH
CH
H3C
O
5i
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30
f1 (ppm)

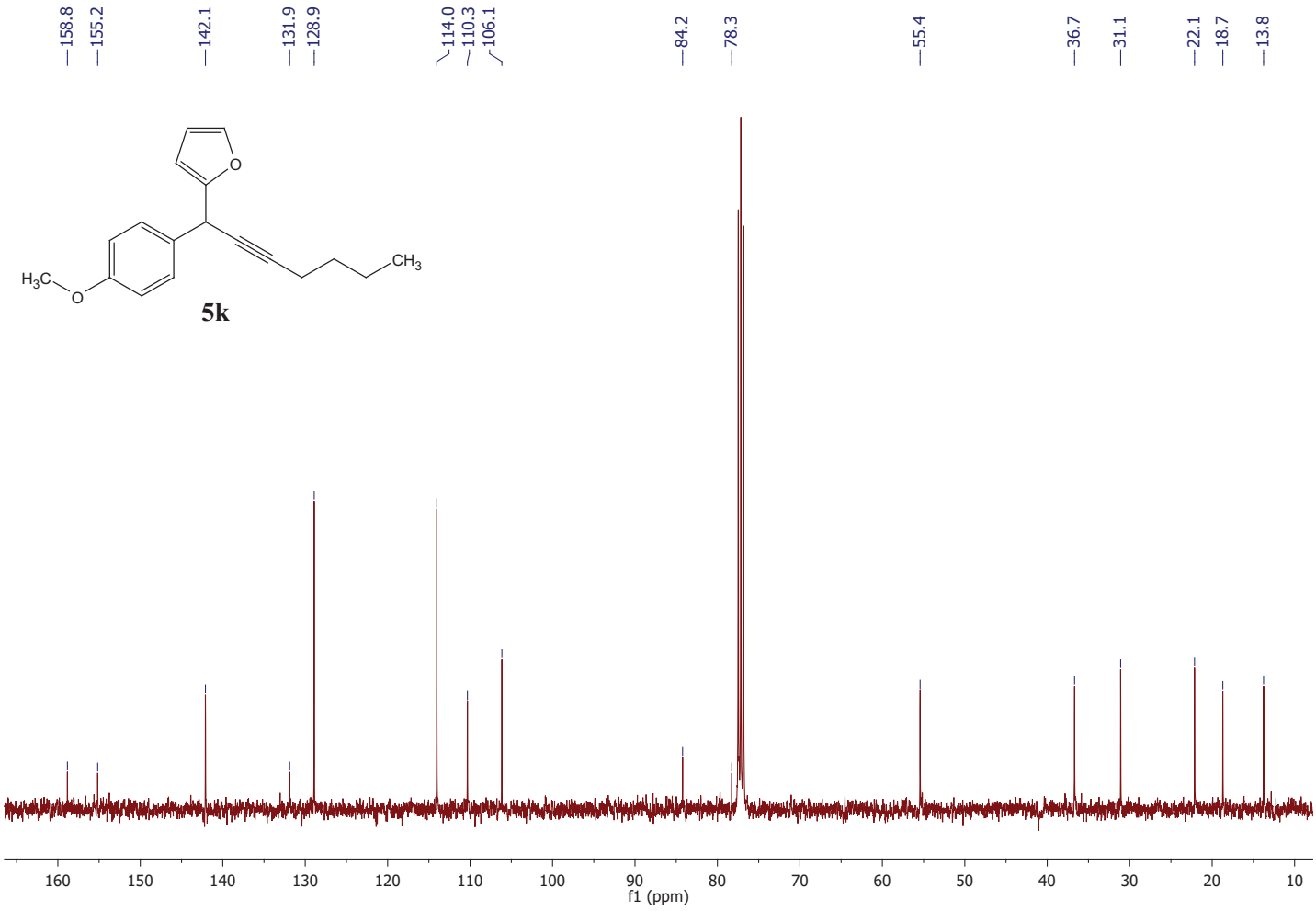

7.27
6.86
5.84
5.08
3.80
3.66
2.50
2.29
CH2
CH
H3C
O
5j
2.02
2.08
1.00
2.09
3.26
1.04
2.15
0.96
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
f1 (ppm)


158.6
135.4
133.0
128.6
117.3
114.0
85.9
71.3
55.4
42.6
37.0
CH2
CH
H3C
O
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35
f1 (ppm)

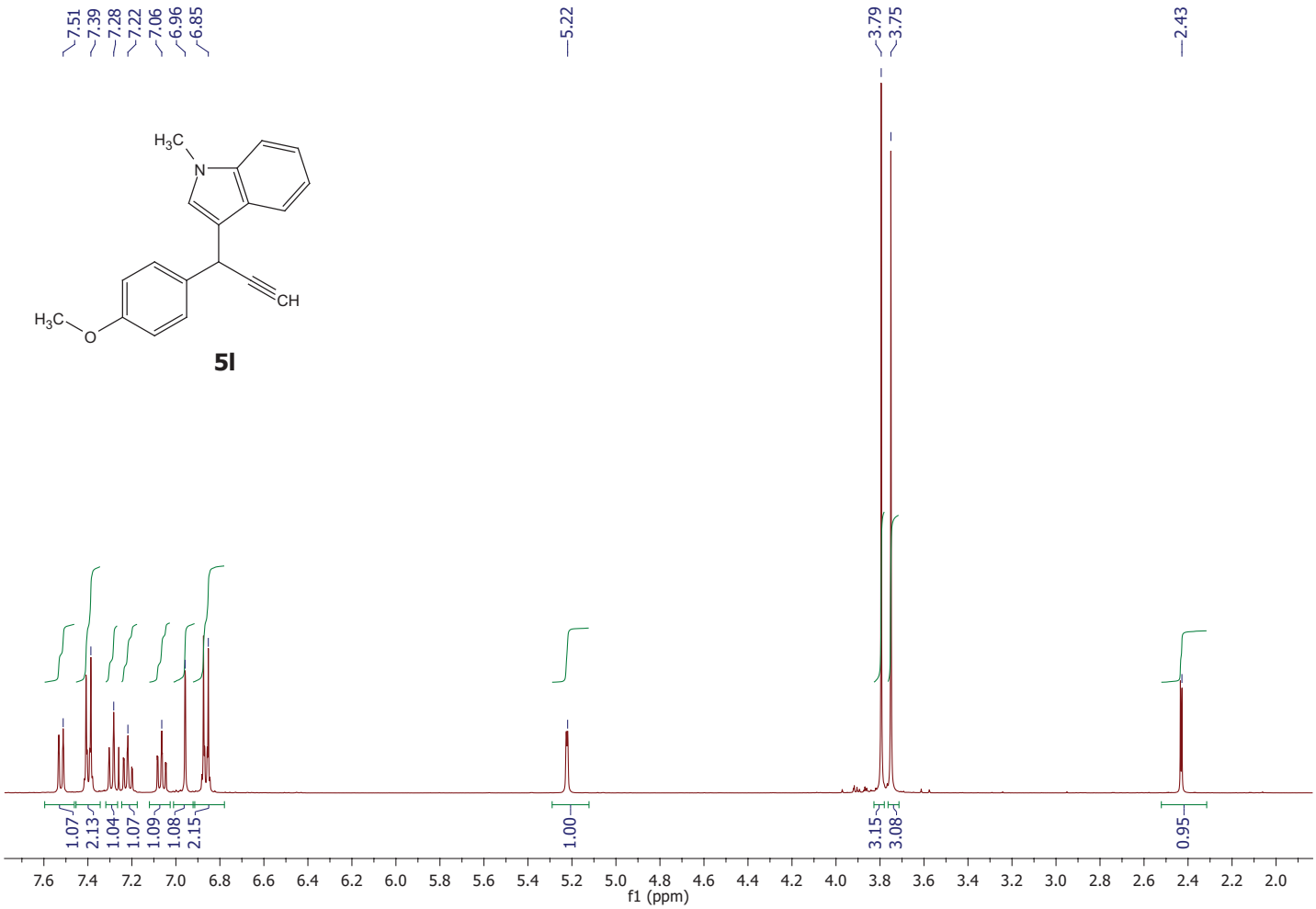
5j


7.31
6.85
6.28
6.16
4.96
3.79
2.26
1.54
1.44
0.92
O
H3C
O
CH3
2.99
2.12
1.00
0.97
1.00
3.20
2.21
2.37
2.22
3.30
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
f1 (ppm)

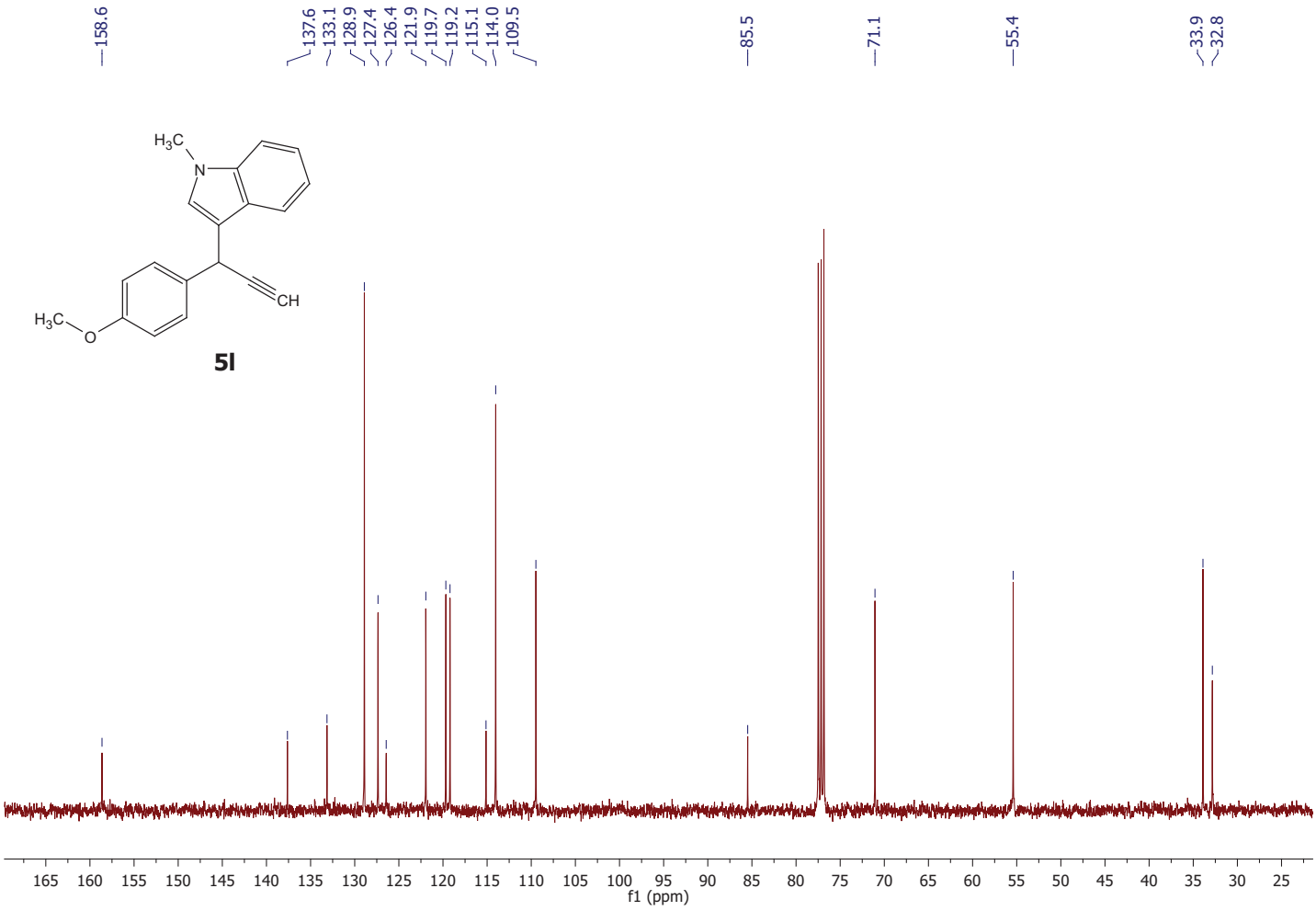
5k

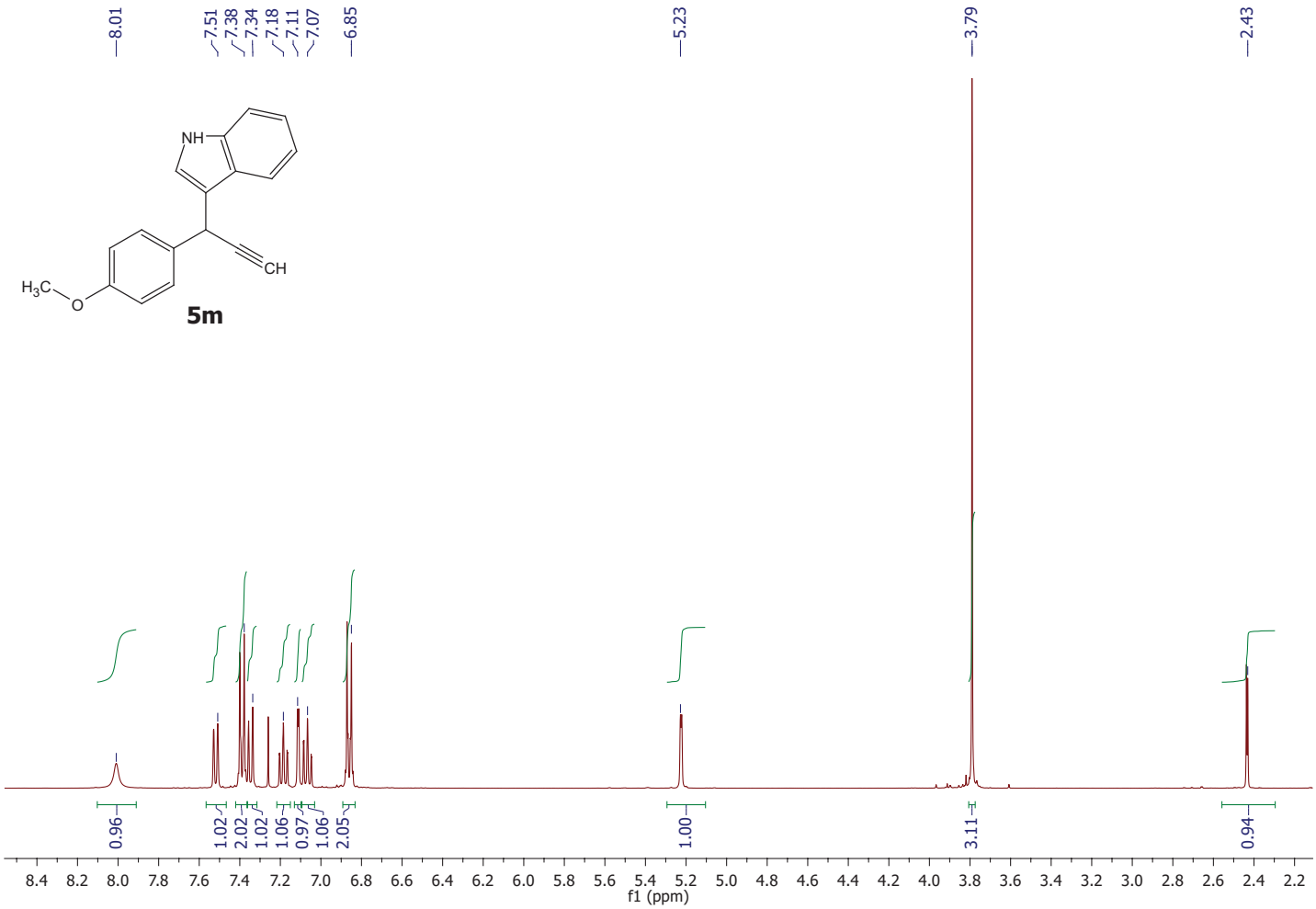

158.8
155.2
142.1
131.9
128.9
114.0
110.3
106.1
84.2
78.3
55.4
36.7
31.1
22.1
18.7
13.8
O
H3C
O
CH3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

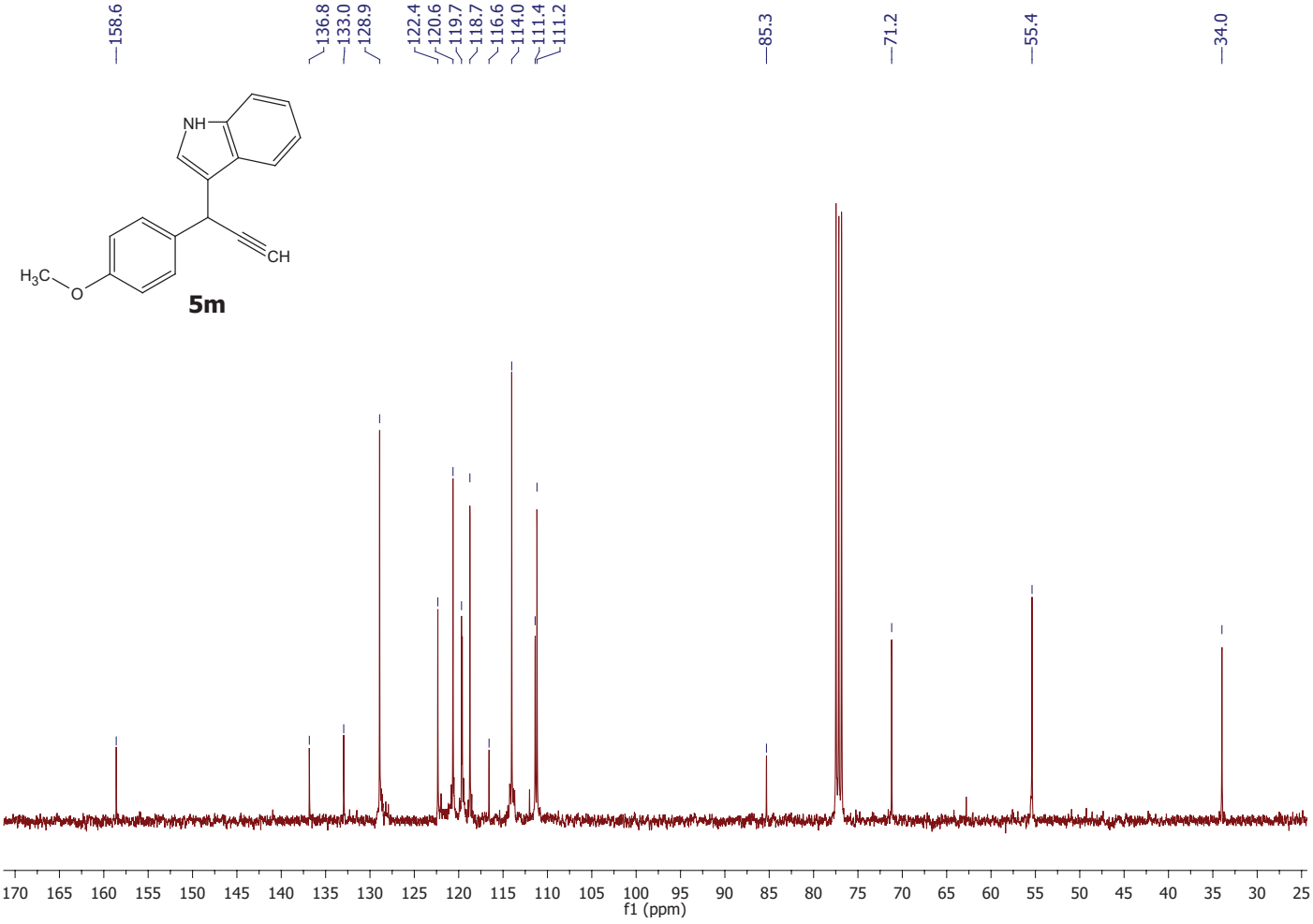
5k

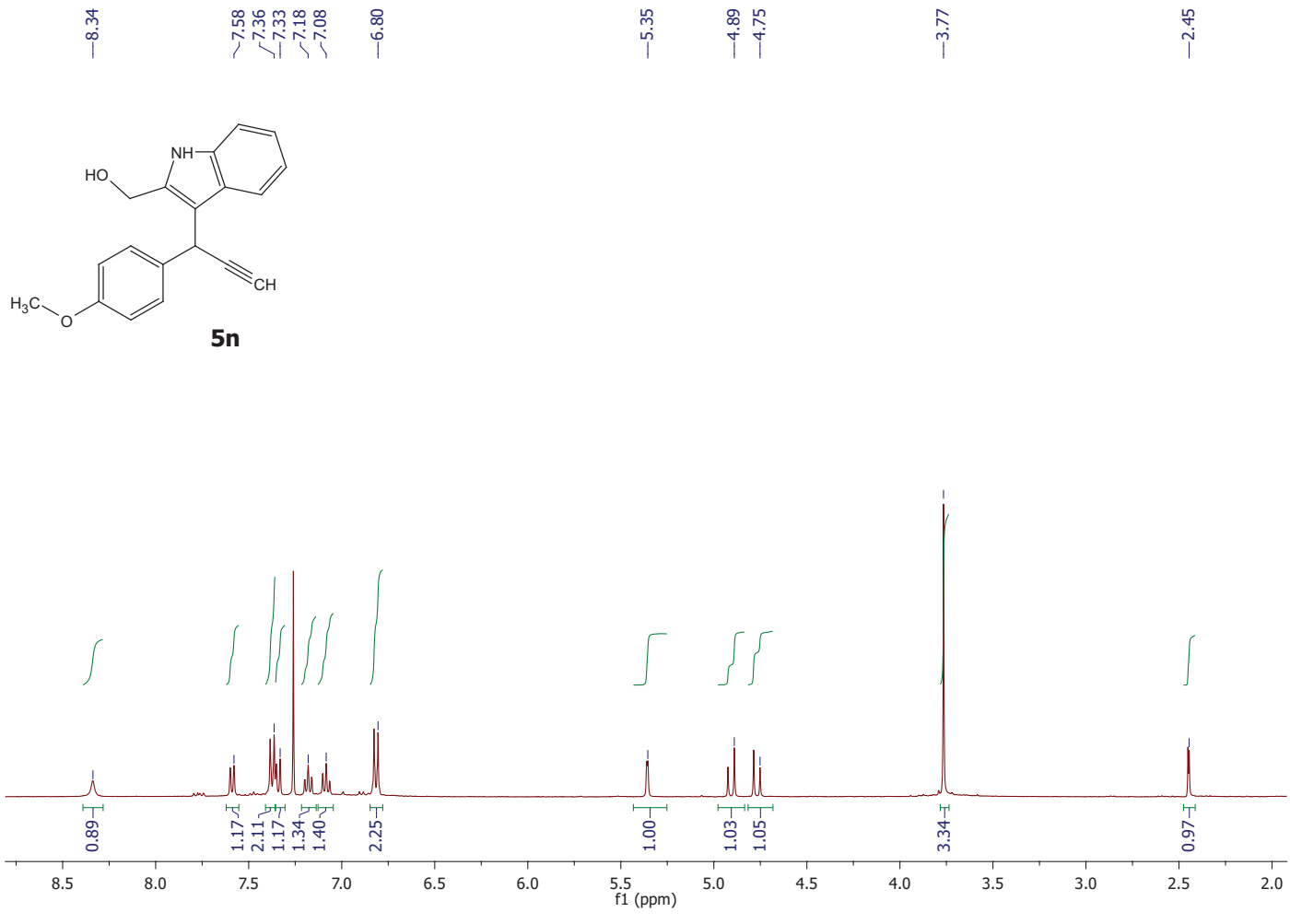

7.51
7.39
7.28
7.22
7.06
6.96
6.85
5.22
3.79
3.75
2.43
H3C
N
CH
H3C
O
1.07
2.13
1.04
1.07
1.09
1.08
2.15
1.00
3.15
3.08
0.95
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
6.0
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
f1 (ppm)

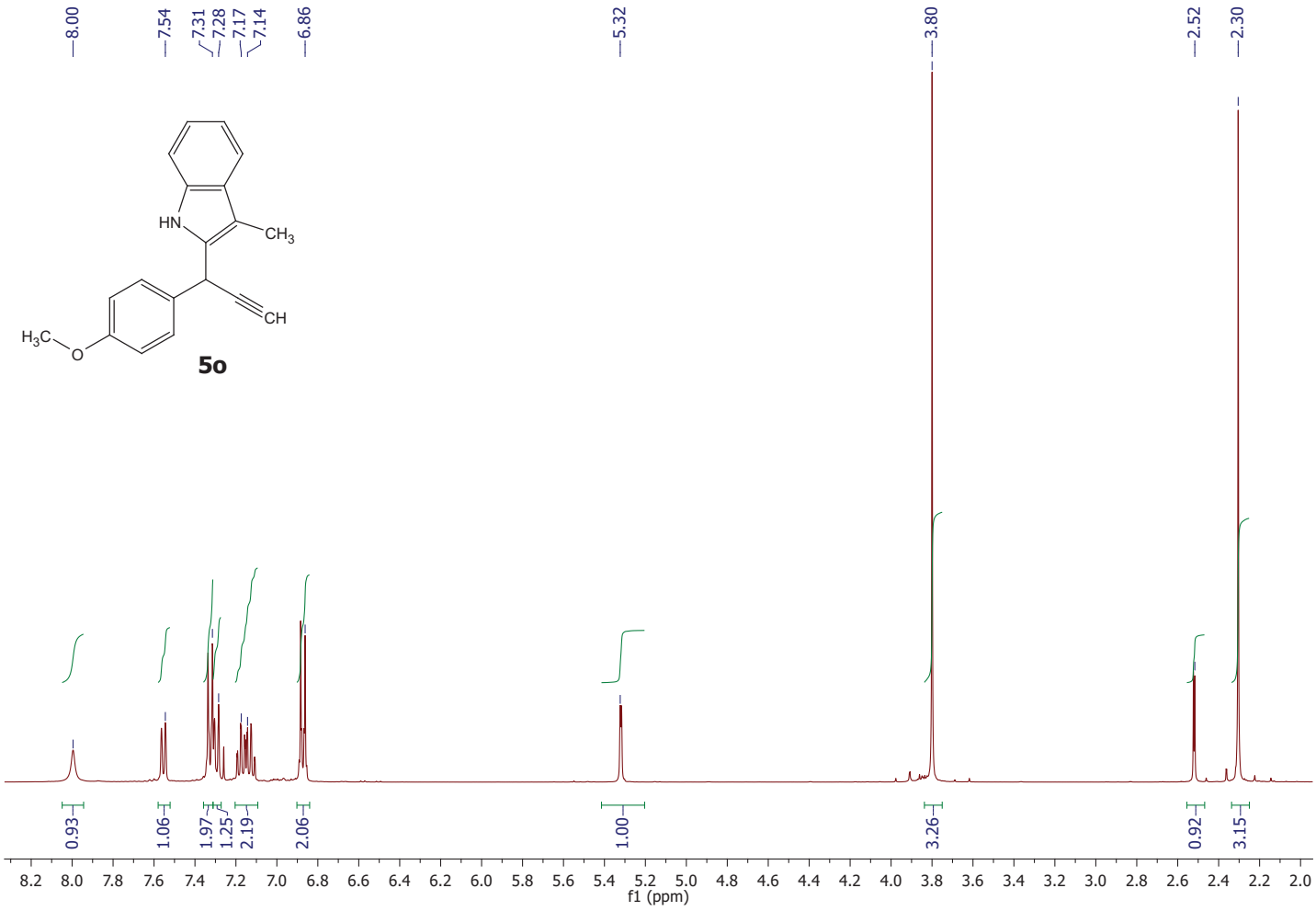
5l

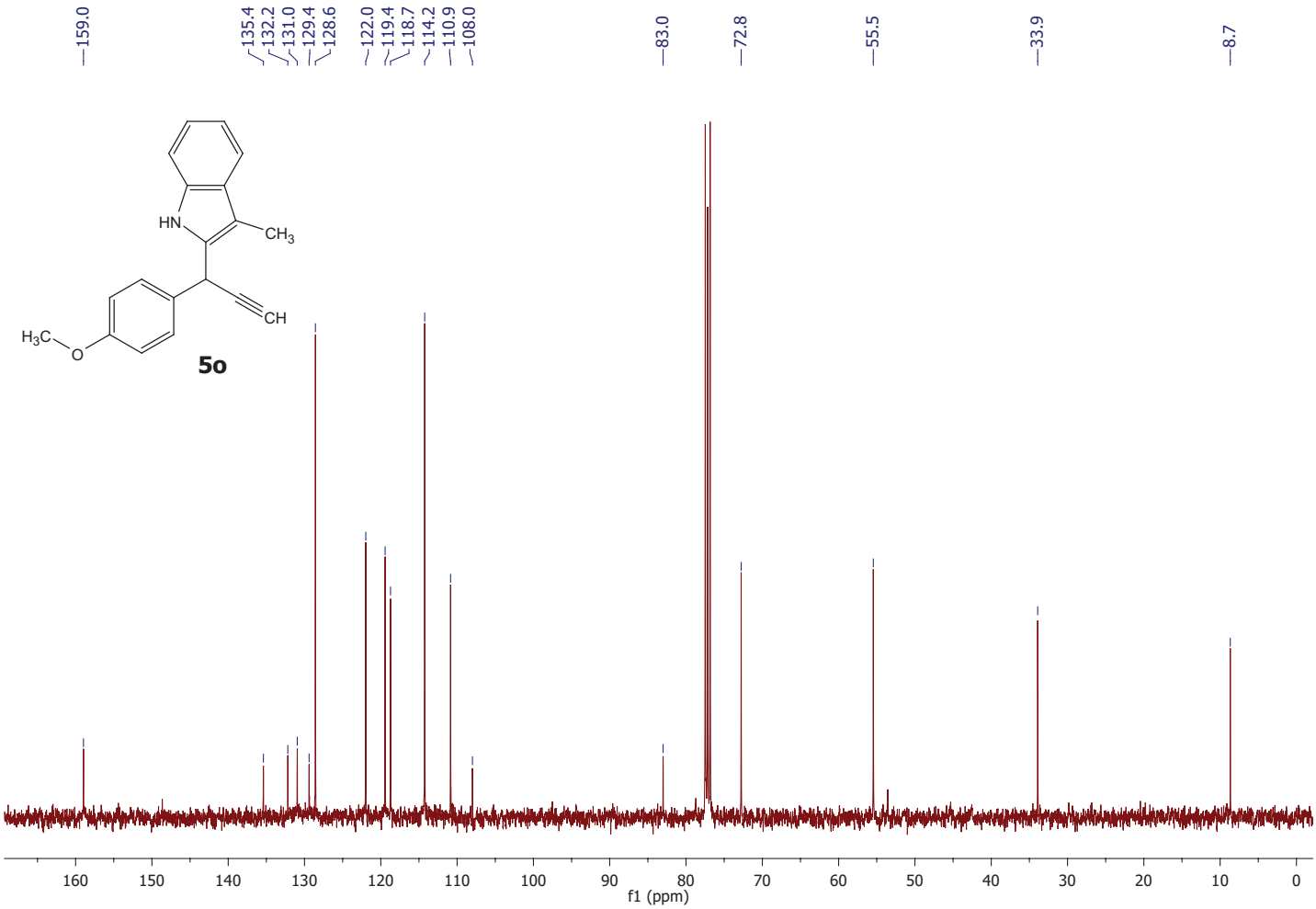

158.6
137.6
133.1
128.9
127.4
126.4
121.9
119.7
119.2
115.1
114.0
109.5
85.5
71.1
55.4
33.9
32.8
H3C
N
CH
H3C
O
5l
165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25
f1 (ppm)

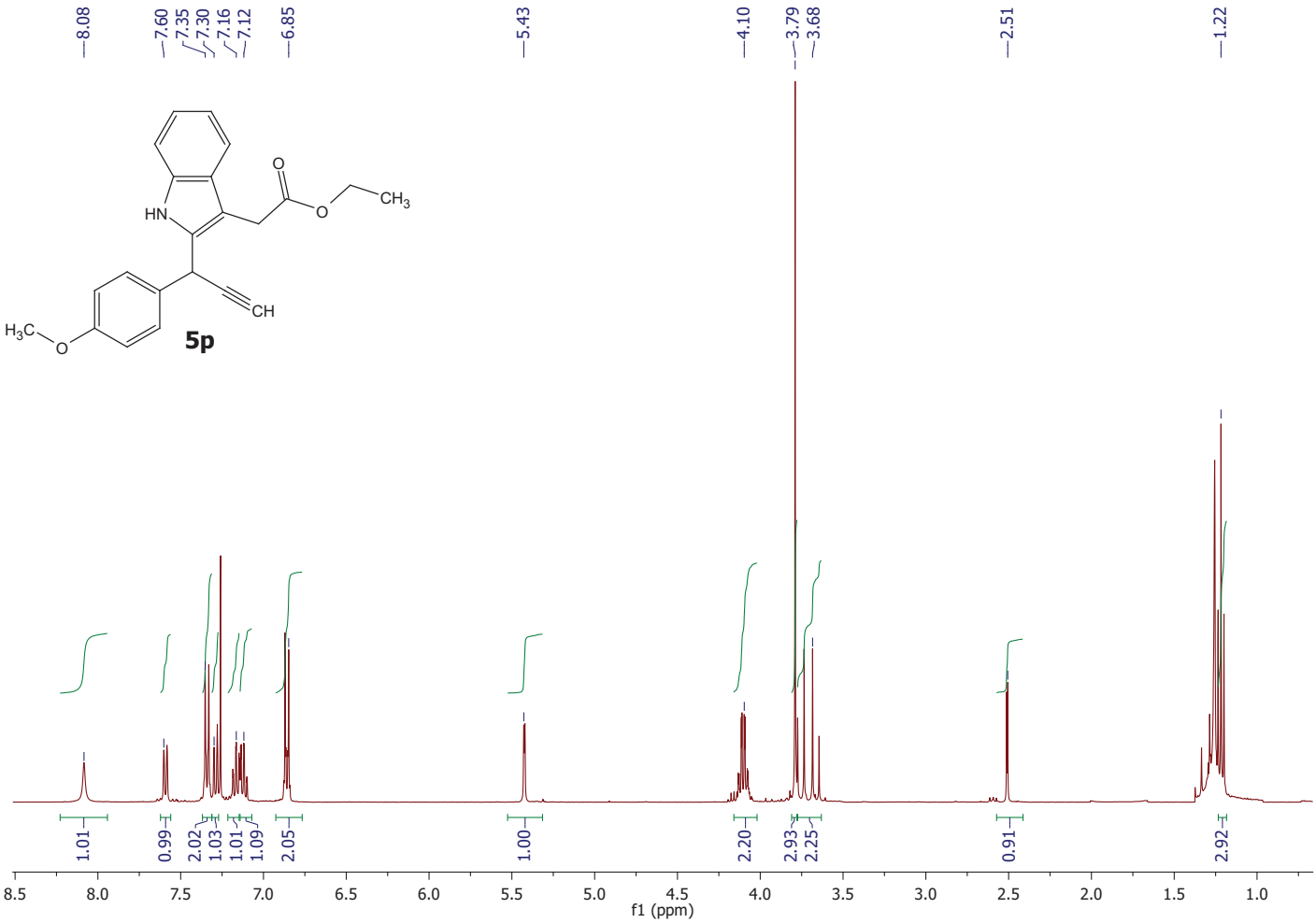

8.01
7.51
7.38
7.34
7.18
7.11
7.07
6.85
5.23
3.79
2.43
NH
CH
H3C
O
5m
0.96
1.02
2.02
1.02
1.06
0.97
1.06
2.05
1.00
3.11
0.94
8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2
f1 (ppm)

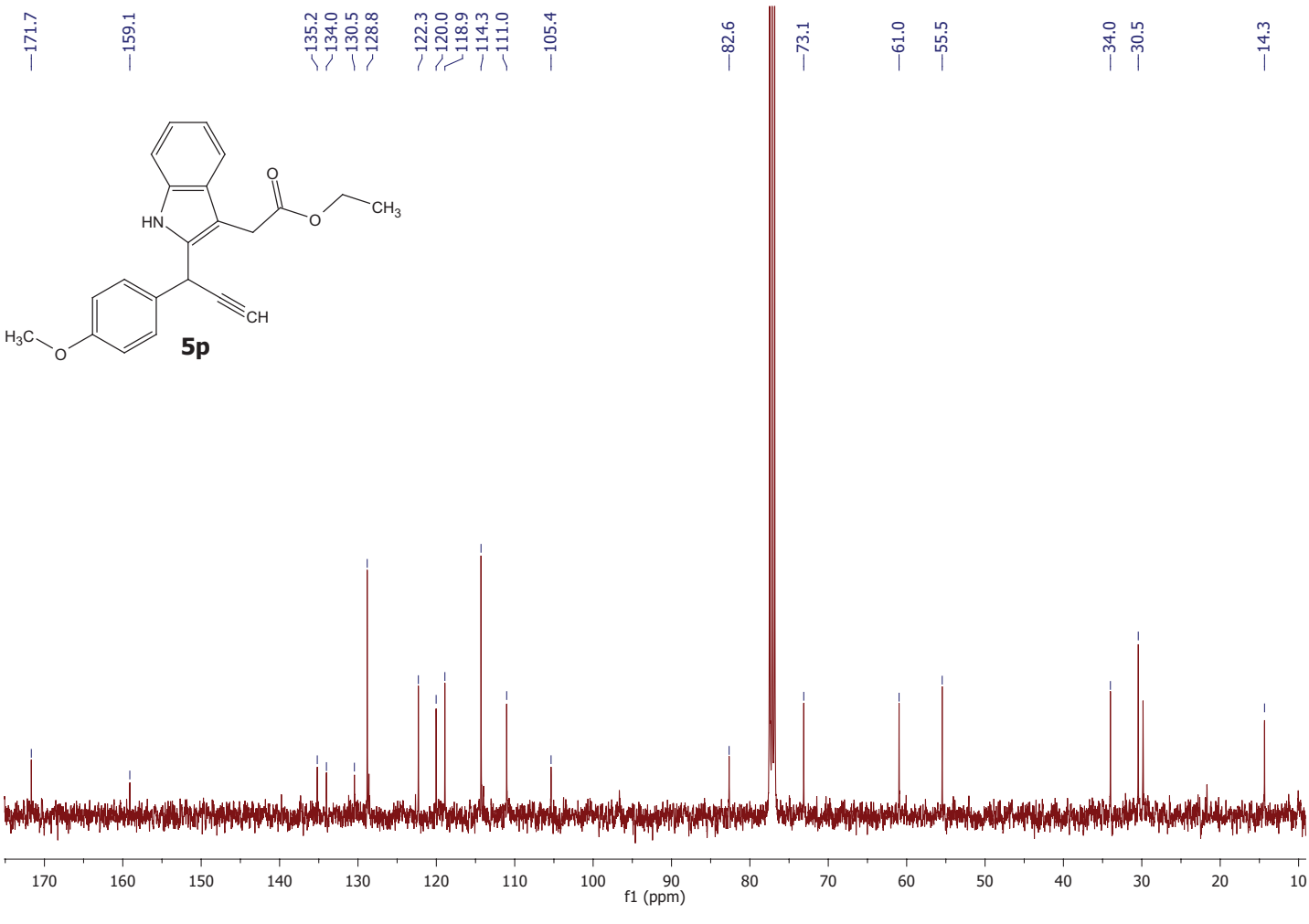

158.6
136.8
133.0
128.9
122.4
120.6
119.7
118.7
116.6
114.0
111.4
111.2
85.3
71.2
55.4
34.0
NH
CH
H3C
O
5m
170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25
f1 (ppm)

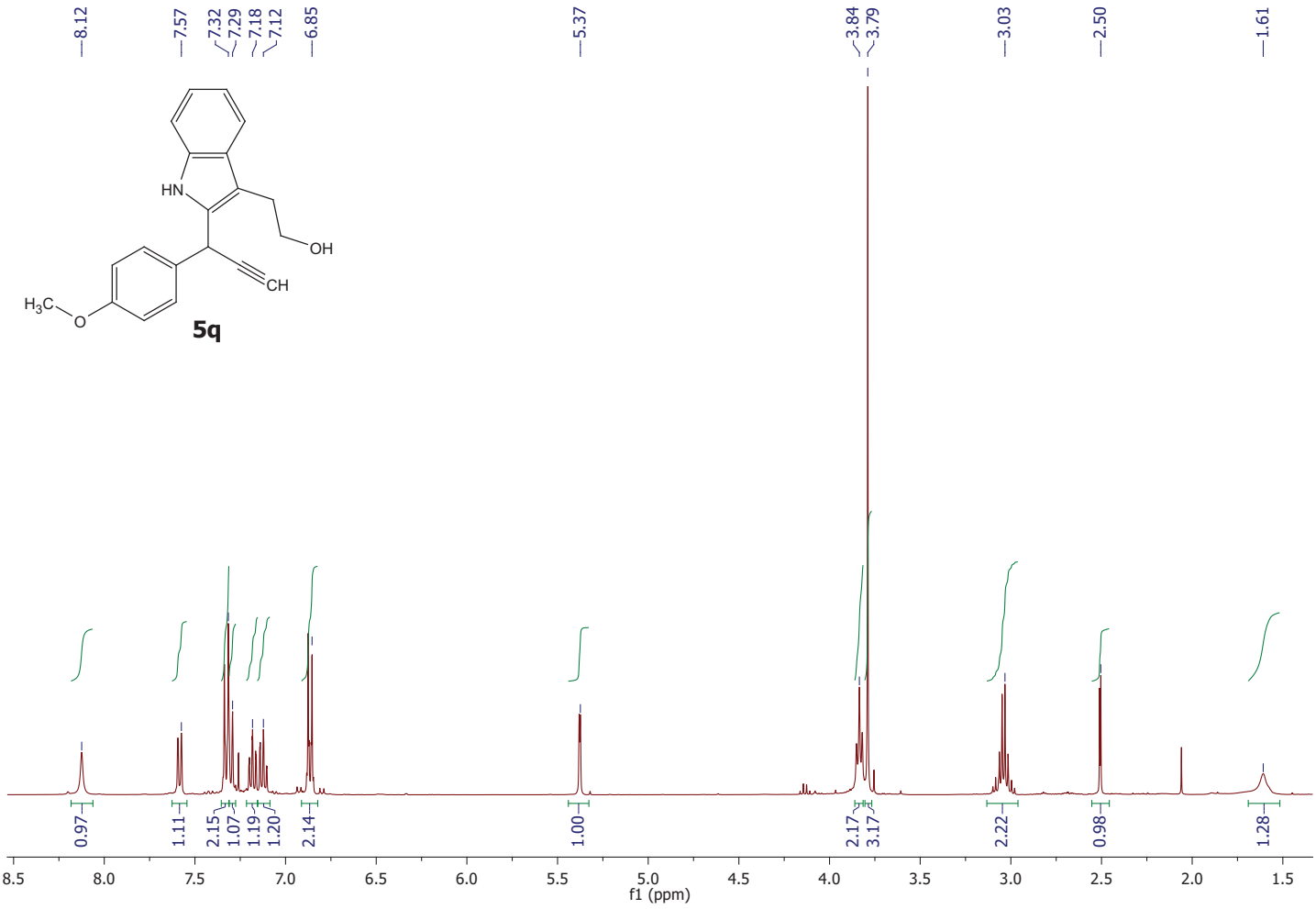

8.34
7.58
7.36
7.33
7.18
7.08
6.80
5.35
4.89
4.75
3.77
2.45
NH
HO
CH
H3C
O
5n
0.89
1.17
2.11
1.17
1.34
1.40
2.25
1.00
1.03
1.05
3.34
0.97
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
f1 (ppm)

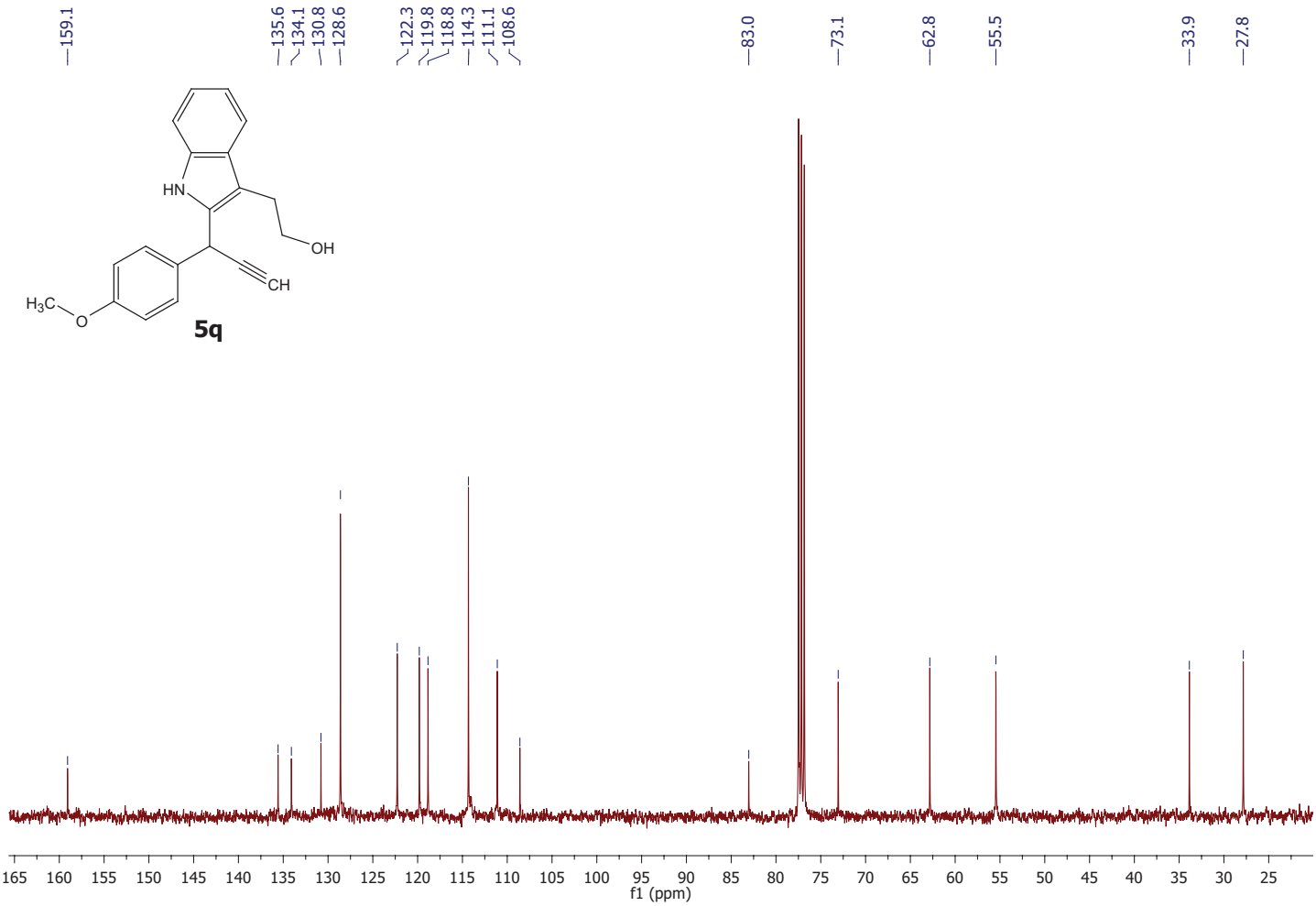

8.00
7.54
7.31
7.28
7.17
7.14
6.86
5.32
3.80
2.52
2.30
HN
CH3
CH
H3C
O
5o
0.93
1.06
1.97
1.25
2.19
2.06
1.00
3.26
0.92
3.15
f1 (ppm)

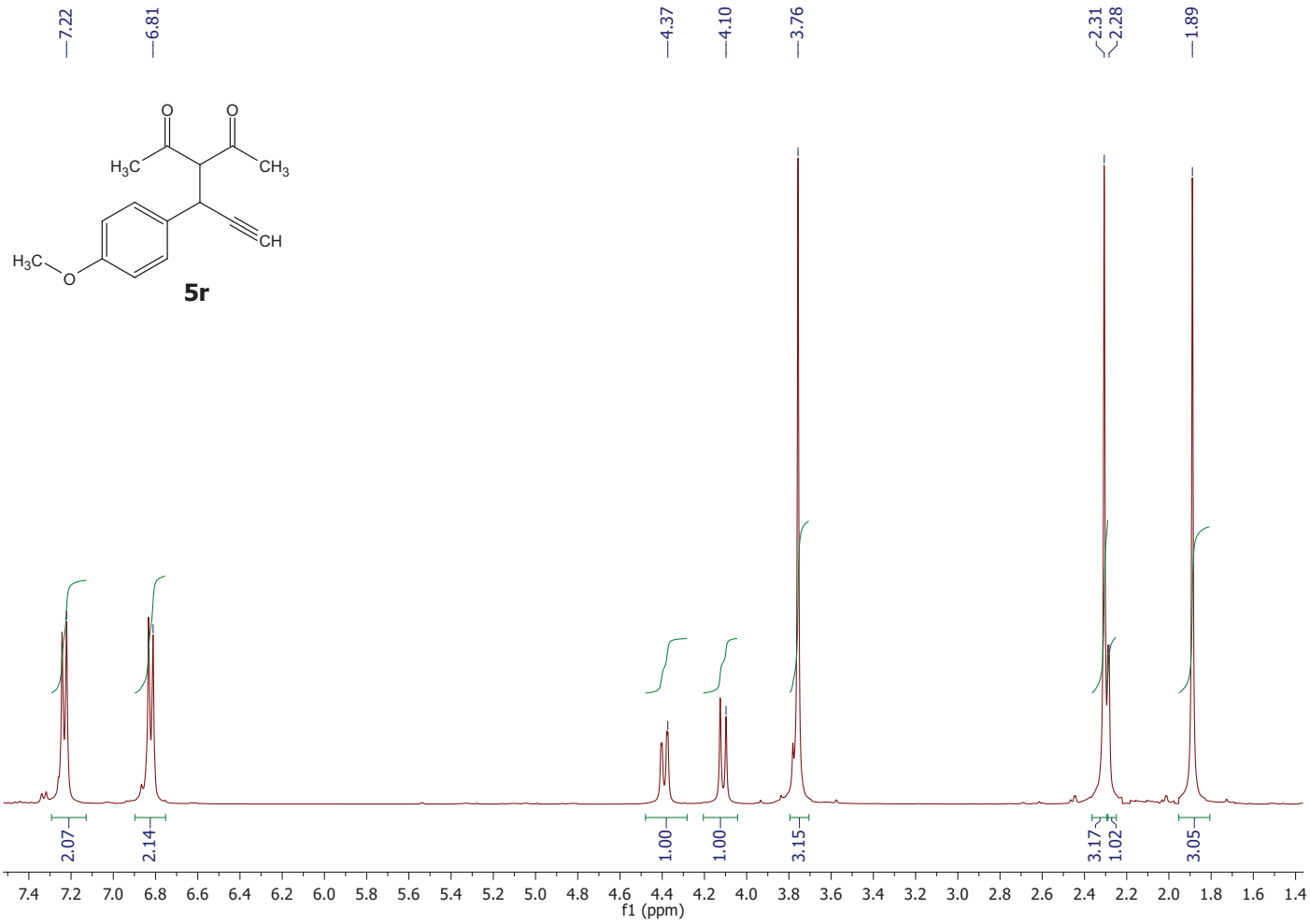

159.0
135.4
132.2
131.0
129.4
128.6
122.0
119.4
118.7
114.2
110.9
108.0
83.0
72.8
55.5
33.9
8.7
HN
CH3
CH
H3C
O
5o
f1 (ppm)

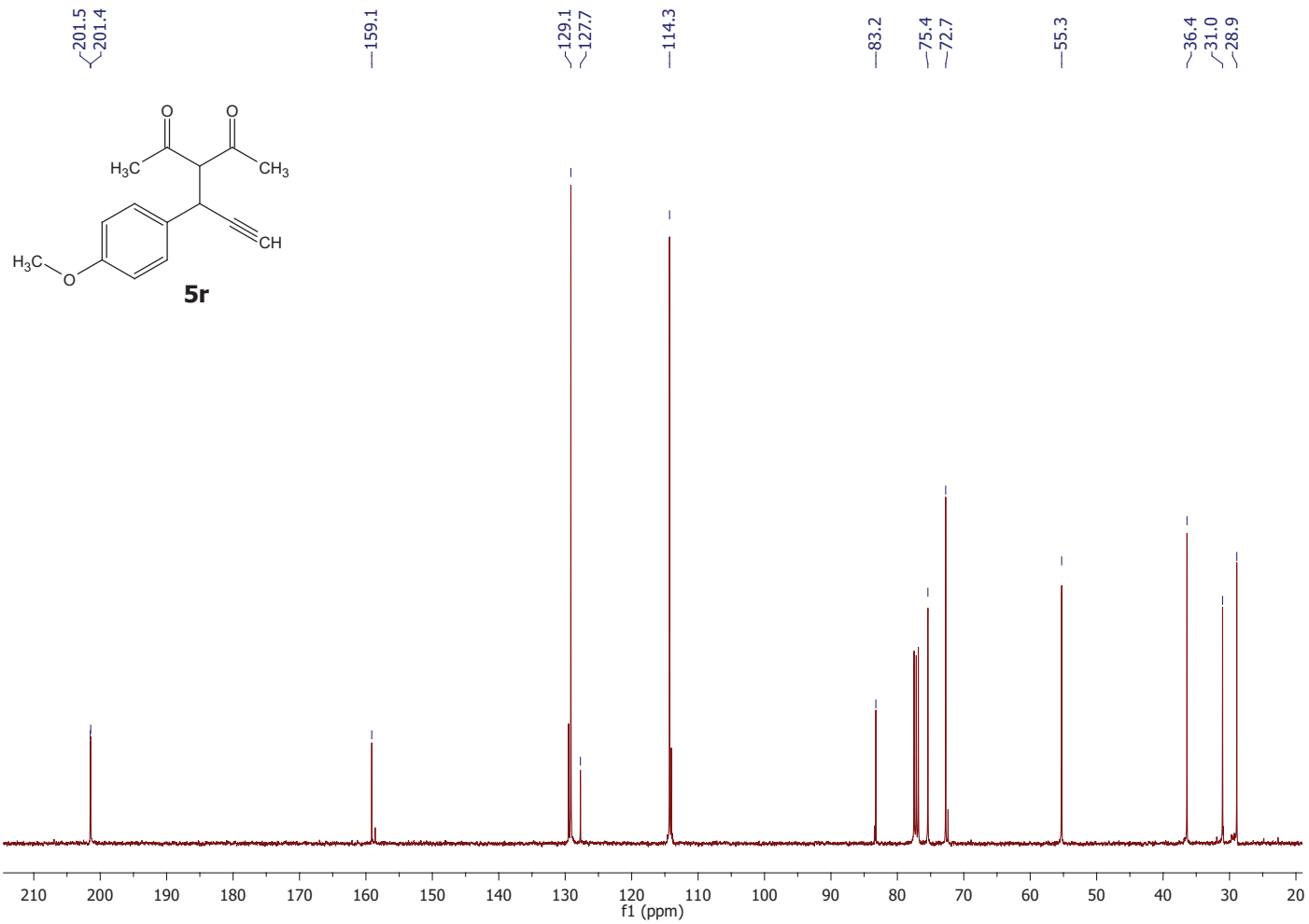

8.08
7.60
7.35
7.30
7.16
7.12
6.85
5.43
4.10
3.79
3.68
2.51
1.22
HN
O
O
CH3
H3C
O
CH
5p
1.01
0.99
2.02
1.03
1.01
1.09
2.05
1.00
2.20
2.93
2.25
0.91
2.92
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)

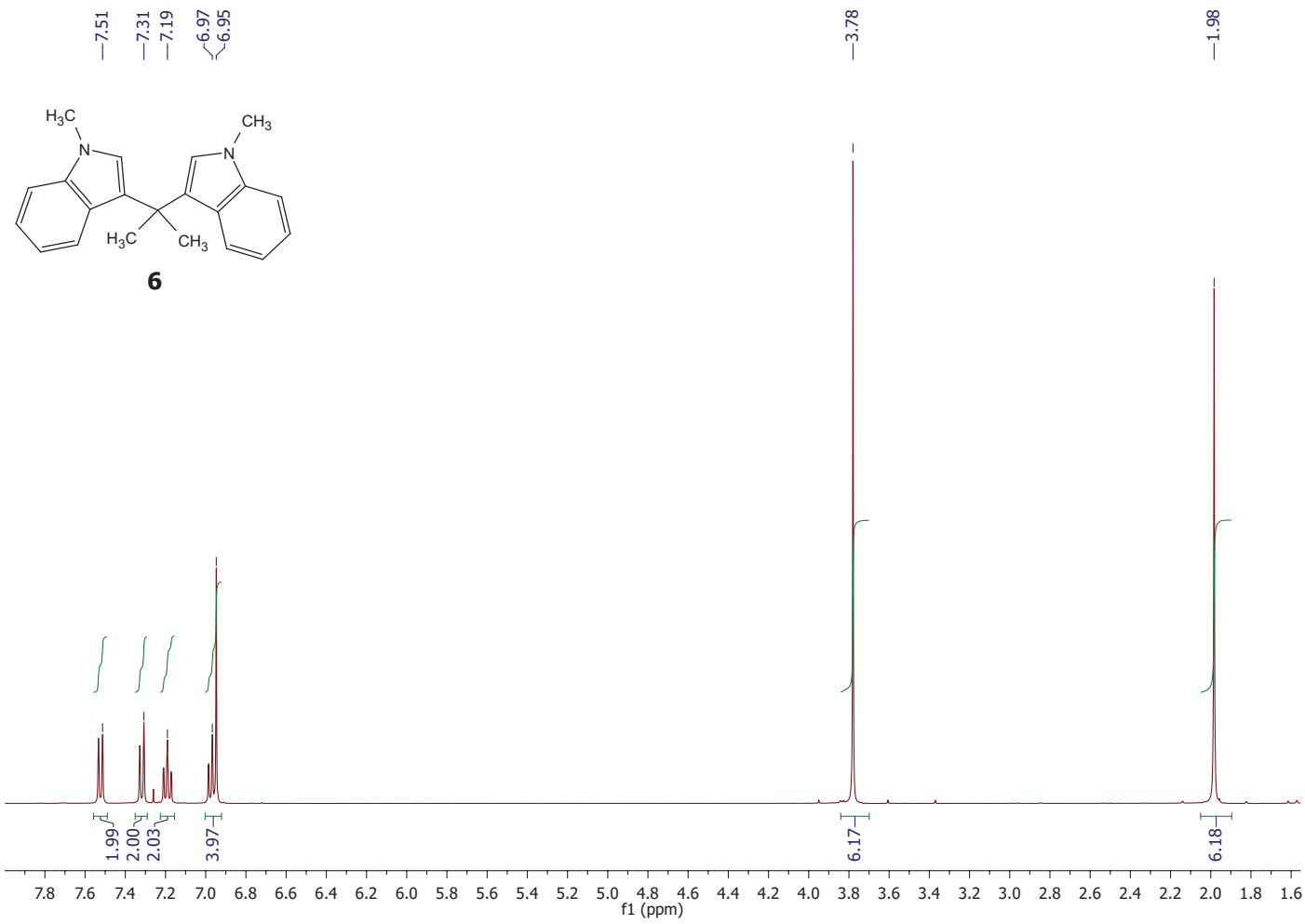

171.7
159.1
135.2
134.0
130.5
128.8
122.3
120.0
118.9
114.3
111.0
105.4
82.6
73.1
61.0
55.5
34.0
30.5
14.3
HN
O
O
CH3
H3C
O
CH
5p
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


8.12
7.57
7.32
7.29
7.18
7.12
6.85
5.37
3.84
3.79
3.03
2.50
1.61
HN
OH
CH
H3C
O
5q
0.97
1.11
2.15
1.07
1.19
1.20
2.14
1.00
2.17
3.17
2.22
0.98
1.28
f1 (ppm)

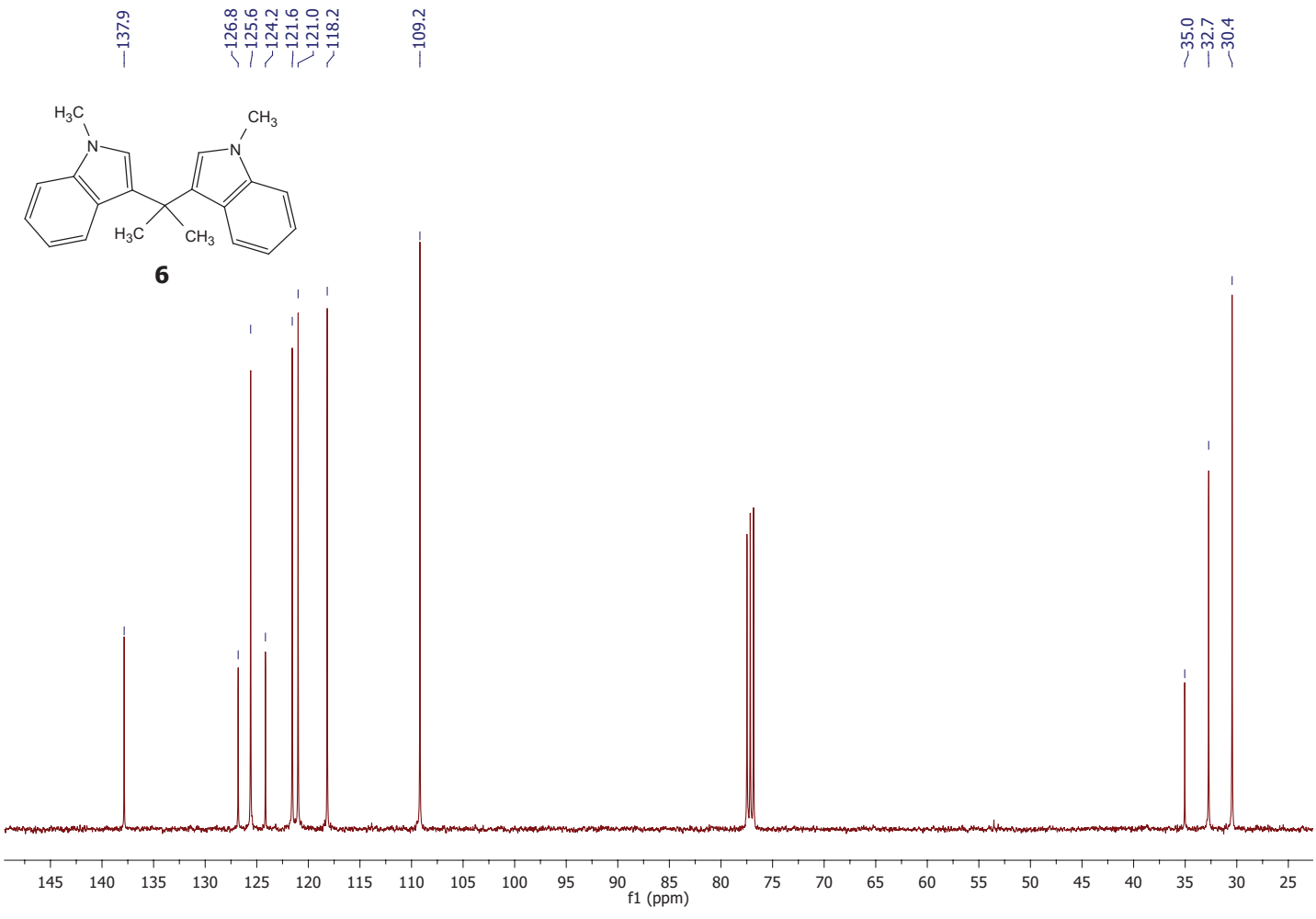

159.1
135.6
134.1
130.8
128.6
122.3
119.8
118.8
114.3
111.1
108.6
83.0
73.1
62.8
55.5
33.9
27.8
HN
OH
CH
H3C
O
5q
f1 (ppm)


7.22
6.81
4.37
4.10
3.76
2.31
2.28
1.89
O
O
H3C
CH3
CH
H3C
O
5r
2.07
2.14
1.00
1.00
3.15
3.17
1.02
3.05
7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 5.8 5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4
f1 (ppm)

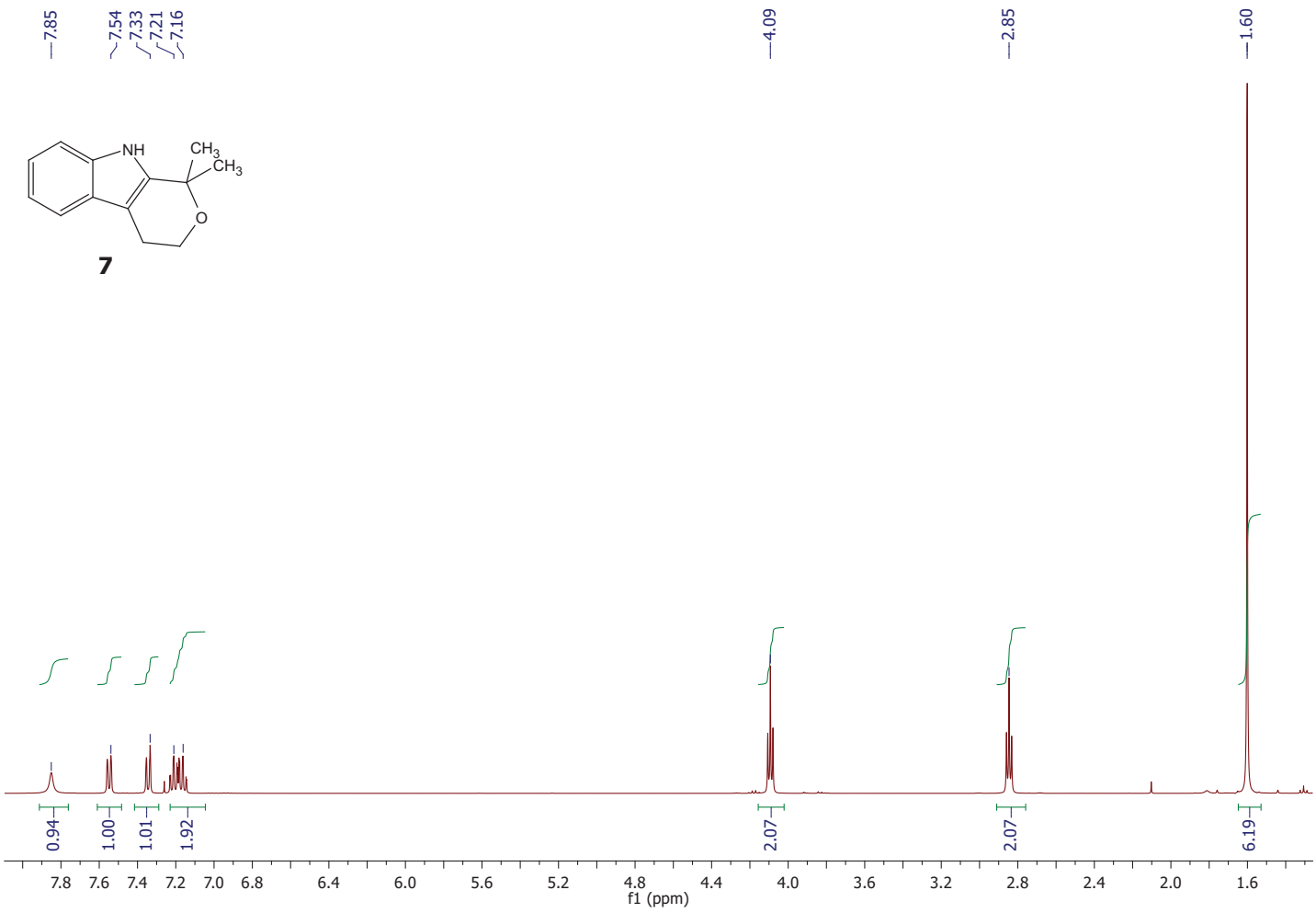

201.5
201.4
159.1
129.1
127.7
114.3
83.2
75.4
72.7
55.3
36.4
31.0
28.9
O
O
H3C
CH3
CH
H3C
O
5r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)


7.51
7.31
7.19
6.97
6.95
3.78
1.98
H3C
N
CH3
H3C
CH3
1.99
2.00
2.03
3.97
6.17
6.18
f1 (ppm)

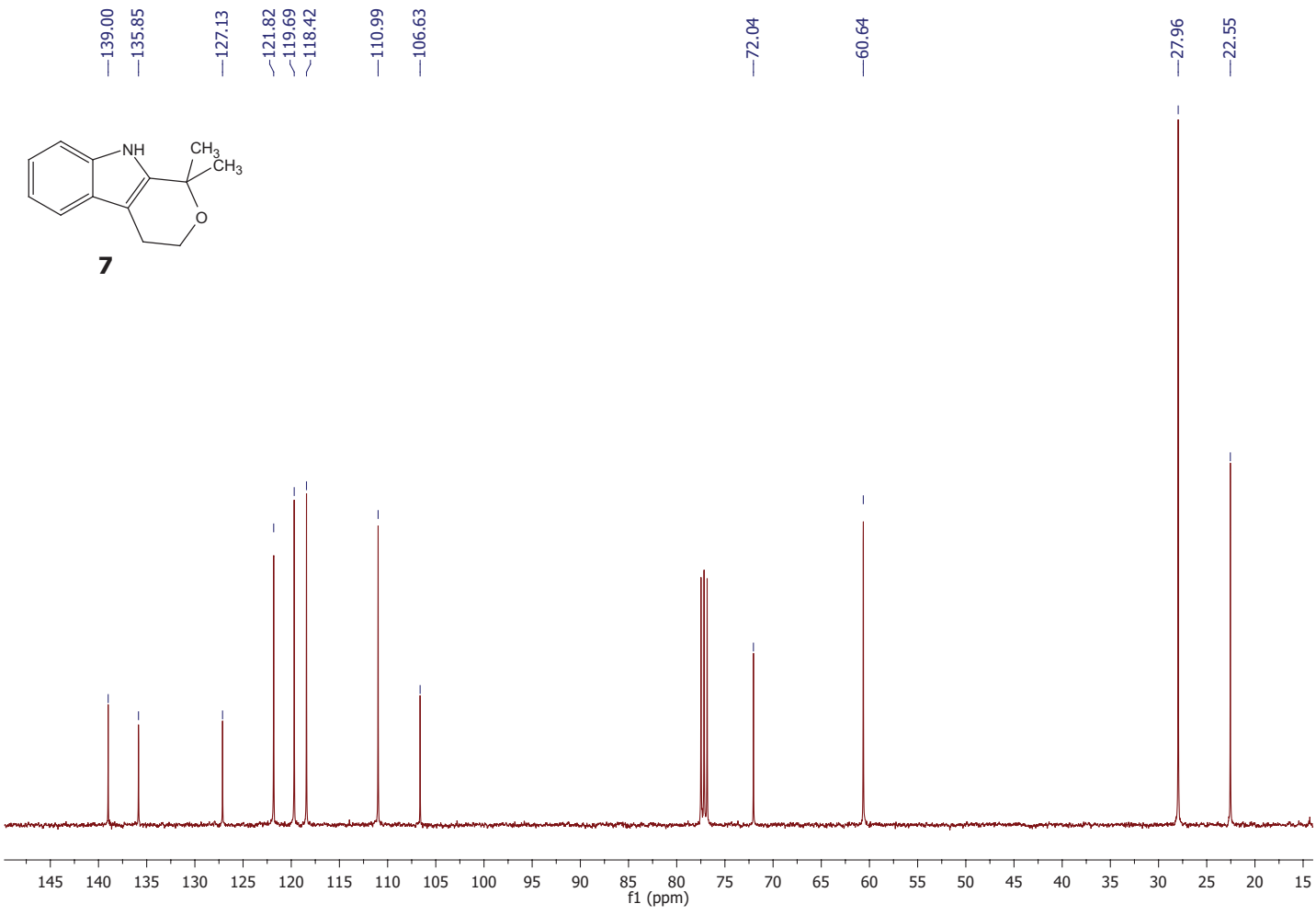
6


137.9
126.8
125.6
124.2
121.6
121.0
118.2
109.2
35.0
32.7
30.4
H3C
N
CH3
H3C
CH3
f1 (ppm)

6


7.85
7.54
7.33
7.21
7.16
4.09
2.85
1.60
NH
CH3
CH3
O
0.94
1.00
1.01
1.92
2.07
2.07
6.19
f1 (ppm)

7


139.00
135.85
127.13
121.82
119.69
118.42
110.99
106.63
72.04
60.64
27.96
22.55
NH
CH3
CH3
O
f1 (ppm)

7