

Supporting Information

Nickel-Catalyzed Hydroarylation of in Situ Generated 1,3-Dienes with Arylboronic Acids Using a Secondary Homoallyl Carbonate as a Surrogate for the 1,3-Diene and Hydride Source

Takashi Hamaguchi, Yoshiyuki Takahashi, Hiroaki Tsuji*, and Motoi Kawatsura*

Department of Chemistry, College of Humanities & Sciences, Nihon University,
Sakurajosui, Setagaya-ku, Tokyo 156-8550, Japan

tsuji@chs.nihon-u.ac.jp

kawatsur@chs.nihon-u.ac.jp

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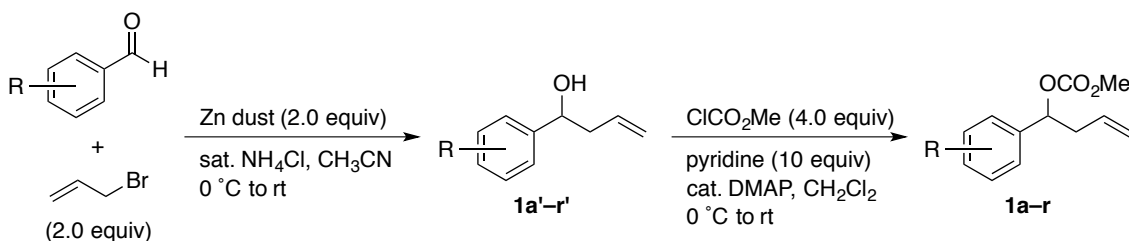
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1. General experimental remarks

Commercially available chemicals were purchased from Aldrich, TCI, Kanto, and Wako and used without further purification unless otherwise noted. Ni(cod)₂ was purchased from Aldrich and stored at -30 °C under N₂. 1-Phenylbutan-1-ol (**8'**) was prepared by the reduction of butyrophenone with NaBH₄. NMR spectra were recorded at 25 °C on a JEOL EX-270 spectrometer (270 MHz for ¹H, 67.9 MHz for ¹³C) or a JEOL JNM ECP-500 spectrometer (126 MHz for ¹³C, 471 MHz for ¹⁹F). Chemical shifts are reported in δ ppm referenced to an internal tetramethylsilane (0 ppm) for ¹H NMR. Chemical shifts of ¹³C NMR are given relative to the solvent peak as an internal standard. ¹⁹F NMR data are reported relative to external α,α,α-trifluorotoluene (-63.7 ppm). Multiplicities are indicated as br (broad), s (singlet), d (doublet), t (triplet), q (quartet), or m (multiplet). Coupling constants (*J*) are reported in Hertz (Hz). Melting points were measured on a Yanako MP-500P. Infrared (IR) spectra were recorded on JASCO FT/IR-460 Plus. GC-MS measurements were carried out with JEOL JMS-Q1000GC/K9. HRMS analyses were carried out using a JEOL AccuTOF LCplus for ESI-MS and APCI-MS, Waters G2-S Q-TOF for ESI-MS, and JEOL GCmate for EI-MS. Column chromatography was conducted with silica gel 60N (KANTO CHEMICAL, spherical, neutral, 40-50 or 63-210 μm). For thin-layer chromatography (TLC) analyses throughout this work, Merck precoated TLC plates (silica gel 60 F254 0.25 mm) were used. Visualization was accomplished by UV light (254 nm) and phosphomolybdic acid. Oil bath was used as the heat source for the reactions that require heating.

2. General procedure for the preparation of starting materials

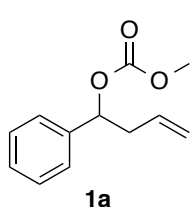
Scheme S1. Preparation of homoallyl carbonates **1a-r**



Preparation of methyl (1-phenylbut-3-en-1-yl) carbonate (**1a**):

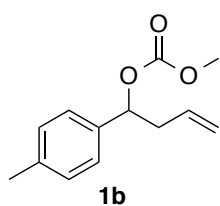
Homoallyl alcohol **1a'** was prepared according to the slightly modified method reported by Gadakh and Sudalai.¹ Benzaldehyde (1.06 g, 10 mmol), allyl bromide (2.42 g, 20

mmol, 1.7 mL), zinc dust (1.3 g, 20 mmol), and CH₃CN (30 mL) were placed in a round flask. Saturated NH₄Cl solution (5 mL) was added to the mixture at 0 °C under air and the reaction mixture was stirred at room temperature for 2 h. The resulting suspension was filtrated with a pad of celite and 1N HCl (15 mL) was added to the filtrate. The resulting aqueous phase was extracted with EtOAc (30 mL × 3). The combined organic phase was washed with brine and dried over MgSO₄. After removal of the solvent, the desired homoallyl alcohol **1a'** was obtained as a crude mixture and the alcohol was used for the next step without further purification. The crude alcohol (741 mg, 5.0 mmol) and catalytic amount of DMAP were charged into a round flask and the flask was refilled with N₂. CH₂Cl₂ (10 mL) and pyridine (3.96 g, 4.0 mL, 50 mmol) were added. Methyl chloroformate (2.42 g, 1.5 mL, 20 mmol) was added dropwise to the resulting mixture at 0 °C and the reaction mixture was stirred at room temperature for 2 h. The reaction was quenched with saturated NH₄Cl solution (10 mL) and the resulting aqueous phase was extracted with CH₂Cl₂ (20 mL × 3). The combined organic phase was washed with brine and dried over MgSO₄. After removal of the solvent, the resulting crude mixture was purified by silica gel column chromatography (Hexane/EtOAc = 95/5) to give methyl (1-phenylbut-3-en-1-yl) carbonate (**1a**) as a colorless oil (915 mg, 4.44 mmol, 89% yield).



IR (neat) 3079, 2956, 2853, 1748, 1643, 1442, 1268, 946, 700 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 2.53–2.63 (m, 1H), 2.67–2.78 (m, 1H), 3.75 (s, 3H), 5.61 (dd, *J* = 7.8, 6.2 Hz, 1H), 5.72 (dddd, *J* = 17.3, 10.3, 6.9, 6.9 Hz 1H), 7.30–7.37 (m, 5H); ¹³C NMR (67.9 MHz, CDCl₃): δ 40.7, 54.6, 79.3, 118.3, 126.4, 128.2, 128.4, 132.8, 139.4, 155.1; HRMS (ESI/TOF) *m/z*: [M+Na]⁺ Calcd for C₁₂H₁₄O₃Na 229.0841; Found 229.0847.

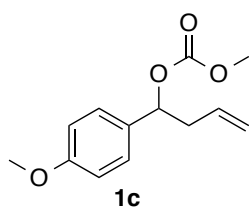
Methyl (1-(*p*-tolyl)but-3-en-1-yl) carbonate (**1b**)²



This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (991 mg, 4.50 mmol, 90% yield); ¹H NMR (270 MHz, CDCl₃): δ 2.33 (s, 3H), 2.51–2.61 (m, 1H), 2.66–2.77 (m, 1H), 3.73 (s, 3H), 5.04–5.14 (m, 2H), 5.58 (dd, *J* = 8.0, 6.1 Hz, 1H), 5.71 (dddd, *J* = 16.9, 10.1, 7.0, 7.0

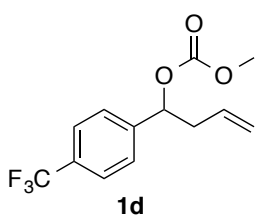
Hz, 1H), 7.16 (d, $J = 8.1$ Hz, 2H), 7.23–7.26 (m, 2H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 20.9, 40.5, 54.4, 79.1, 118.0, 126.3, 129.0, 132.9, 136.4, 137.8, 155.0; MS (ESI) m/z : $[\text{M}+\text{Na}]^+$ 243.

1-(4-Methoxyphenyl)but-3-en-1-yl methyl carbonate (**1c**)



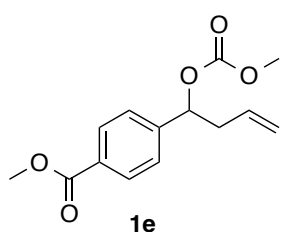
This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (425 mg, 1.80 mmol, 36% yield); IR (neat) 3078, 3005, 2956, 2838, 1747, 1613, 1516, 1248, 1035, 833 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.51–2.61 (m, 1H), 2.67–2.78 (m, 1H), 3.73 (s, 3H), 3.80 (s, 3H), 5.05–5.14 (m, 2H), 5.56 (dd, $J = 7.8, 6.5$ Hz, 1H), 5.71 (dddd, $J = 17.0, 10.0, 6.9, 6.9$ Hz, 1H), 6.86–6.91 (m, 2H), 7.27–7.32 (m, 2H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 40.3, 54.4, 54.9, 78.9, 113.6, 118.0, 127.8, 131.3, 132.9, 155.0, 159.4; HRMS (ESI/TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_4\text{Na}$ 259.0946; Found 259.0949.

Methyl (1-(4-(trifluoromethyl)phenyl)but-3-en-1-yl) carbonate (**1d**)



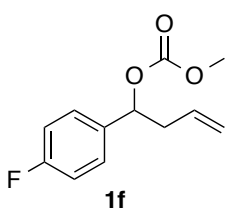
This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (835 mg, 3.04 mmol, 61% yield); IR (neat) 3082, 2960, 2855, 1753, 1623, 1263, 1122, 842 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.52–2.62 (m, 1H), 2.65–2.76 (m, 1H), 3.76 (s 3H), 5.07–5.15 (m, 2H), 5.63–5.78 (m, 2H), 7.46 (d, $J = 8.0$ Hz, 1H), 7.62 (d, $J = 8.0$ Hz, 1H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 40.6, 54.9, 78.5, 118.9, 123.9 (q, $J_{\text{CF}} = 272$ Hz), 125.5 (q, $J_{\text{CF}} = 3.7$ Hz), 126.7, 130.4 (q, $J_{\text{CF}} = 32.6$ Hz), 132.1, 143.4 (2C), 155.0; ^{19}F NMR (471 MHz, CDCl_3): δ -63.6 (s) HRMS (ESI/QTOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{13}\text{F}_3\text{O}_3\text{Na}$ 297.0714; Found 297.0717.

Methyl 4-(1-((methoxycarbonyl)oxy)but-3-en-1-yl)benzoate (**1e**)



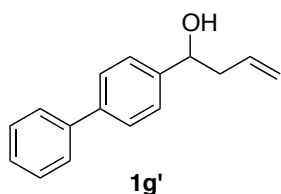
This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (1.00 g, 3.78 mmol, 76% yield); IR (neat) 2955, 2851, 1749, 1614, 1439, 1270, 946, 771 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.53–2.63 (m, 1H), 2.65–2.76 (m, 1H), 3.76 (s, 3H), 3.91 (s, 3H), 5.06–5.13 (m, 2H), 5.63–5.78 (m, 2H), 7.40–7.44 (m, 2H), 8.01–8.06 (m, 2H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 40.6, 52.0, 54.8, 78.6, 118.8, 126.2, 129.8, 129.9, 132.2, 144.4, 155.0, 166.6; HRMS (ESI/TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{16}\text{O}_5\text{Na}$ 287.0895; Found 287.0905.

1-(4-Fluorophenyl)but-3-en-1-yl methyl carbonate (**1f**)



This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (836 mg, 3.73 mmol, 75% yield); IR (neat) 3080, 2958, 2853, 1749, 1607, 1513, 1270, 939, 838, 547 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.50–2.60 (m, 1H), 2.65–2.76 (m, 1H), 3.75 (s, 3H), 5.06–5.13 (m, 2H), 5.56–5.77 (m, 2H), 7.00–7.08 (m, 2H), 7.31–7.37 (m, 2H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 40.6, 54.6, 78.6, 115.3 (d, $J_{\text{CF}} = 21.3$ Hz), 118.5, 128.3 (d, $J_{\text{CF}} = 8.4$ Hz), 132.5, 135.2 (d, $J_{\text{CF}} = 3.3$ Hz), 155.0, 162.5 (d, $J_{\text{CF}} = 247$ Hz); ^{19}F NMR (471 MHz, CDCl_3): δ -114.6 – -114.7 (m) HRMS (ESI/QTOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{12}\text{H}_{13}\text{FO}_3\text{Na}$ 247.0746; Found 247.0747.

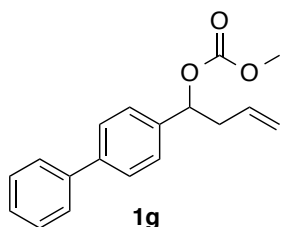
1-([1,1'-Biphenyl]-4-yl)but-3-en-1-ol (**1g'**)³



This compound was prepared according to the similar method to **1a'** and the resulting crude alcohol **1g'** was purified by silica gel column chromatography (Hexane/EtOAc = 85/15). White solid (2.15 g, 9.59 mmol, 96% yield); ^1H NMR (270 MHz, CDCl_3): δ 2.01 (brs, 1H), 2.47–2.64 (m, 2H), 4.79 (dd, $J = 7.4, 5.5$ Hz, 1H), 5.15–5.24 (m, 2H), 5.85 (dddd, $J = 17.3, 10.0, 7.1, 7.1$ Hz, 1H), 7.31–7.37 (m, 1H), 7.40–7.47 (m, 4H), 7.56–7.61 (m, 4H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 43.7,

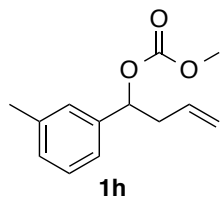
73.0, 118.4, 126.2, 127.0, 127.1, 127.2, 128.7, 134.4, 140.4, 140.8, 142.9; MS (EI) m/z : $[M]^+$ 224.

1-([1,1'-Biphenyl]-4-yl)but-3-en-1-yl methyl carbonate (**1g**)



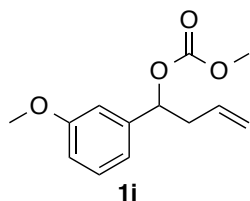
This compound was prepared according to the similar method to **1a** using the isolated alcohol **1g'** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). White solid (1.29 g, 4.57 mmol, 91% yield); M.p. 62.2–63.2 °C; IR (KBr) 3051, 2956, 2851, 1742, 1439, 1281, 980, 762, 689 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.56–2.66 (m, 1H), 2.70–2.81 (m, 1H), 3.76 (s, 3H), 5.08–5.18 (m, 2H), 5.63–5.83 (m, 2H), 7.31–7.37 (m, 1H), 7.41–7.47 (m, 4H), 7.56–7.60 (m, 4H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 40.6, 54.7, 79.1, 118.4, 126.9, 127.1, 127.2, 127.4, 128.7, 132.8, 138.4, 140.6, 141.1, 155.1; HRMS (ESI/TOF) m/z : $[M+\text{Na}]^+$ Calcd for $\text{C}_{18}\text{H}_{18}\text{O}_3\text{Na}$ 305.1154; Found 305.1140.

Methyl (1-(*m*-tolyl)but-3-en-1-yl) carbonate (**1h**)



This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (966 mg, 4.39 mmol, 88% yield); IR (neat) 2956, 1748, 1441, 1267, 924, 790, 703 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.12 (s, 3H), 2.51–2.61 (m, 1H), 2.65–2.76 (m, 1H), 3.74 (s, 3H), 5.06–5.15 (m, 2H), 5.58 (dd, J = 8.1, 5.9 Hz, 1H), 5.72 (dddd, J = 17.3, 10.3, 6.9, 6.9 Hz, 1H), 7.10–7.16 (m, 3H), 7.21–7.27 (m, 1H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 21.3, 40.7, 54.6, 79.4, 118.2, 123.5, 127.0, 128.3, 128.9, 133.0, 138.1, 139.4, 155.1; HRMS (ESI/TOF) m/z : $[M+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_3\text{Na}$ 243.0997; Found 243.1005.

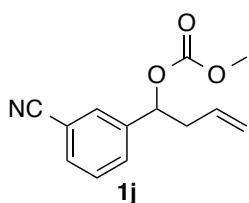
1-(3-Methoxyphenyl)but-3-en-1-yl methyl carbonate (**1i**)



This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (923 mg, 3.91 mmol, 78% yield); IR (neat) 3077, 2956, 2840,

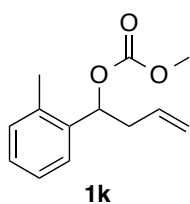
1741, 1604, 1442, 1240, 1029, 939, 756 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.51–2.62 (m, 1H), 2.64–2.75 (m, 1H), 3.76 (s, 3H), 3.81 (s, 3H), 5.06–5.15 (m, 2H), 5.58 (dd, $J = 7.8, 5.9$ Hz, 1H), 5.72 (dddd, $J = 17.0, 10.3, 6.9, 6.9$ Hz, 1H), 6.82–6.95 (m, 3H), 7.24–7.30 (m, 1H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 40.7, 54.7, 55.2, 79.2, 111.9, 113.6, 118.3, 118.7, 129.5, 132.9, 141.1, 155.1, 159.6; HRMS (ESI/TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_4\text{Na}$ 259.0946; Found 259.0947.

1-(3-Cyanophenyl)but-3-en-1-yl methyl carbonate (**1j**)



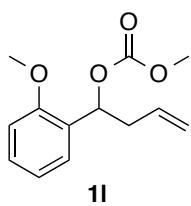
This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 90/10). Colorless oil (966 mg, 4.18 mmol, 82% yield); IR (neat) 3080, 2958, 2853, 2231, 1751, 1443, 1267, 930, 792, 694 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.51–2.62 (m, 1H), 2.65–2.75 (m, 1H), 3.77 (s, 3H), 5.06–5.13 (m, 2H), 5.59–5.76 (m, 2H), 7.45–7.51 (m, 1H), 7.56–7.65 (m, 3H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 40.4, 54.9, 78.0, 112.7, 118.4, 119.2, 129.3, 130.0, 130.8, 131.8 (2C), 141.0, 154.9; HRMS (ESI/TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{13}\text{NO}_3\text{Na}$ 254.0793; Found 254.0794.

Methyl (1-(*o*-tolyl)but-3-en-1-yl) carbonate (**1k**)



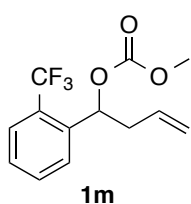
This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (805 mg, 3.65 mmol, 73% yield); IR (neat) 2956, 1748, 1442, 1273, 942, 759 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.40 (s, 3H), 2.50–2.58 (m, 1H), 2.62–2.73 (m, 1H), 3.73 (s, 3H), 5.06–5.16 (m, 2H), 5.74 (dddd, $J = 17.0, 10.0, 7.0, 7.0$ Hz, 1H), 5.85 (dd, $J = 8.1, 5.7$ Hz, 1H), 7.12–7.24 (m, 3H), 7.36–7.40 (m, 1H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 19.1, 40.2, 54.6, 75.9, 118.2, 125.6, 126.2, 127.8, 130.3, 132.9, 134.8, 138.0, 155.1; HRMS (ESI/TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_3\text{Na}$ 243.0997; Found 243.1006.

1-(2-Methoxyphenyl)but-3-en-1-yl methyl carbonate (**1l**)



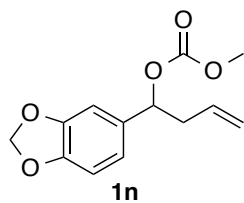
This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 90/10). Colorless oil (937 mg, 3.97 mmol, 79% yield); IR (neat) 3077, 2956, 2840, 1741, 1604, 1494, 1291, 1029, 980, 756 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.58–2.63 (m, 2H), 3.75 (s, 3H), 3.85 (s, 3H), 5.03–5.13 (m, 2H), 5.79 (dddd, $J = 17.0, 10.3, 6.9, 6.9$ Hz, 1H), 6.08 (t, $J = 6.5$ Hz, 1H), 6.88 (d, $J = 8.1$ Hz, 1H), 6.93–6.98 (m, 1H), 7.23–7.29 (m, 1H), 7.34 (dd, $J = 7.7, 1.8$ Hz, 1H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 39.6, 54.6, 55.4, 73.8, 110.5, 117.7, 120.5, 126.2, 128.2, 128.8, 133.4, 155.1, 156.0; HRMS (ESI/TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_4\text{Na}$ 259.09463; Found 259.09460.

Methyl (1-(2-(trifluoromethyl)phenyl)but-3-en-1-yl) carbonate (**1m**)



This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (855 mg, 3.12 mmol, 62% yield); IR (neat) 3082, 2959, 2856, 1754, 1443, 1273, 1165, 947, 770 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.58 (dd, $J = 6.6, 6.6$ Hz, 2H), 3.75 (s, 3H), 5.10–5.17 (m, 2H), 5.84 (dddd, $J = 16.7, 10.5, 6.9, 6.9$ Hz, 1H), 6.03 (t, $J = 6.2$ Hz, 1H), 7.38–7.44 (m, 1H), 7.55–7.60 (m, 1H), 7.63–7.68 (m, 2H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 41.4, 54.9, 75.1, 118.6, 124.1 (q, $J_{\text{CF}} = 274$ Hz), 125.8 (q, $J_{\text{CF}} = 5.8$ Hz), 127.0 (q, $J_{\text{CF}} = 30.7$ Hz), 127.2, 128.0, 132.2, 132.7, 139.1, 154.8; ^{19}F NMR (471 MHz, CDCl_3): δ -59.4 (s) HRMS (ESI/QTOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{13}\text{F}_3\text{O}_3\text{Na}$ 297.0714; Found 297.0717.

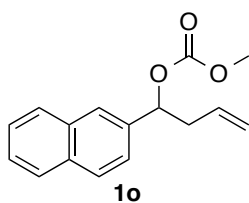
1-(Benzo[d][1,3]dioxol-5-yl)but-3-en-1-yl methyl carbonate (**1n**)



This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (873 mg, 3.49 mmol, 70% yield); IR (neat) 3078, 2957, 2903, 2781, 1749, 1490, 1244, 1043, 920, 813 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.48–2.59 (m, 1H), 2.63–2.74 (m, 1H), 3.74 (s, 3H), 5.05–5.15 (m, 2H), 5.51 (dd, $J = 7.8, 6.2$ Hz, 1H), 5.70 (dddd, $J = 17.0, 10.3, 7.0, 7.0$ Hz, 1H), 6.76–6.79 (m,

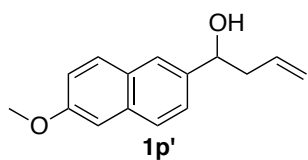
1H), 6.81–6.86 (m, 2H); ¹³C NMR (67.9 MHz, CDCl₃): δ 40.6, 54.6, 79.2, 101.1, 106.9, 108.1, 118.3, 120.4, 132.8, 133.2, 147.5, 147.7, 155.0; HRMS (ESI/TOF) *m/z*: [M+Na]⁺ Calcd for C₁₃H₁₄O₅Na 273.0739; Found 273.0741.

Methyl (1-(naphthalen-2-yl)but-3-en-1-yl) carbonate (**1o**)



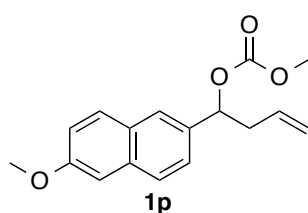
This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 90/10). Pale red oil (1.11 g, 4.33 mmol, 87% yield); IR (neat) 3058, 2955, 2852, 1748, 1441, 1266, 941, 792, 748, 479 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 2.62–2.72 (m, 1H), 2.76–2.87 (m, 1H), 3.75 (s, 3H), 5.06–5.16 (m, 2H), 5.67–5.82 (m, 2H), 7.46–7.52 (m, 3H), 7.80–7.86 (m, 4H); ¹³C NMR (67.9 MHz, CDCl₃): δ 40.7, 54.7, 79.5, 118.5, 124.0, 125.8, 126.2 (2C), 127.7, 128.1, 128.4, 132.8, 133.0, 133.2, 136.8, 155.1; HRMS (ESI/TOF) *m/z*: [M+Na]⁺ Calcd for C₁₆H₁₆O₃Na 279.0997; Found 279.0998.

1-(6-Methoxynaphthalen-2-yl)but-3-en-1-ol (**1p'**)⁴



This compound was prepared according to the similar method to **1a'** and the resulting crude alcohol **1p'** was purified by silica gel column chromatography (Hexane/EtOAc = 95/5). White solid (1.59 g, 6.97 mmol, 70% yield); ¹H NMR (270 MHz, CDCl₃): δ 2.05 (brs, 1H), 2.51–2.64 (m, 2H), 3.91 (s, 3H), 4.86 (dd, *J* = 6.5, 6.5 Hz, 1H), 5.11–5.22 (m, 2H), 5.83 (dddd, *J* = 17.4, 10.1, 7.2, 7.2 Hz, 1H), 7.13–7.17 (m, 2H), 7.44 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.70–7.74 (m, 3H); ¹³C NMR (67.9 MHz, CDCl₃): δ 43.6, 55.2, 73.4, 105.6, 118.3, 118.9, 124.4, 124.5, 127.0, 128.6, 129.4, 134.0, 134.5, 139.0, 157.6; MS (EI) *m/z*: [M]⁺ 228.

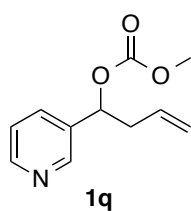
1-(6-Methoxynaphthalen-2-yl)but-3-en-1-yl methyl carbonate (**1p**)



This compound was prepared according to the similar method to **1a** using the isolated alcohol **1p'** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). White solid (1.35

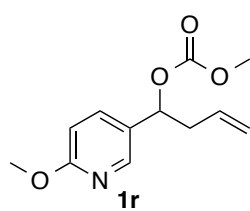
g, 4.71 mmol, 94% yield); M.p. 71.0–72.2 °C; IR (KBr) 3066, 2960, 2844, 1736, 1607, 1444, 1281, 1027, 819, 794, 480 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 2.61–2.71 (m, 1H), 2.75–2.86 (m, 1H), 3.75 (s, 3H), 3.92 (s, 3H), 5.05–5.16 (m, 2H), 5.66–5.82 (m, 2H), 7.12–7.17 (m, 2H), 7.44 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.72–7.75 (m, 3H); ¹³C NMR (67.9 MHz, CDCl₃): δ 40.6, 54.7, 55.3, 79.6, 105.6, 118.4, 119.1, 124.7, 125.7, 127.2, 128.5, 129.6, 132.9, 134.4 (2C), 155.2, 157.9; HRMS (ESI/TOF) *m/z*: [M+Na]⁺ Calcd for C₁₇H₁₈O₄Na 309.1103; Found 309.1101.

Methyl (1-(pyridin-3-yl)but-3-en-1-yl) carbonate (**1q**)



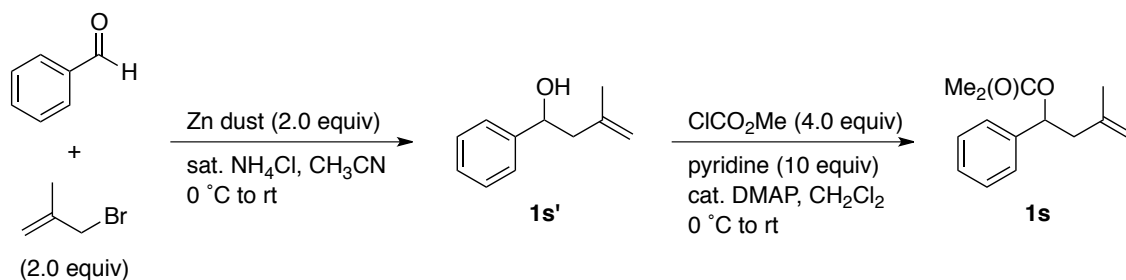
This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 50/50). Yellow oil (340 mg, 1.64 mmol, 26% yield); IR (neat) 2957, 2854, 1748, 1443, 1257, 943, 792, 714cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 2.55–2.65 (m, 1H), 2.69–2.81 (m, 1H), 3.76 (s, 3H), 5.07–5.15 (m, 2H), 5.63–5.79 (m, 2H), 7.34 (dd, *J* = 7.8, 4.9 Hz, 1H), 7.72 (ddd, *J* = 7.8, 1.8, 1.8 Hz, 1H), 8.59–8.64 (m, 2H); ¹³C NMR (67.9 MHz, CDCl₃): δ 40.3, 54.8, 76.9, 119.1, 123.3, 131.9, 134.0, 134.8, 148.1, 149.5, 154.8; HRMS (ESI/TOF) *m/z*: [M+H]⁺ Calcd for C₁₁H₁₄NO₃ 208.0974; Found 208.0967.

1-(6-Methoxypyridin-3-yl)but-3-en-1-yl methyl carbonate (**1r**)



This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 80/20). Colorless oil (161 mg, 0.68 mmol, 76% yield); IR (neat) 3080, 2954, 2852, 1748, 1610, 1496, 1341, 1259, 1025, 930, 834, 791 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 2.51–2.62 (m, 1H), 2.69–2.80 (m, 1H), 3.75 (s, 3H), 3.94 (s, 3H), 5.07–5.15 (m, 2H), 5.58 (dd, *J* = 7.2, 7.2 Hz, 1H), 5.70 (dddd, *J* = 17.0, 10.3, 6.9, 6.9 Hz, 1H), 6.75 (d, *J* = 8.4 Hz, 1H), 7.59 (dd, *J* = 8.4, 2.4 Hz, 1H), 8.16 (d, *J* = 2.4 Hz, 1H); ¹³C NMR (67.9 MHz, CDCl₃): δ 40.0, 53.5, 54.7, 76.9, 110.9, 118.8, 127.6, 132.3, 137.1, 145.7, 155.0, 164.1; HRMS (ESI/TOF) *m/z*: [M+H]⁺ Calcd for C₁₂H₁₆NO₄ 238.1079; Found 238.1084.

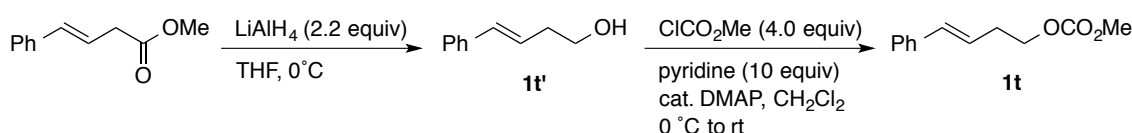
Scheme S2. Preparation of homoallyl carbonate **1s**



Preparation of methyl(3-methyl-1-phenylbut-3-en-1-yl) carbonate (**1s**)

This compound was prepared according to the similar method to **1a** using 3-bromo-2-methylpropene instead of allyl bromide and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (810 mg, 3.68 mmol, 72% yield); IR (neat) 3034, 2957, 2853, 1748, 1651, 1442, 1269, 938, 792, 761, 699 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 1.75 (s, 3H), 2.45 (dd, $J = 14.3, 5.7$ Hz, 1H), 2.71 (dd, $J = 14.2, 8.6$ Hz, 1H), 3.73 (s, 3H), 4.74–4.82 (m, 2H), 5.74 (dd, $J = 8.6, 5.7$ Hz, 1H), 7.27–7.39 (m, 5H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 22.5, 44.7, 54.6, 78.2, 113.9, 126.4, 128.1, 128.4, 139.8, 140.5, 155.1; HRMS (ESI/TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_3\text{Na}$ 243.0997; Found 243.1002.

Scheme S3. Preparation of homoallyl carbonate (**1t**)

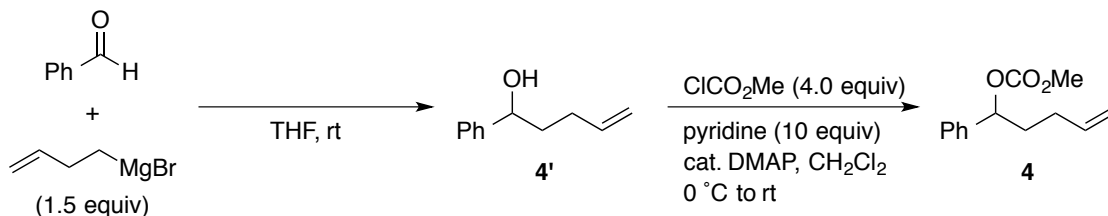


Preparation of (*E*)-methyl (4-phenylbut-3-en-1-yl) carbonate (**1t**)

(*E*)-4-Phenylbut-3-en-1-ol (**1t'**) was prepared according to the literature procedure⁵ and ^1H NMR data was consistent with the reported value.⁶ (*E*)-Methyl (4-phenylbut-3-en-1-yl) carbonate (**1t**) was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 90/10). Yellow oil (210 mg, 1.02 mmol, 82% yield); IR (neat) 3027, 2957, 2857, 1748, 1443, 1270, 966, 793, 745, 694 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 2.59 (dtd, $J = 6.9, 6.9, 1.4$ Hz, 2H), 3.78 (s, 3H), 4.26 (t, $J = 6.9$ Hz, 2H), 6.18 (dt, $J = 16.1, 6.9$ Hz, 1H), 6.49 (d, $J = 16.1$ Hz, 1H), 7.16–7.37 (m, 5H); ^{13}C NMR (67.9MHz, CDCl_3): δ 32.4, 54.7, 67.2, 124.9, 126.1, 127.3, 128.5, 132.7,

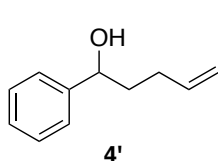
137.1, 155.7; HRMS (ESI/QTOF) m/z : $[M+Na]^+$ Calcd for $C_{12}H_{14}O_3Na$ 229.0841; Found 229.0841.

Scheme S4. Preparation of methyl (1-phenylpent-4-en-1-yl) carbonate (**4**)



Preparation of 1-phenylpent-4-en-1-ol (**4'**):

Magnesium turnings (437 mg, 18 mmol) was charged into a two-necked round flask equipped with a condenser and a dropping funnel and the flask was refilled with N_2 . After addition of a small amount of THF, a solution of 4-bromo-1-butene (2.02 g, 1.5 mL, 15 mmol) in THF (12 mL) was added dropwise to the mixture. The reaction mixture was cooled to room temperature and a solution of benzaldehyde (1.06 g, 1.0 mL, 10 mmol) in THF (10 mL) was added to the mixture. The resulting mixture was stirred at room temperature overnight. The reaction was quenched with 1N HCl (20 mL) and the resulting aqueous phase was extracted with EtOAc. The combined organic phase was washed with brine and dried over $MgSO_4$. After removal of the solvent, the resulting crude mixture was purified by silica gel column chromatography (Hexane/EtOAc = 9/1, 4/1) to give 1-phenylpent-4-en-1-ol (**4'**)^{7a,b} as a slightly yellow oil (1.39 g, 8.57 mmol, 86% yield).

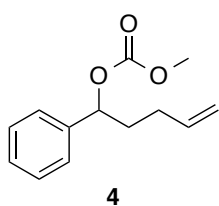


1H NMR (270 MHz, $CDCl_3$): δ 1.73–1.97 (m, 3H), 2.02–2.25 (m, 2H), 4.69 (dd, J = 7.6, 5.7 Hz, 1H), 4.95–5.08 (m, 2H), 5.84 (ddm, J = 17.0, 10.3 Hz, 1H), 7.25–7.36 (m, 5H); ^{13}C NMR (67.9 MHz, $CDCl_3$): δ 30.0, 38.0, 73.9, 114.9, 125.8, 127.5, 128.4, 138.1, 144.5;

MS (EI) m/z : $[M]^+$ 162.

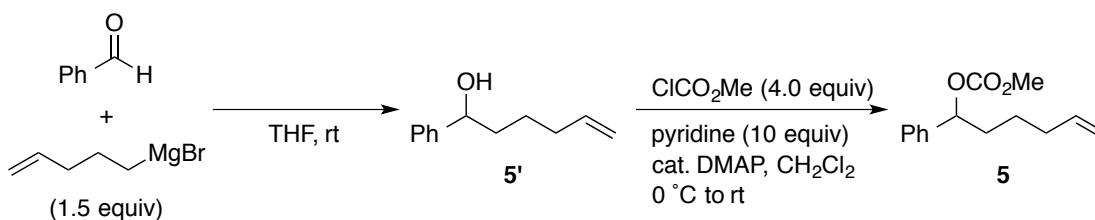
Preparation of methyl (1-phenylpent-4-en-1-yl) carbonate (**4**):

This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 19/1).



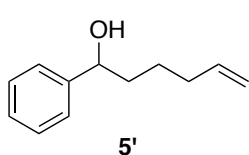
Colorless oil (474 mg, 2.15 mmol, 86% yield). IR (neat) 3033, 2955, 2852, 1748, 1642, 1442, 1265, 938, 792, 762, 700 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 1.80–1.96 (m, 1H), 2.02–2.16 (m, 3H), 3.74 (s, 3H), 4.98–5.06 (m, 2H), 5.57 (dd, $J = 7.2, 5.8$ Hz, 1H), 5.80 (ddm, $J = 16.7, 10.5$ Hz, 1H), 7.28–7.40 (m, 5H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 29.5, 35.4, 54.6, 79.6, 115.4, 126.4, 128.2, 128.5, 137.2, 139.9, 155.2; HRMS (ESI/TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{16}\text{O}_3\text{Na}$ 243.0997; Found 243.0999.

Scheme S5. Preparation of methyl (1-phenylhex-5-en-1-yl) carbonate (5)



Preparation of 1-phenylhex-5-en-1-ol (**5'**):

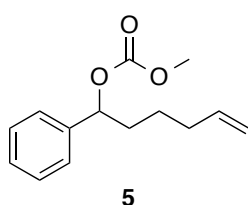
Magnesium turnings (437 mg, 18 mmol) was charged into a two-necked round flask equipped with a condenser and a dropping funnel and the flask was refilled with N_2 . After addition of a small amount of THF, a solution of 5-bromo-1-pentene (2.23 g, 1.8 mL, 15 mmol) in THF (14 mL) was added dropwise to the mixture. The reaction mixture was cooled to room temperature and a solution of benzaldehyde (1.06 g, 1.0 mL, 10 mmol) in THF (10 mL) was added to the mixture. The resulting mixture was stirred at room temperature overnight. The reaction was quenched with 1N HCl (30 mL) and the resulting aqueous phase was extracted with EtOAc (30 mL \times 3). The combined organic phase was washed with brine, dried over MgSO_4 . After removal of the solvent, the resulting crude mixture was purified by silica gel column chromatography (Hexane/EtOAc = 9/1, 4/1) to give 1-phenylhex-5-en-1-ol (**5'**)^{7b,c} as a colorless oil (1.38 g, 7.83 mmol, 78% yield).



^1H NMR (270 MHz, CDCl_3): δ 1.29–1.61 (m, 2H), 1.65–1.88 (m, 3H), 2.03–2.11 (m, 2H), 4.66 (dd, $J = 7.3, 5.9$ Hz, 1H), 4.91–5.03 (m, 2H), 5.78 (ddm, $J = 17.3, 10.3$ Hz, 1H), 7.23–7.30 (m, 1H), 7.31–7.38 (m, 4H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 25.0, 33.5, 38.4, 74.4, 114.6, 125.8, 127.4, 128.3, 138.5, 144.8; MS (EI) m/z : $[\text{M}]^+$ 176.

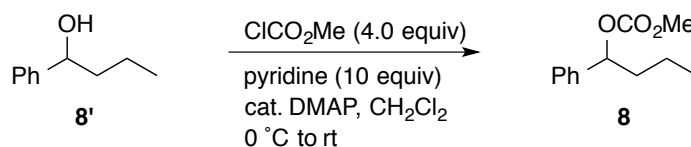
Preparation of methyl (1-phenylhex-5-en-1-yl) carbonate (**5**):

This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 19/1, 9/1).

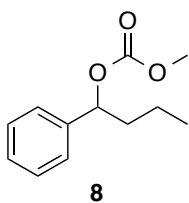


Colorless oil (980 mg, 4.18 mmol, 91% yield); IR (neat) 3033, 2952, 2862, 1748, 1640, 1442, 1265, 944, 792, 764, 700 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 1.28–1.57 (m, 2H), 1.73–1.87 (m, 1H), 1.91–2.11 (m, 3H), 3.74 (s, 3H), 4.92–5.03 (m, 2H), 5.55 (dd, $J = 7.8, 5.9$ Hz, 1H), 5.75 (ddm, $J = 16.7, 10.3$ Hz, 1H), 7.27–7.40 (m, 5H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 24.5, 33.2, 35.6, 54.5, 80.1, 114.9, 126.3, 128.0, 128.4, 138.0, 140.0, 155.2; HRMS (ESI/TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{18}\text{O}_3\text{Na}$ 257.1154; Found 257.1152.

Scheme S6. Preparation of methyl (1-phenylbutyl) carbonate (**8**)



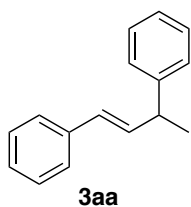
This compound was prepared according to the similar method to **1a** and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 9/1).



Colorless oil (957 mg, 4.60 mmol, 90% yield); IR (neat) 3033, 2959, 2874, 1748, 1442, 1268, 939, 793, 765, 700 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 0.93 (t, $J = 7.4$ Hz, 3H), 1.20–1.47 (m, 2H), 1.70–1.83 (m, 1H), 1.90–2.03 (m, 1H), 3.74 (s, 3H), 5.56 (dd, $J = 7.8, 6.2$ Hz, 1H), 7.27–7.39 (m, 5H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 13.6, 18.6, 38.3, 54.5, 80.0, 126.3, 128.0, 128.3, 140.1, 155.2; HRMS (ESI/TOF) m/z : $[\text{M}+\text{Na}]^+$ Calcd for $\text{C}_{12}\text{H}_{16}\text{O}_3\text{Na}$ 231.0997; Found 231.1008.

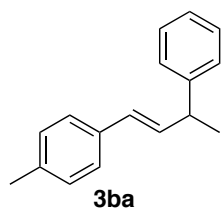
3. General procedure for the nickel-catalyzed hydroarylation of in situ generated 1,3-dienes with arylboronic acids

Methyl (1-phenylbut-3-en-1-yl) carbonate (**1a**) (51.5 mg, 0.25 mmol), P(*p*-MeOC₆H₄)₃ (17.6 mg, 0.05 mmol, 0.2 equiv), PhB(OH)₂ (49.0 mg, 0.38 mmol, 1.5 equiv), and K₂CO₃ (52.0 mg, 0.38 mmol, 1.5 equiv) were charged into a screw cap vial and the vial was taken into a glove box. In the glove box, Ni(cod)₂ (6.9 mg, 0.025 mmol, 0.1 equiv) and CH₃CN (1.2 mL) were added to the vial and the reaction vial was taken from the glove box. The resulting mixture was stirred at 60 °C for 36 h. The reaction mixture was allowed to cool to room temperature and the reaction was quenched with water (1 mL). The resulting aqueous phase was extracted with EtOAc (1 mL × 1). The organic phase was washed with brine and dried over MgSO₄. After removal of the solvent, the crude reaction mixture was purified by silica gel column chromatography to give (*E*)-but-1-ene-1,3-diylidibenzene (**3aa**)⁸ as a colorless oil (49.1 mg, 0.236 mmol, 94% yield as a mixture with a trace amount of *Z* isomer).



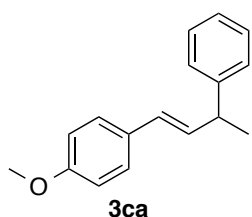
¹H NMR (270 MHz, CDCl₃): δ 1.47 (d, *J* = 6.8 Hz, 3H), 3.59–3.69 (m, 1H), 6.33–6.46 (m, 2H), 7.16–7.38 (m, 10H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.2, 42.5, 126.1, 126.2, 127.0, 127.3, 128.5 (2C), 135.2, 137.5, 145.6, one peak was not found probably due to overlapping; MS (EI) *m/z*: [M]⁺ 208.

(*E*)-1-Methyl-4-(3-phenylbut-1-en-1-yl)benzene (**3ba**)⁹



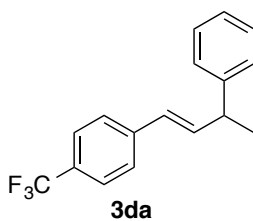
This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 98/2). Colorless oil (49.3 mg, 0.222 mmol, 89% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.46 (d, *J* = 6.8 Hz, 3H), 2.32 (s, 3H), 3.58–3.67 (m, 1H), 6.32 (dd, *J* = 15.8, 5.4 Hz, 1H), 6.40 (d, *J* = 15.8 Hz, 1H), 7.09 (d, *J* = 7.8 Hz, 2H), 7.14–7.30 (m, 6H), 7.31–7.35 (m, 1H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.1, 21.2, 42.5, 126.0, 126.1, 127.3, 128.3, 128.4, 129.2, 134.2, 134.8, 136.7, 145.8; MS (EI) *m/z*: [M]⁺ 222.

(*E*)-1-Methoxy-4-(3-phenylbut-1-en-1-yl)benzene (**3ca**)¹⁰



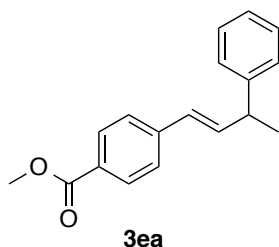
This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 98/2). Colorless oil (54.8 mg, 0.230 mmol, 92% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.45 (d, *J* = 6.8 Hz, 3H), 3.62 (dq, *J* = 6.8, 6.8 Hz, 1H), 3.79 (s, 3H), 6.24 (dd, *J* = 16.1, 6.8 Hz, 1H), 6.36 (d, *J* = 16.1 Hz, 1H), 6.80–6.85 (m, 2H), 7.17–7.23 (m, 1H), 7.25–7.35 (m, 6H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.3, 42.5, 55.2, 113.9, 126.1, 127.2, 127.3, 127.8, 128.4, 130.3, 133.1, 145.9, 158.8; MS (EI) *m/z*: [M]⁺ 238.

(*E*)-1-(3-Phenylbut-1-en-1-yl)-4-(trifluoromethyl)benzene (**3da**)¹¹



This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 98/2). Colorless oil (64.2 mg, 0.232 mmol, 93% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.48 (d, *J* = 6.4 Hz, 3H), 3.67 (dq, *J* = 6.4, 6.4 Hz, 1H), 6.42 (d, *J* = 15.7, Hz, 1H), 6.50 (dd, *J* = 15.7, 6.4 Hz, 1H), 7.20–7.37 (m, 5H), 7.43 (d, *J* = 8.4 Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.0, 42.6, 124.3 (q, *J*_{CF} = 272 Hz), 125.4 (q, *J*_{CF} = 3.9 Hz), 126.3, 126.4, 127.3 (2C), 128.6, 128.8 (q, *J*_{CF} = 32.4 Hz), 138.0, 141.0, 145.0; ¹⁹F NMR (471 MHz, CDCl₃): δ -63.4 (s); MS (EI) *m/z*: [M]⁺ 276.

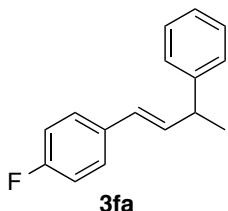
(*E*)-Methyl 4-(3-phenylbut-1-en-1-yl)benzoate (**3ea**)



This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (55.5 mg, 0.208 mmol, 83% yield as a mixture with a trace amount of *Z* isomer); IR (neat) 2965, 1717, 1606, 1435, 1280, 1178, 1110, 759, 700 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 1.45 (d, *J* = 6.7 Hz, 3H), 3.62 (dq, *J* = 6.7, 6.7 Hz, 1H), 3.79 (s, 3H), 6.24 (dd, *J* = 16.1, 6.7 Hz, 1H), 6.36 (d, *J* = 16.1 Hz, 1H), 6.80–6.86 (m, 2H), 7.17–7.23 (m, 1H), 7.26–7.35 (m, 6H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.0,

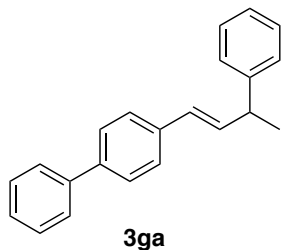
42.6, 51.9, 126.0, 126.4, 127.2, 127.7, 128.4, 128.5, 129.8, 138.0, 142.1, 145.0, 166.9; HRMS (APCI/TOF) m/z : $[M+H]^+$ Calcd for $C_{18}H_{19}O_2$ 267.1385; Found 267.1381.

(*E*)-1-Fluoro-4-(3-phenylbut-1-en-1-yl)benzene (**3fa**)¹²



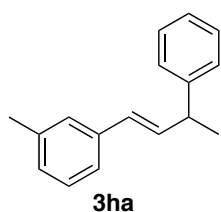
This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (49.3 mg, 0.218 mmol, 87% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.46 (d, *J* = 6.5 Hz, 3H), 3.62 (dq, *J* = 6.5, 6.5 Hz, 1H), 6.28 (dd, *J* = 15.8, 6.5 Hz, 1H), 6.38 (d, *J* = 15.8 Hz, 1H), 6.94–7.01 (m, 2H), 7.19–7.35 (m, 7H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.2, 42.5, 115.3 (d, *J*_{CF} = 21.2 Hz), 126.3, 127.3 (2C), 127.5 (d, *J*_{CF} = 7.8 Hz), 128.5, 133.7 (d, *J*_{CF} = 3.9 Hz), 135.0 (d, *J*_{CF} = 2.2 Hz), 145.5, 162.0 (d, *J*_{CF} = 246 Hz); ¹⁹F NMR (471 MHz, CDCl₃): δ -116.4 (brs); MS (EI) m/z : $[M]^+$ 226.

(*E*)-4-(3-Phenylbut-1-en-1-yl)-1,1'-biphenyl (**3ga**)



This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 98/2). White solid (63.6 mg, 0.224 mmol, 89% yield); M.p. 81.5–83.4 °C; IR (KBr) 3023, 2957, 1491, 962, 817, 699 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 1.48 (d, *J* = 6.8 Hz, 3H), 3.56–3.75 (m, 1H), 6.38–6.50 (m, 2H), 7.19–7.36 (m, 6H), 7.39–7.45 (m, 4H), 7.51–7.60 (m, 4H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.2, 42.6, 126.2, 126.5, 126.9, 127.1, 127.3, 128.0, 128.5, 128.7, 135.4, 136.6, 139.7, 140.7, 145.5, one peak was not found probably due to overlapping; HRMS (EI) m/z : $[M]^+$ Calcd for $C_{22}H_{20}$ 284.1565; Found 284.1566.

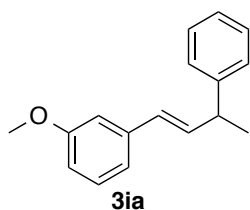
(*E*)-1-Methyl-3-(3-phenylbut-1-en-1-yl)benzene (**3ha**)¹³



This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (50.7

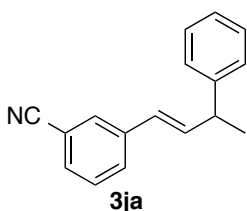
mg, 0.228 mmol, 91% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.46 (d, *J* = 7.3 Hz, 3H), 2.32 (s, 3H), 3.59–3.67 (m, 1H), 6.31–6.43 (m, 2H), 7.00–7.02 (m, 1H), 7.14–7.35 (m, 8H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.2, 21.4, 42.5, 123.3, 126.2, 126.8, 127.3, 127.8, 128.4 (2C), 128.5., 135.0, 137.5, 138.0, 145.7; MS (EI) *m/z*: [M]⁺ 222.

(*E*)-1-Methoxy-3-(3-phenylbut-1-en-1-yl)benzene (**3ia**)



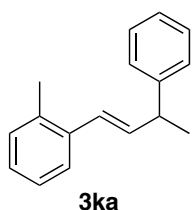
This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 98/2). Colorless oil (50.9 mg, 0.214 mmol, 86% yield as a mixture with a trace amount of *Z* isomer); IR (neat) 3026, 2963, 2834, 1599, 1491, 1263, 1156, 1047, 969, 761, 700 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 1.47 (d, *J* = 6.8 Hz, 3H), 3.60–3.69 (m, 1H), 3.80 (s, 3H), 6.32–6.45 (m, 2H), 6.76 (dd, *J* = 8.2, 2.6 Hz, 1H), 6.89–6.90 (m, 1H), 6.95 (d, *J* = 7.6 Hz, 1H) 7.18–7.24 (m, 2H), 7.28–7.35 (m, 4H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.1, 42.5, 55.1, 111.3, 112.8, 118.8, 126.2, 127.3, 128.4, 128.5, 129.4, 135.5, 139.0, 145.5, 159.8; HRMS (EI) *m/z*: [M]⁺ Calcd for C₁₇H₁₈O 238.1358; Found 238.1356.

(*E*)-3-(3-Phenylbut-1-en-1-yl)benzonitrile (**3ja**)



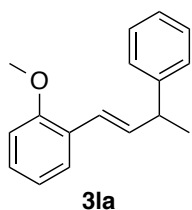
This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 80/20). Colorless oil (50.5 mg, 0.216 mmol, 86% yield as a mixture with a trace amount of *Z* isomer); IR (neat) 3027, 2967, 2871, 2230, 1599, 1493, 1451, 966, 795, 762, 701, 686 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 1.48 (d, *J* = 6.7 Hz, 3H), 3.66 (dq, *J* = 6.7, 6.7 Hz, 1H), 6.37 (d, *J* = 16.1 Hz, 1H), 6.47 (dd, *J* = 16.1, 6.7 Hz, 1H), 7.20–7.27 (m, 3H), 7.31–7.40 (m, 3H), 7.47 (ddd, *J* = 7.8, 1.4, 1.4 Hz, 1H), 7.56 (ddd, *J* = 7.8, 1.4, 1.4 Hz, 1H), 7.62 (dd, *J* = 1.4, 1.4 Hz, 1H); ¹³C NMR (67.9 MHz, CDCl₃): δ 20.9, 42.5, 112.6, 118.8, 126.4, 126.5, 127.2, 128.6, 129.2, 129.6, 130.3, 138.1, 138.7, 144.7, one peak was not found probably due to overlapping; HRMS (EI) *m/z*: [M]⁺ Calcd for C₁₇H₁₅N 233.12045; Found 233.12049.

(*E*)-1-Methyl-2-(3-phenylbut-1-en-1-yl)benzene (**3ka**)



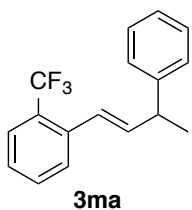
This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 98/2). Colorless oil (44.6 mg, 0.201 mmol, 80% yield as a mixture with a trace amount of *Z* isomer); IR (neat) 3025, 2965, 2870, 1491, 1452, 965, 745, 699 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 1.48 (d, $J = 7.1$ Hz, 3H), 2.33 (s, 3H), 3.67 (dq, $J = 7.1, 7.1, 1.3$ Hz, 1H), 6.25 (dd, $J = 15.6, 7.1$ Hz, 1H), 6.62 (dd, $J = 15.6, 1.3$ Hz, 1H), 7.11–7.24 (m, 4H), 7.27–7.35 (m, 4H), 7.40–7.43 (m, 1H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 19.8, 21.4, 42.8, 125.5, 126.0, 126.2, 126.4, 127.0, 127.3, 128.5, 130.1, 135.1, 136.6, 136.7, 145.7; HRMS (EI) m/z : $[\text{M}]^+$ Calcd for $\text{C}_{17}\text{H}_{18}$ 222.1409; Found 222.1408.

(*E*)-1-Methoxy-2-(3-phenylbut-1-en-1-yl)benzene (**3la**)¹⁰



This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 90/10). Colorless oil (54.5 mg, 0.229 mmol, 91% yield as a mixture with a trace amount of *Z* isomer); ^1H NMR (270 MHz, CDCl_3): δ 1.47 (d, $J = 7.0$ Hz, 3H), 3.66 (dq, $J = 7.0, 7.0$ Hz, 1H), 3.84 (s, 3H), 6.37 (dd, $J = 16.1, 7.0$ Hz, 1H), 6.76–6.92 (m, 3H), 7.15–7.23 (m, 2H), 7.27–7.34 (m, 4H), 7.42 (dd, $J = 7.7, 1.2$ Hz, 1H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 21.3, 43.0, 55.4, 110.8, 120.6, 123.0, 126.1, 126.4, 126.6, 127.3, 128.0, 128.4, 135.6, 146.0, 156.4; MS (EI) m/z : $[\text{M}]^+$ 238.

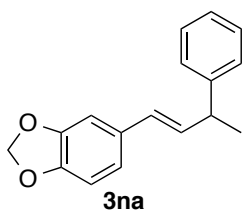
(*E*)-1-(3-Phenylbut-1-en-1-yl)-2-(trifluoromethyl)benzene (**3ma**)



This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 99/1). Colorless oil (48.9 mg, 0.177 mmol, 71% yield as a mixture with a trace amount of *Z* isomer); IR (neat) 3028, 2968, 1315, 1121, 1036, 765, 700 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 1.48 (d, $J = 7.3$ Hz, 3H), 3.63–3.74 (m, 1H), 6.33 (dd, $J = 15.7, 6.9$ Hz, 1H), 6.83 (d, $J = 15.7$ Hz, 1H), 7.19–7.37 (m, 6H), 7.41–7.47 (m, 1H), 7.59 (dd, $J = 7.6, 7.6$ Hz, 2H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 20.8, 42.7, 124.4 (q, $J_{\text{CF}} = 274$ Hz), 124.7 (q, $J_{\text{CF}} = 2.0$ Hz), 125.6 (q, $J_{\text{CF}} = 5.8$ Hz), 126.4, 126.7 (q, $J_{\text{CF}} = 30.7$ Hz), 126.7, 127.3

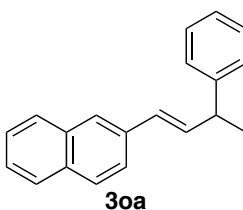
(2C), 128.5, 131.7 (q, $J_{CF} = 1.2$ Hz), 136.8 (q, $J_{CF} = 1.7$ Hz), 139.4, 145.1; ^{19}F NMR (471 MHz, CDCl_3): δ -60.6 (s); HRMS (EI) m/z : $[\text{M}]^+$ Calcd for $\text{C}_{17}\text{H}_{15}\text{F}_3$ 276.1126; Found 276.1125.

(E)-5-(3-Phenylbut-1-en-1-yl)benzo[*d*][1,3]dioxole (3na)



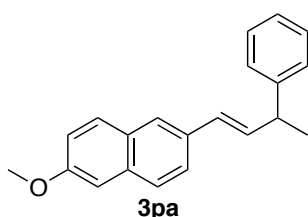
This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 90/10). Colorless oil (56.1 mg, 0.222 mmol, 89% yield as a mixture with a trace amount of *Z* isomer); IR (neat) 3027, 2965, 2875, 2777, 1603, 1252, 1190, 965, 804 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 1.44 (d, $J = 6.8$ Hz, 3H), 3.60 (dq, $J = 6.8, 6.8$ Hz, 1H), 5.92 (s, 2H), 6.20 (dd, $J = 15.9, 6.8$ Hz, 1H), 6.32 (d, $J = 15.9$ Hz, 1H), 6.71–6.79 (m, 2H), 6.90 (d, $J = 1.1$ Hz, 1H), 7.17–7.34 (m, 5H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 21.3, 42.4, 100.9, 105.5, 108.2, 120.5, 126.2, 127.2, 128.1, 128.4, 132.1, 133.5, 145.7, 146.7, 147.9; HRMS (EI) m/z : $[\text{M}]^+$ Calcd for $\text{C}_{17}\text{H}_{16}\text{O}_2$ 252.1150; Found 252.1154.

(E)-2-(3-Phenylbut-1-en-1-yl)naphthalene (3oa)¹⁴



This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). White solid (47.9 mg, 0.185 mmol, 74% yield as a mixture with a trace amount of *Z* isomer); ^1H NMR (270 MHz, CDCl_3): δ 1.51 (d, $J = 7.3$ Hz, 3H), 3.65–3.75 (m, 1H), 6.50 (dd, $J = 15.9, 5.4$ Hz, 1H), 6.59 (d, $J = 15.9$ Hz, 1H), 7.19–7.25 (m, 1H), 7.28–7.38 (m, 4H), 7.40–7.47 (m, 2H), 7.58 (dd, $J = 8.6, 1.6$ Hz, 1H), 7.69 (s, 1H), 7.73–7.79 (m, 3H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 21.2, 42.7, 123.6, 125.5, 125.7, 126.1, 126.3, 127.3, 127.6, 127.8, 128.1, 128.5, 128.6, 132.7, 133.6, 135.0, 135.7, 145.6; MS (EI) m/z : $[\text{M}]^+$ 258.

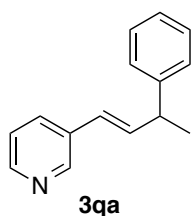
(E)-2-Methoxy-6-(3-phenylbut-1-en-1-yl)naphthalene (3pa)



This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography

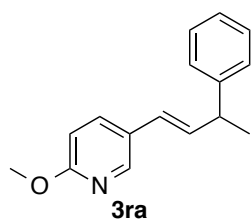
(Hexane/EtOAc = 90/10). White solid (64.4 mg, 0.223 mmol, 89% yield as a mixture with a trace amount of *Z* isomer); M.p. 76.8–79.4 °C; IR (KBr) 2962, 1600, 1482, 1242, 1031, 858, 700 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 1.50 (d, *J* = 6.7 Hz, 3H), 3.69 (dq, *J* = 6.7, 6.7 Hz, 1H), 3.91 (s, 3H), 6.45 (dd, *J* = 15.9, 6.7 Hz, 1H), 6.55 (d, *J* = 15.9 Hz, 1H), 7.09–7.13 (m, 2H), 7.20–7.26 (m, 1H), 7.28–7.32 (m, 3H), 7.33–7.36 (m, 1H), 7.54 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.63–7.69 (m, 3H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.3, 42.6, 55.2, 105.8, 118.8, 124.2, 125.6, 126.2, 126.9, 127.3, 128.5, 128.6, 129.1, 129.3, 132.9, 133.8, 134.6, 145.7, 157.5; HRMS (APCI/TOF) *m/z*: [M+H]⁺ Calcd for C₂₁H₂₁O 289.1592; Found 289.1582.

(*E*)-3-(3-Phenylbut-1-en-1-yl)pyridine (**3qa**)¹⁵



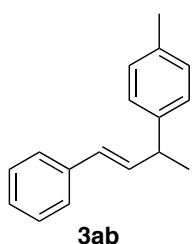
This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 95/5). Colorless oil (34.1 mg, 0.163 mmol, 65% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.48 (d, *J* = 6.6 Hz, 3H), 3.67 (dq, *J* = 6.6, 6.6 Hz, 1H), 6.38 (d, *J* = 16.1 Hz, 1H), 6.48 (dd, *J* = 16.1, 6.6 Hz, 1H), 7.20–7.28 (m, 4H), 7.31–7.37 (m, 2H), 7.68 (d, *J* = 8.1 Hz, 1H), 8.43 (d, *J* = 3.5 Hz, 1H), 8.57 (s, 1H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.0, 42.7, 123.3, 124.9, 126.4, 127.2, 128.6, 132.6, 133.1, 137.7, 144.9, 148.0 (2C); MS (EI) *m/z*: [M]⁺ 209.

(*E*)-2-Methoxy-5-(3-phenylbut-1-en-1-yl)pyridine (**3ra**)¹¹



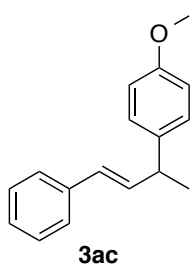
This compound was prepared according to the general procedure and the desired product was obtained after purification by silica gel column chromatography (Hexane/EtOAc = 80/20). Colorless oil (55.6 mg, 0.232 mmol, 93% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.46 (d, *J* = 6.4 Hz, 3H), 3.63 (dq, *J* = 6.4, 6.4 Hz, 1H), 3.92 (s, 3H), 6.26 (dd, *J* = 16.0, 6.4 Hz, 1H), 6.35 (d, *J* = 16.0 Hz, 1H), 6.68 (d, *J* = 8.9 Hz, 1H), 7.19–7.28 (m, 2H), 7.30–7.31 (m, 2H), 7.32–7.36 (m, 1H), 7.63 (dd, *J* = 8.9, 2.2 Hz, 1H), 8.07 (d, *J* = 2.2 Hz, 1H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.4, 42.6, 53.4, 110.7, 124.5, 126.2, 126.7, 127.2, 128.5, 134.6, 135.3, 145.1, 145.4, 163.2; MS (EI) *m/z*: [M]⁺ 239.

(*E*)-1-Methyl-4-(4-phenylbut-3-en-2-yl)benzene (**3ab**)¹⁶



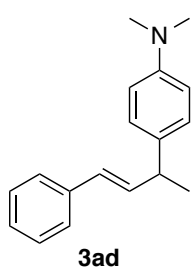
This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 95/5). Colorless oil (51.6 mg, 0.232 mmol, 93% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.45 (d, *J* = 6.8 Hz, 3H), 2.33 (s, 3H), 3.53–3.69 (m, 1H), 6.32–6.45 (m, 2H), 7.08–7.14 (m, 2H), 7.15–7.22 (m, 3H), 7.27–7.37 (m, 4H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.0, 21.2, 42.1, 126.1, 127.0, 127.2, 128.3, 128.4, 129.1, 135.4, 135.7, 137.6, 142.6; MS (EI) *m/z*: [M]⁺ 222.

(*E*)-1-Methoxy-4-(4-phenylbut-3-en-2-yl)benzene (**3ac**)¹⁶



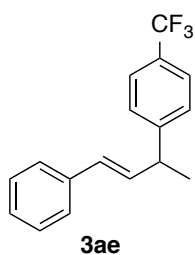
This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 9/1). Colorless oil (56.5 mg, 0.237 mmol, 95% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.44 (d, *J* = 7.3 Hz, 3H), 3.53–3.67 (m, 1H), 3.79 (s, 3H), 6.30–6.43 (m, 2H), 6.83–6.88 (m, 2H), 7.16–7.21 (m, 3H), 7.25–7.31 (m, 2H), 7.33–7.36 (m, 2H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.3, 41.7, 55.2, 113.8, 126.1, 127.0, 128.2, 128.4, 135.5, 137.6 (2C), 158.0, one peak was not found probably due to overlapping; MS (EI) *m/z*: [M]⁺ 238.

(*E*)-*N,N*-Dimethyl-4-(4-phenylbut-3-en-2-yl)aniline (**3ad**)¹⁷



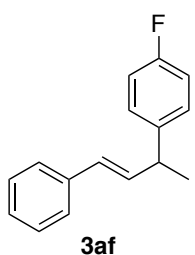
This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 9/1 × 2). Yellow oil (53.1 mg, 0.211 mmol, 84% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.43 (d, *J* = 7.0 Hz, 3H), 2.93 (s, 6H), 3.51–3.61 (m, 1H), 6.31–6.44 (m, 2H), 6.74 (d, *J* = 8.1 Hz, 2H), 7.14–7.21 (m, 3H), 7.25–7.37 (m, 4H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.2, 40.8, 41.5, 113.0, 126.1, 126.8, 127.8, 127.9, 128.4, 133.7, 136.0, 137.8, 149.2; MS (EI) *m/z*: [M]⁺ 251.

(*E*)-1-(4-Phenylbut-3-en-2-yl)-4-(trifluoromethyl)benzene (**3ae**)⁵



This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 95/5 and Hexane). Colorless oil (56.3 mg, 0.204 mmol, 82% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.48 (d, *J* = 6.6 Hz, 3H), 3.70 (dq, *J* = 6.6, 6.6 Hz, 1H), 6.34 (dd, *J* = 16.1, 6.6 Hz, 1H), 6.43 (d, *J* = 16.1 Hz, 1H), 7.18–7.24 (m, 1H), 7.27–7.33 (m, 2H), 7.34–7.40 (m, 4H), 7.57 (d, *J* = 7.8 Hz, 2H); ¹³C NMR (126 MHz, CDCl₃): δ 21.0, 42.4, 124.3 (q, *J*_{CF} = 273 Hz), 125.4 (q, *J*_{CF} = 3.6 Hz), 126.2, 127.3, 127.6, 128.5, 128.6 (q, *J*_{CF} = 32.0 Hz), 129.3, 134.0, 137.2, 149.7; ¹⁹F NMR (471 MHz, CDCl₃): δ -63.3 (s); MS (EI) *m/z*: [M]⁺ 276.

(*E*)-1-Fluoro-4-(4-phenylbut-3-en-2-yl)benzene (**3af**)¹⁶

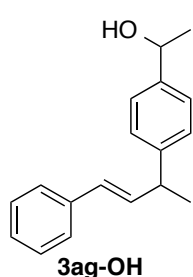


This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 95/5). Colorless oil (51.1 mg, 0.223 mmol, 90% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.45 (d, *J* = 6.8 Hz, 3H), 3.58–3.68 (m, 1H), 6.33 (dd, *J* = 15.9, 5.1 Hz, 1H), 6.40 (d, *J* = 15.9 Hz, 1H), 6.95–7.04 (m, 2H), 7.17–7.20 (m, 1H), 7.22–7.25 (m, 2H), 7.26–7.32 (m, 2H), 7.34–7.37 (m, 2H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.3, 41.8, 115.2 (d, *J*_{CF} = 21.2 Hz), 126.1, 127.1, 128.5, 128.6, 128.7 (d, *J*_{CF} = 7.8 Hz), 134.9, 137.4, 141.2 (d, *J*_{CF} = 2.8 Hz), 161.4 (d, *J*_{CF} = 244 Hz); ¹⁹F NMR (471 MHz, CDCl₃): δ -118.16 – -118.22 (m); MS (EI) *m/z*: [M]⁺ 226.

(*E*)-1-(4-(4-Phenylbut-3-en-2-yl)phenyl)ethanol (**3ag-OH**)

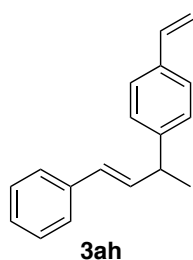
Methyl (1-phenylbut-3-en-1-yl) carbonate (**1a**) (51.6 mg, 0.25 mmol), P(*p*-MeOC₆H₄)₃ (17.6 mg, 0.05 mmol, 0.2 equiv), 4-AcC₆H₄B(OH)₂ (61.5 mg, 0.375 mmol, 1.5 equiv), and K₂CO₃ (51.8 mg, 0.375 mmol, 1.5 equiv) were charged into a screw cap vial and the vial was taken into a glove box. In the glove box, Ni(cod)₂ (6.9 mg, 0.025 mmol, 0.1 equiv) and CH₃CN (1.25 mL) were added to the vial and the reaction vial was taken from the glove box. The resulting mixture was stirred at 60 °C for 36 h. The reaction

mixture was allowed to cool to room temperature and the reaction was quenched with water (1 mL). The resulting aqueous phase was extracted with EtOAc (2 mL \times 1). The organic phase was washed with brine and dried over MgSO₄. After removal of the solvent, the crude reaction mixture was purified by silica gel column chromatography (Hexane/EtOAc = 98/2, 95/5) to give (*E*)-1-(4-(4-phenylbut-3-en-2-yl)phenyl)ethanone as a mixture with P(*p*-MeOC₆H₄)₃. The resulting mixture was dissolved in MeOH (1 mL) and NaBH₄ (11.3 mg, 0.30 mmol, 1.2 equiv) was added to the mixture. The reaction mixture was stirred at room temperature for overnight and then concentrated by evaporation. Water (1 mL) was added to the resulting mixture and the aqueous phase was extracted with EtOAc (2 mL \times 1). The organic phase was washed with brine and dried over MgSO₄. After removal of the solvent, the resulting crude mixture was purified by preparative thin-layer chromatography (Hexane/EtOAc = 4/1) to give (*E*)-1-(4-(4-phenylbut-3-en-2-yl)phenyl)ethanol (**3ag-OH**) as a pale yellow oil (56.6 mg, 0.224 mmol, 89% yield as a mixture with a trace amount of *Z* isomer).



IR (neat) 3348, 3024, 2968, 2870, 1599, 1448, 1089, 966, 830, 752, 693 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): δ 1.46 (d, *J* = 7.0 Hz, 3H), 1.49 (d, *J* = 6.2 Hz, 3H), 1.73 (brs, 1H), 3.59–3.69 (m, 1H), 4.88 (q, *J* = 6.3 Hz, 1H), 6.36 (dd, *J* = 15.7, 5.3 Hz, 1H), 6.43 (d, *J* = 15.7 Hz, 1H), 7.16–7.22 (m, 1H), 7.24–7.37 (m, 8H); ¹³C NMR (67.9 MHz, CDCl₃): δ 21.2, 25.0, 42.2, 70.1, 125.6, 126.1, 127.0, 127.4, 128.5 (2C), 135.1, 137.5, 143.7, 144.9; HRMS (ESI/TOF) *m/z*: [M-OH]⁺ Calcd for C₁₈H₁₉ 235.1487; Found 235.1489.

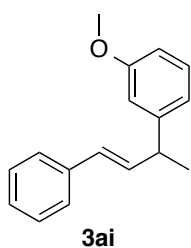
(*E*)-1-(4-Phenylbut-3-en-2-yl)-4-vinylbenzene (**3ah**)¹⁸



This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 95/5). Colorless oil (36.1 mg, 0.154 mmol, 61% yield as a mixture with a trace amount of *Z* isomer); ¹H NMR (270 MHz, CDCl₃): δ 1.46 (d, *J* = 7.0 Hz, 3H), 3.58–3.68 (m, 1H), 5.21 (dd, *J* = 10.9, 1.0 Hz, 1H), 5.72 (dd, *J* = 17.5, 1.0 Hz, 1H), 6.31–6.45 (m, 2H), 6.70 (dd, *J* = 17.5, 10.9 Hz, 1H), 7.13–7.22 (m, 2H),

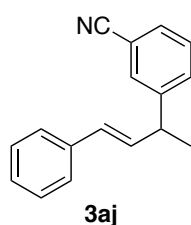
7.25–7.31 (m, 3H), 7.34–7.38 (m, 4H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 21.1, 42.3, 113.2, 126.1, 126.3, 127.0, 127.5, 128.5, 128.6, 135.0, 135.6, 136.5, 137.5, 145.3; MS (EI) m/z : $[\text{M}]^+$ 234.

(*E*)-1-Methoxy-3-(4-phenylbut-3-en-2-yl)benzene (**3ai**)¹⁹



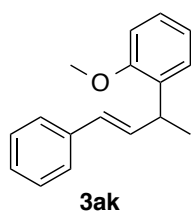
This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 95/5). Colorless oil (55.9 mg, 0.235 mmol, 94% yield as a mixture with a trace amount of *Z* isomer); ^1H NMR (270 MHz, CDCl_3): δ 1.46 (d, $J = 6.8$ Hz, 3H), 3.56–3.66 (m, 1H), 3.80 (s, 3H), 6.36 (dd, $J = 15.9, 5.1$ Hz, 1H), 6.43 (d, $J = 15.9$ Hz, 1H), 6.76 (ddd, $J = 8.3, 2.6, 1.0$ Hz, 1H), 6.82 (dd, $J = 2.2, 2.2$ Hz, 1H), 6.87 (d, $J = 7.6$ Hz, 1H), 7.16–7.24 (m, 2H), 7.27–7.31 (m, 2H), 7.33–7.37 (m, 2H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 21.1, 42.6, 55.1, 111.2, 113.3, 119.7, 126.1, 127.0, 128.4, 128.5, 129.4, 135.0, 137.5, 147.3, 159.7; MS (EI) m/z : $[\text{M}]^+$ 238.

(*E*)-3-(4-Phenylbut-3-en-2-yl)benzonitrile (**3aj**)¹⁸



This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 9/1). Pale yellow oil (55.0 mg, 0.236 mmol, 94% yield as a mixture with a trace amount of *Z* isomer); ^1H NMR (270 MHz, CDCl_3): δ 1.48 (d, $J = 6.9$ Hz, 3H), 3.68 (dq, $J = 6.9, 6.9$ Hz, 1H), 6.30 (dd, $J = 15.7, 6.9$ Hz, 1H), 6.43 (d, $J = 15.7$ Hz, 1H), 7.19–7.25 (m, 1H), 7.28–7.33 (m, 2H), 7.34–7.44 (m, 3H), 7.50–7.56 (m, 3H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 20.9, 42.1, 112.4, 118.9, 126.1, 127.4, 128.5, 129.2, 129.6, 130.0, 130.9, 131.9, 133.4, 136.9, 146.9; MS (EI) m/z : $[\text{M}]^+$ 233.

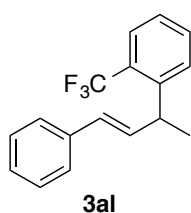
(*E*)-1-Methoxy-2-(4-phenylbut-3-en-2-yl)benzene (**3ak**)²⁰



This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 95/5 and Hexane). Colorless oil (51.2 mg, 0.215 mmol, 86% yield as a mixture with a

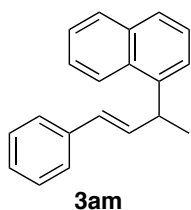
trace amount of *Z* isomer); ^1H NMR (270 MHz, CDCl_3): δ 1.41 (d, $J = 6.8$ Hz, 3H), 3.85 (s, 3H), 4.04–4.13 (m, 1H), 6.37–6.49 (m, 2H), 6.87–6.95 (m, 2H), 7.15–7.23 (m, 3H), 7.25–7.31 (m, 2H), 7.35–7.38 (m, 2H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 20.0, 35.1, 55.4, 110.6, 120.6, 126.1, 126.8, 127.1, 127.5, 128.2, 128.4, 134.0, 134.9, 137.9, 156.7; MS (EI) m/z : $[\text{M}]^+$ 238.

(*E*)-1-(4-Phenylbut-3-en-2-yl)-2-(trifluoromethyl)benzene (**3al**)



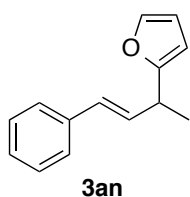
This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 95/5 and Hexane). Colorless oil (45.6 mg, 0.165 mmol, 66% yield as a mixture with a trace amount of *Z* isomer); IR (neat) 3027, 2973, 2875, 1649, 1607, 1494, 1455, 1312, 1119, 966, 768, 693 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 1.47 (d, $J = 6.1$ Hz, 3H), 4.09 (dq, $J = 6.1, 6.1$ Hz, 1H), 6.38 (dd, $J = 16.0, 6.1$ Hz, 1H), 6.46 (d, $J = 16.0$ Hz, 1H), 7.17–7.23 (m, 1H), 7.26–7.38 (m, 5H), 7.44–7.53 (m, 2H), 7.62–7.65 (m, 1H); ^{13}C NMR (126 MHz, CDCl_3): δ 21.7, 37.4, 124.6 (q, $J_{\text{CF}} = 274$ Hz), 125.7 (q, $J_{\text{CF}} = 6.0$ Hz), 126.1, 126.2, 127.2, 127.7 (q, $J_{\text{CF}} = 29.7$ Hz), 128.5, 129.0, 132.0, 134.0, 137.3, 145.1, one peak was not found probably due to overlapping; ^{19}F NMR (471 MHz, CDCl_3): δ -59.6 (s); HRMS (EI) m/z : $[\text{M}]^+$ Calcd for $\text{C}_{17}\text{H}_{15}\text{F}_3$ 276.11257; Found 276.11255.

(*E*)-1-(4-Phenylbut-3-en-2-yl)naphthalene (**3am**)¹⁹



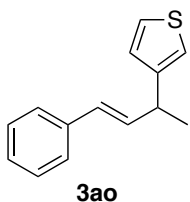
This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 95/5). Colorless oil (62.2 mg, 0.241 mmol, 96% yield as a mixture with a trace amount of *Z* isomer); ^1H NMR (270 MHz, CDCl_3): δ 1.62 (d, $J = 6.2$ Hz, 3H), 4.47 (dq, $J = 6.2, 6.2$ Hz, 1H), 6.47 (d, $J = 15.9$ Hz, 1H), 6.56 (dd, $J = 15.9, 6.2$ Hz, 1H), 7.16–7.21 (m, 1H), 7.23–7.25 (m, 1H), 7.27–7.37 (m, 3H), 7.39–7.44 (m, 1H), 7.45–7.55 (m, 3H), 7.71–7.77 (m, 1H), 7.84–7.89 (m, 1H), 8.16–8.19 (m, 1H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 20.8, 37.3, 123.5, 123.8, 125.4, 125.6, 125.8, 126.1, 126.9, 127.0, 128.5, 128.7, 128.9, 131.4, 134.0, 134.9, 137.5, 141.5; MS (EI) m/z : $[\text{M}]^+$ 258.

(*E*)-2-(4-Phenylbut-3-en-2-yl)furan (**3an**)



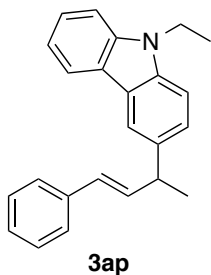
This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 95/5 and Hexane). Pale yellow oil (30.2 mg, 0.152 mmol, 61% yield); IR (neat) 3026, 2971, 2873, 1598, 1505, 1448, 1009, 964, 751, 733, 693 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 1.45 (d, $J = 6.9$ Hz, 3H), 3.69 (dq, $J = 6.9, 6.9$ Hz, 1H), 6.05–6.07 (m, 1H), 6.25–6.33 (m, 2H), 6.45 (d, $J = 15.9$ Hz, 1H), 7.17–7.24 (m, 1H), 7.25–7.39 (m, 5H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 18.9, 36.6, 104.2, 110.0, 126.2, 127.2, 128.5, 129.6, 132.1, 137.2, 141.2, 158.4; HRMS (EI) m/z : $[\text{M}]^+$ Calcd for $\text{C}_{14}\text{H}_{14}\text{O}$ 198.1045; Found 198.1044.

(*E*)-3-(4-Phenylbut-3-en-2-yl)thiophene (**3ao**)



This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 95/5). Yellow oil (39.5 mg, 0.184 mmol, 74% yield as a mixture with a trace amount of *Z* isomer); IR (neat) 3024, 2965, 2871, 1648, 1598, 1494, 1448, 965, 780, 749, 694 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 1.47 (d, $J = 6.8$ Hz, 3H), 3.71 (dq, $J = 6.8, 6.8$ Hz, 1H), 6.32 (dd, $J = 16.0, 6.8$ Hz, 1H), 6.42 (d, $J = 16.0$ Hz, 1H), 7.01–7.02 (m, 2H), 7.17–7.23 (m, 1H), 7.26–7.32 (m, 3H), 7.34–7.38 (m, 2H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 21.0, 38.3, 119.5, 125.4, 126.1, 127.1, 127.4, 128.5, 128.6, 134.8, 137.5, 146.3; HRMS (EI) m/z : $[\text{M}]^+$ Calcd for $\text{C}_{14}\text{H}_{14}\text{S}$ 214.0816; Found 214.0815.

(*E*)-9-Ethyl-3-(4-phenylbut-3-en-2-yl)-9*H*-carbazole (**3ap**)



This compound was prepared according to the general procedure and the desired product was obtained after purification by preparative thin-layer chromatography (Hexane/EtOAc = 9/1 \times 3). Yellow oil (74.9 mg, 0.230 mmol, 92% yield as a mixture with a trace amount of *Z* isomer and inseparable impurities); IR (neat) 3023, 2971, 2870, 1601, 1489, 1470, 1332, 1232, 966, 748, 731 cm^{-1} ; ^1H NMR (270 MHz, CDCl_3): δ 1.41 (t, $J = 7.1$ Hz, 3H), 1.58 (d, $J = 6.7$ Hz, 3H), 3.84 (dq, $J = 6.7, 6.7$ Hz, 1H), 4.34 (q, $J = 7.1$ Hz, 2H), 6.45 (d, $J = 16.2$ Hz, 1H), 6.49–6.57

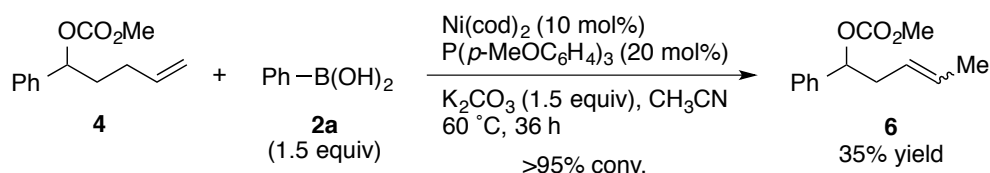
(m, 1H), 7.15–7.25 (m, 3H), 7.28–7.39 (m, 6H), 7.42–7.48 (m, 1H), 7.99 (d, $J = 0.81$ Hz, 1H), 8.04–8.12 (m, 1H); ^{13}C NMR (67.9 MHz, CDCl_3): δ 13.8, 21.8, 37.5, 42.5, 108.3, 118.5, 118.6, 120.4, 122.8, 123.0, 125.3, 125.5, 126.1, 126.9, 128.0, 128.4, 136.1, 136.2, 137.7, 138.7, 140.2, one peak was not found probably due to overlapping; HRMS (APCI/TOF) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{24}\text{H}_{24}\text{N}$ 326.1909; Found 326.1908.

4. Experimental details for the 3 mmol scale experiment

Methyl (1-phenylbut-3-en-1-yl) carbonate (**1a**) (619 mg, 3.0 mmol), $\text{P}(p\text{-MeOC}_6\text{H}_4)_3$ (211 mg, 0.6 mmol, 0.2 equiv), $\text{PhB}(\text{OH})_2$ (585 mg, 4.5 mmol, 1.5 equiv), and K_2CO_3 (622 mg, 4.5 mmol, 1.5 equiv) were charged into 30 mL Schlenk flask and the flask was taken into a glove box. In the glove box, $\text{Ni}(\text{cod})_2$ (82.5 mg, 0.30 mmol, 0.1 equiv) and CH_3CN (15 mL) were added to the vial and the reaction vial was taken from the glove box. The resulting mixture was stirred at 60 °C for 36 h. The reaction mixture was allowed to cool to room temperature and the reaction was quenched with water. The resulting mixture was filtrated with a pad of celite. The filtrate was transferred to a separate funnel and the aqueous phase was extracted with EtOAc. The organic phase was washed with brine and dried over MgSO_4 . After removal of the solvent, the crude reaction mixture was purified by silica gel column chromatography (Hexane/EtOAc = 99/1, 98/2, 97/3, 95/5, 9/1) to give (*E*)-but-1-ene-1,3-diylidibenzene (**3aa**) as a colorless oil (586 mg, 2.81 mmol, 93% yield as a mixture with a trace amount of *Z* isomer).

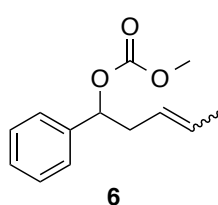
5. Experimental details for the control experiments

Scheme S7. Reaction of methyl (1-phenylpent-4-en-1-yl) carbonate (**4**) with phenylboronic acid (**2a**)



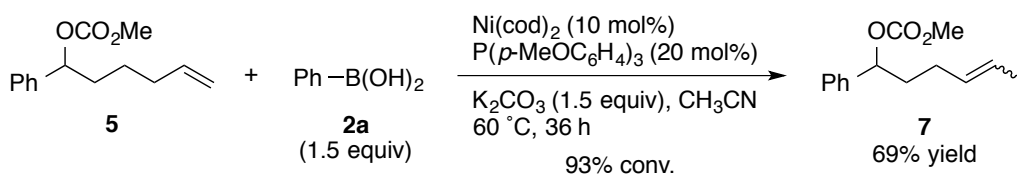
Methyl (1-phenylpent-4-en-1-yl) carbonate (**4**) (55.1 mg, 0.25 mmol), $\text{P}(p\text{-MeOC}_6\text{H}_4)_3$ (17.6 mg, 0.05 mmol, 0.2 equiv), $\text{PhB}(\text{OH})_2$ (48.7 mg, 0.375 mmol, 1.5 equiv), and K_2CO_3 (51.8 mg, 0.375 mmol, 1.5 equiv) were charged into a screw cap vial and the vial was taken into a glove box. In the glove box, $\text{Ni}(\text{cod})_2$ (6.9 mg, 0.025 mmol, 0.1

equiv) and CH₃CN (1.2 mL) were added to the vial and the reaction vial was taken from the glove box. The resulting mixture was stirred at 60 °C for 36 h. The reaction mixture was allowed to cool to room temperature and the reaction was quenched with water (1 mL). The resulting aqueous phase was extracted with EtOAc (2 mL × 1). The organic phase was washed with brine and dried over MgSO₄. After removal of the solvent, the crude reaction mixture was purified by silica gel column chromatography to give methyl (1-phenylpent-3-en-1-yl) carbonate (**6**) as a colorless oil (19.2 mg, 0.0872 mmol, 35% yield as a 66:34 mixture of stereoisomers).



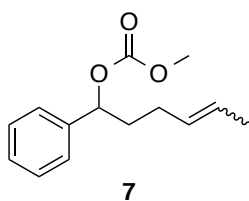
IR (neat) 3032, 2956, 2855, 1748, 1442, 967, 936, 792, 763, 700 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): (as a 66:34 mixture of stereoisomers) δ 1.55 (d, *J* = 7.3 Hz, 1.6 H), 1.62 (dd, *J* = 6.3, 0.95 Hz, 3H), 2.44–2.83 (m, 3H), 3.74 (s, 4H), 5.27–5.38 (m, 1.4 H), 5.45–5.60 (m, 3H), 7.26–7.35 (m, 7.6 H); ¹³C NMR (67.9 MHz, CDCl₃): (*major* isomer) δ 17.9, 39.6, 54.7, 79.9, 125.3, 126.5, 128.1, 128.4, 129.0, 139.7, 155.2; (*minor* isomer) δ 12.8, 33.9, 79.7, 124.2, 127.5, 128.1, 155.2, other peaks were not found probably due to overlapping; HRMS (ESI/TOF) *m/z*: [M+Na]⁺ Calcd for C₁₃H₁₆O₃Na 243.0997; Found 243.0992.

Scheme S8. Reaction of methyl (1-phenylhex-5-en-1-yl) carbonate (5**) with phenylboronic acid (**2a**)**



Methyl (1-phenylhex-5-en-1-yl) carbonate (**5**) (58.7 mg, 0.25 mmol), P(*p*-MeOC₆H₄)₃ (17.6 mg, 0.05 mmol, 0.2 equiv), PhB(OH)₂ (48.7 mg, 0.375 mmol, 1.5 equiv), and K₂CO₃ (51.8 mg, 0.375 mmol, 1.5 equiv) were charged into a screw cap vial and the vial was taken into a glove box. In the glove box, Ni(cod)₂ (6.9 mg, 0.025 mmol, 0.1 equiv) and CH₃CN (1.2 mL) were added to the vial and the reaction vial was taken from the glove box. The resulting mixture was stirred at 60 °C for 36 h. The reaction mixture was allowed to cool to room temperature and the reaction was quenched with

water (1 mL). The resulting aqueous phase was extracted with EtOAc (2 mL \times 1). The organic phase was washed with brine and dried over MgSO₄. After removal of the solvent, the crude reaction mixture was purified by silica gel column chromatography to give methyl (1-phenylhex-4-en-1-yl) carbonate (**7**) as a colorless oil (40.5 mg, 0.173 mmol, 69% yield as a 57:43 mixture of stereoisomers).



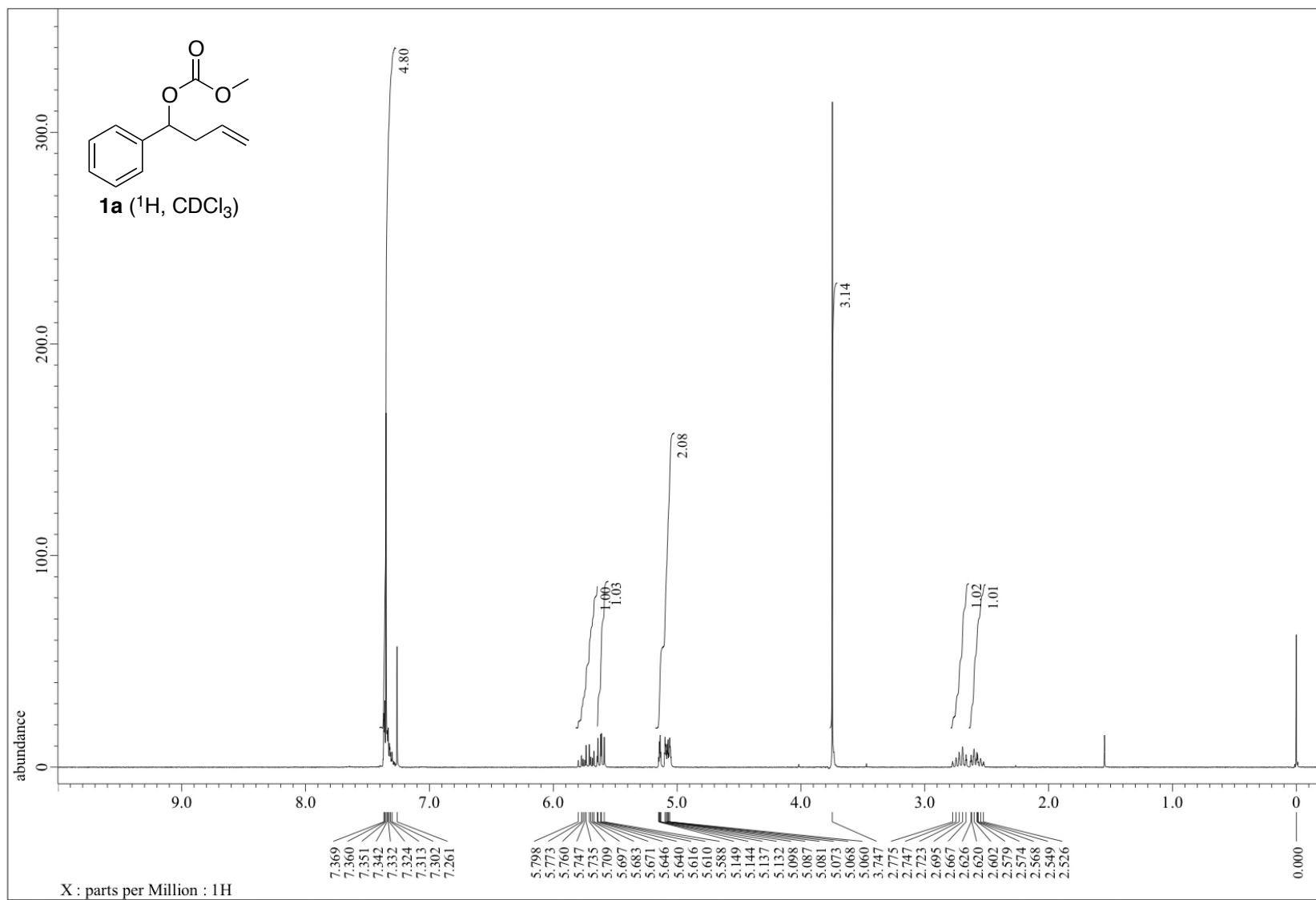
IR (neat) 3032, 2955, 2854, 1749, 1442, 1265, 942, 792, 763, 699 cm⁻¹; ¹H NMR (270 MHz, CDCl₃): (as a 57:43 mixture of stereoisomers) δ 1.55 (d, J = 6.8 Hz, 2H), 1.64 (d, J = 4.3 Hz, 3H), 1.77–1.91 (m, 1.5 H), 1.94–2.13 (m, 5H), 3.77 (s, 5H), 5.33–5.58 (m, 5H), 7.26–7.38 (m, 9H); ¹³C NMR (67.9 MHz, CDCl₃): (*major* isomer) δ 17.9, 28.4, 36.1, 54.6, 79.8, 126.0, 126.5, 128.1, 128.2, 128.5, 140.0, 155.2; (*minor* isomer) δ 12.7, 22.9, 36.0, 125.1, 128.8, 129.6, 140.0, other peaks were not found probably due to overlapping; HRMS (ESI/TOF) m/z : [M+Na]⁺ Calcd for C₁₄H₁₈O₃Na 257.1154; Found 257.1155.

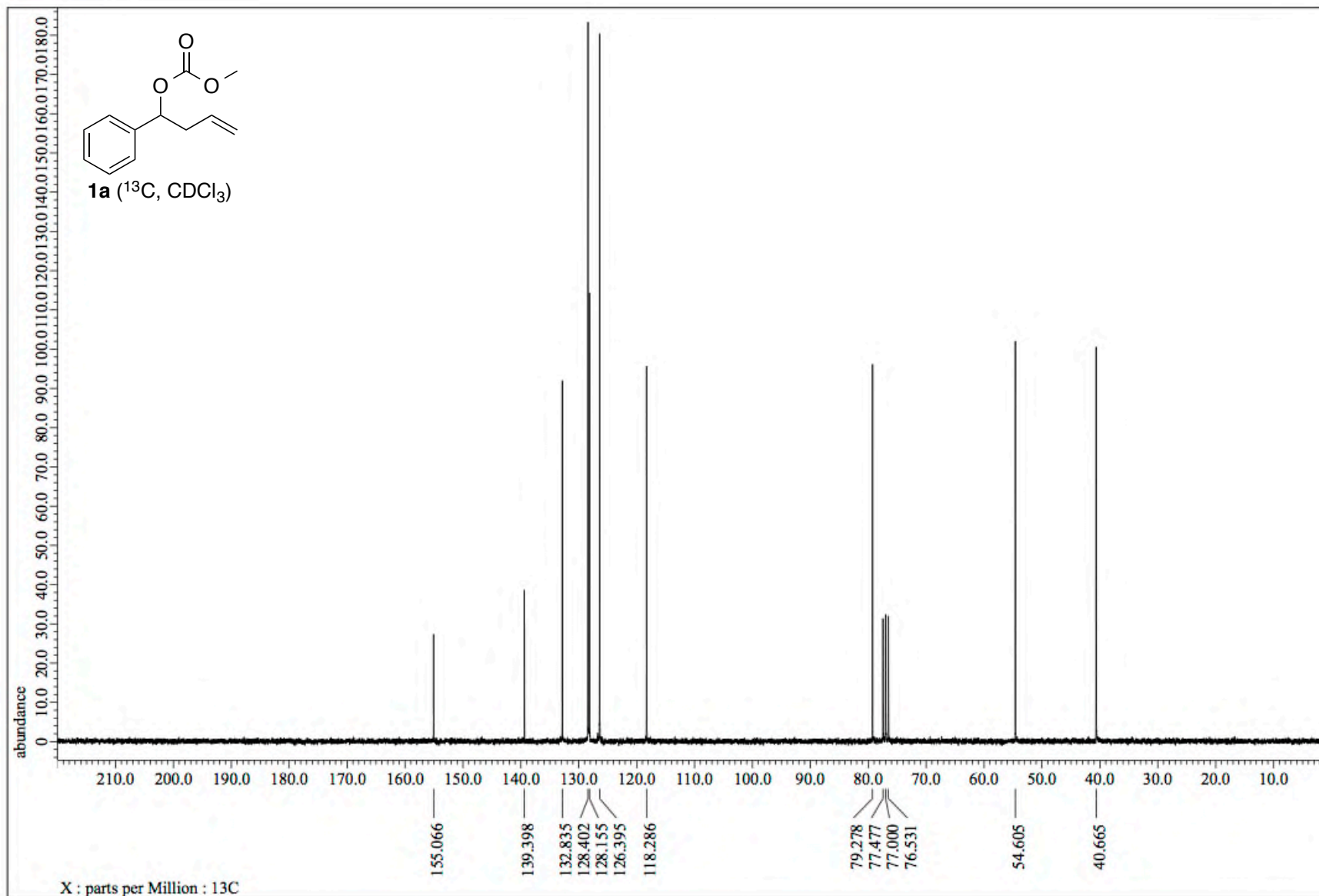
6. References

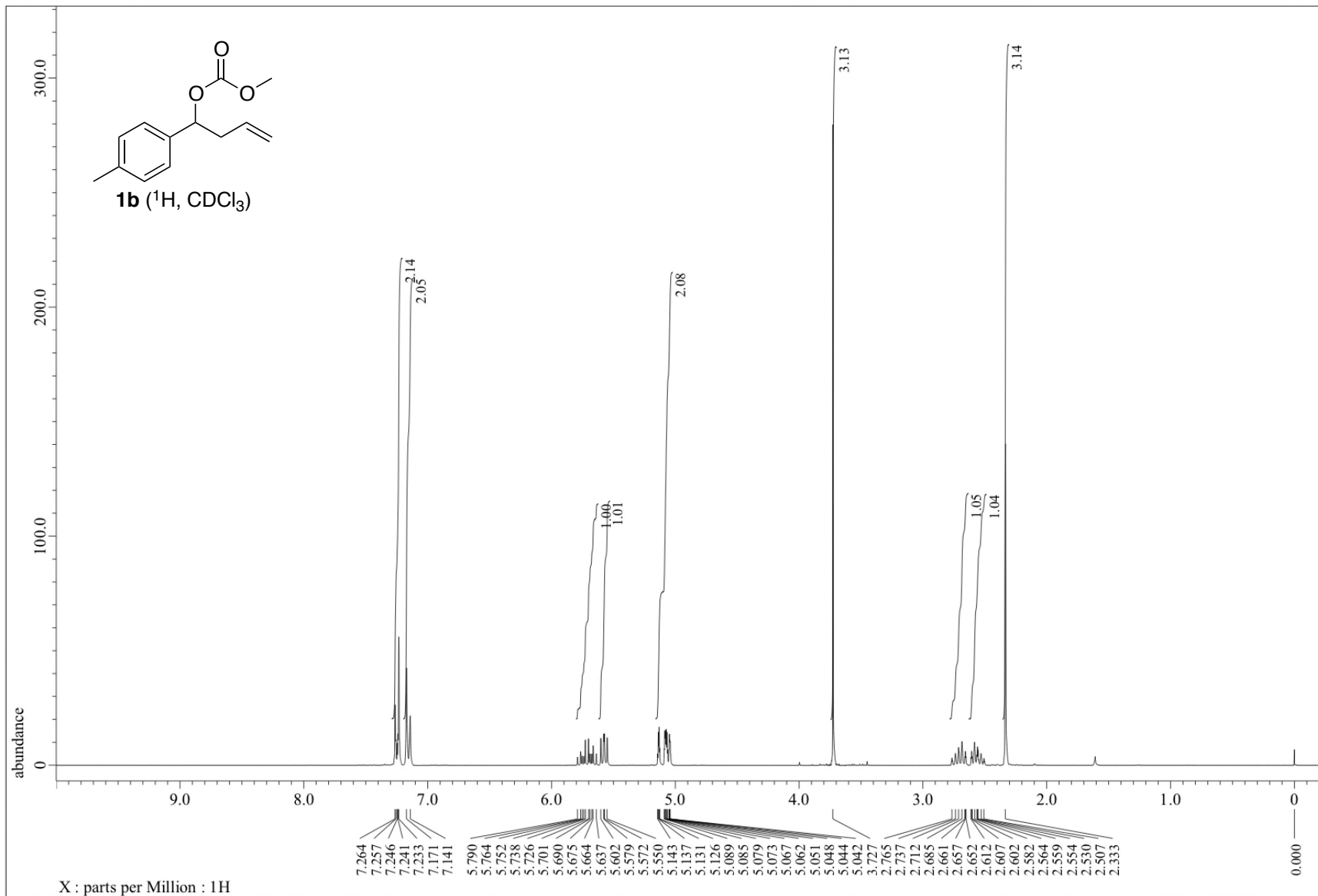
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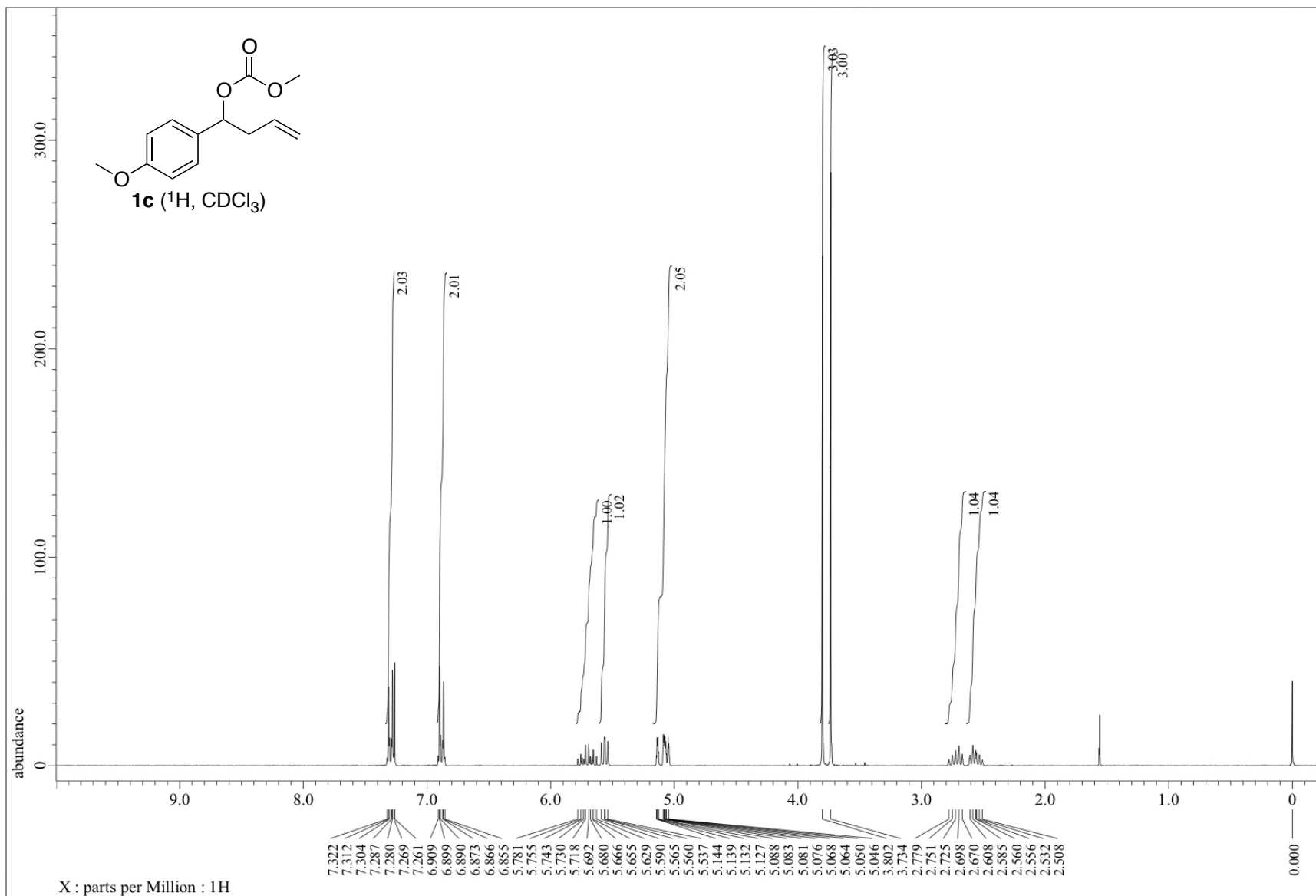
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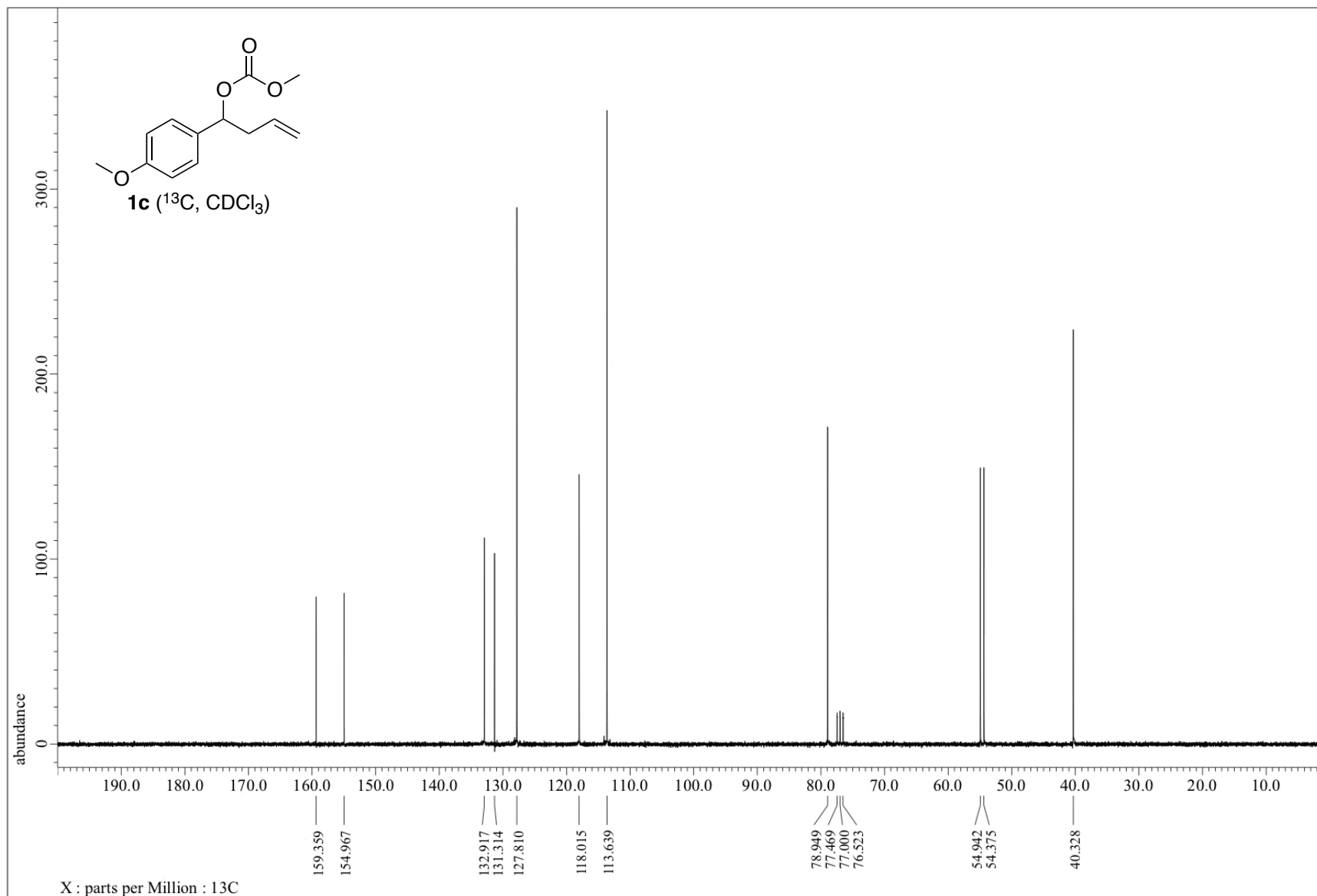
7. Copies of NMR spectra (^1H , ^{13}C , and ^{19}F)

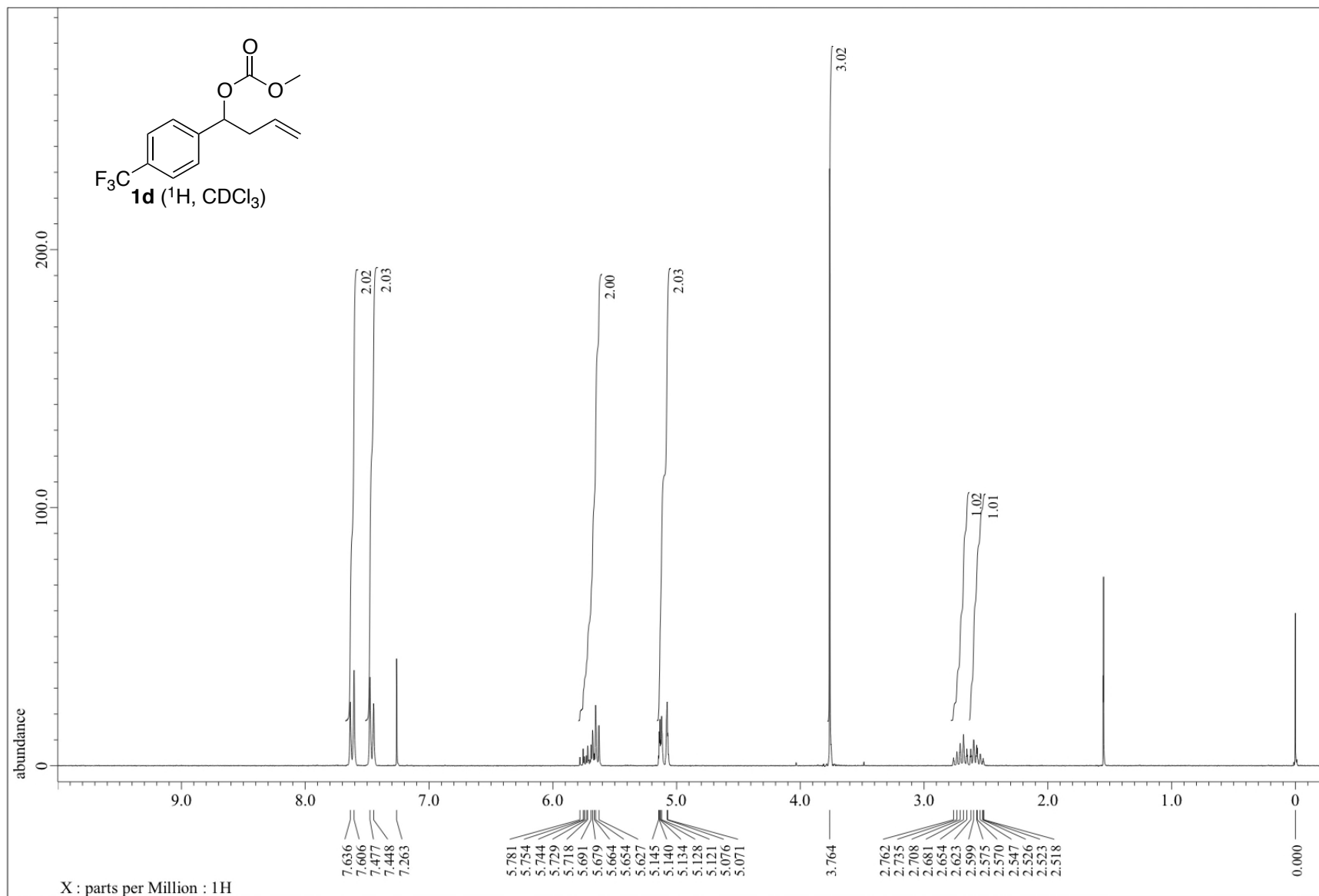


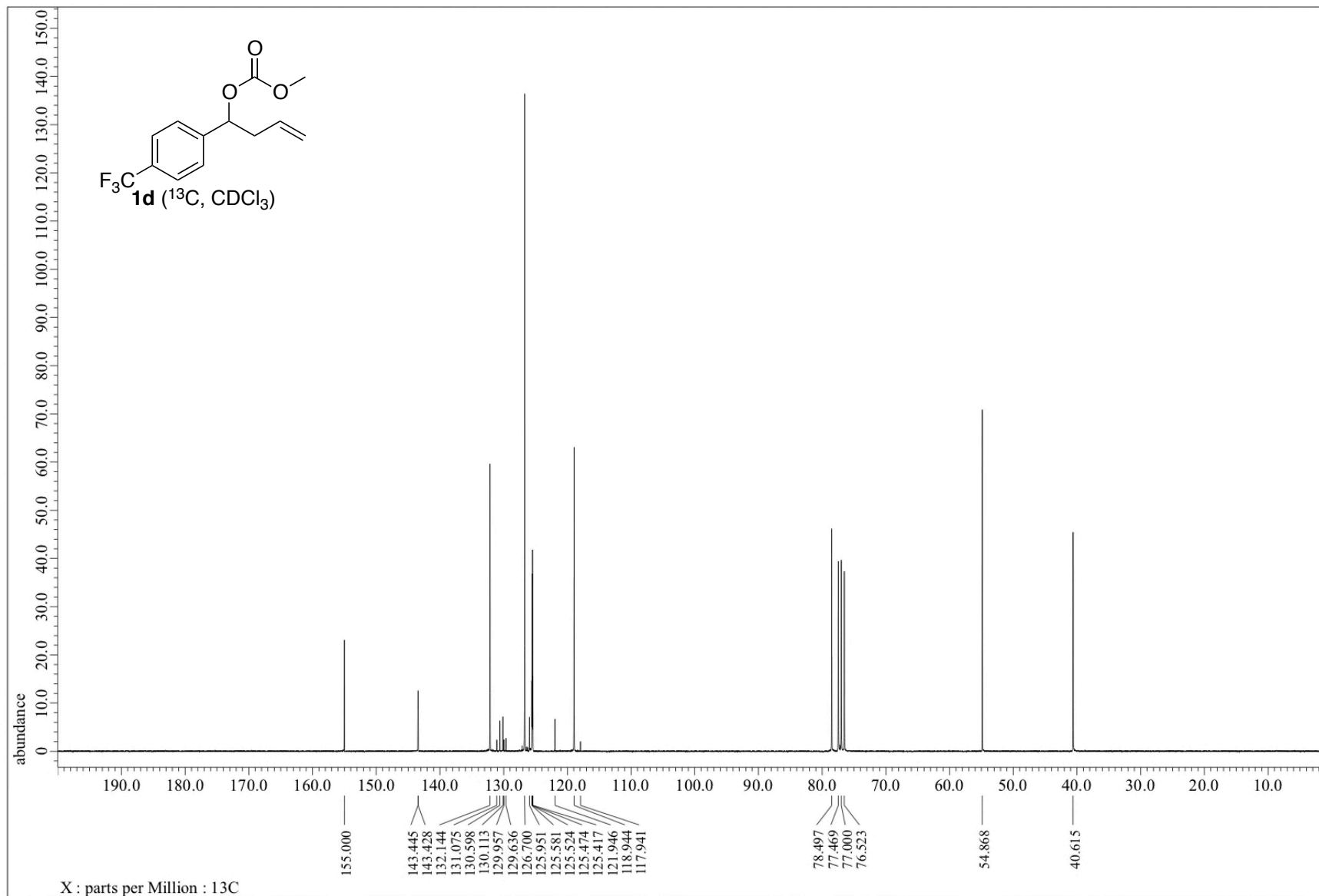


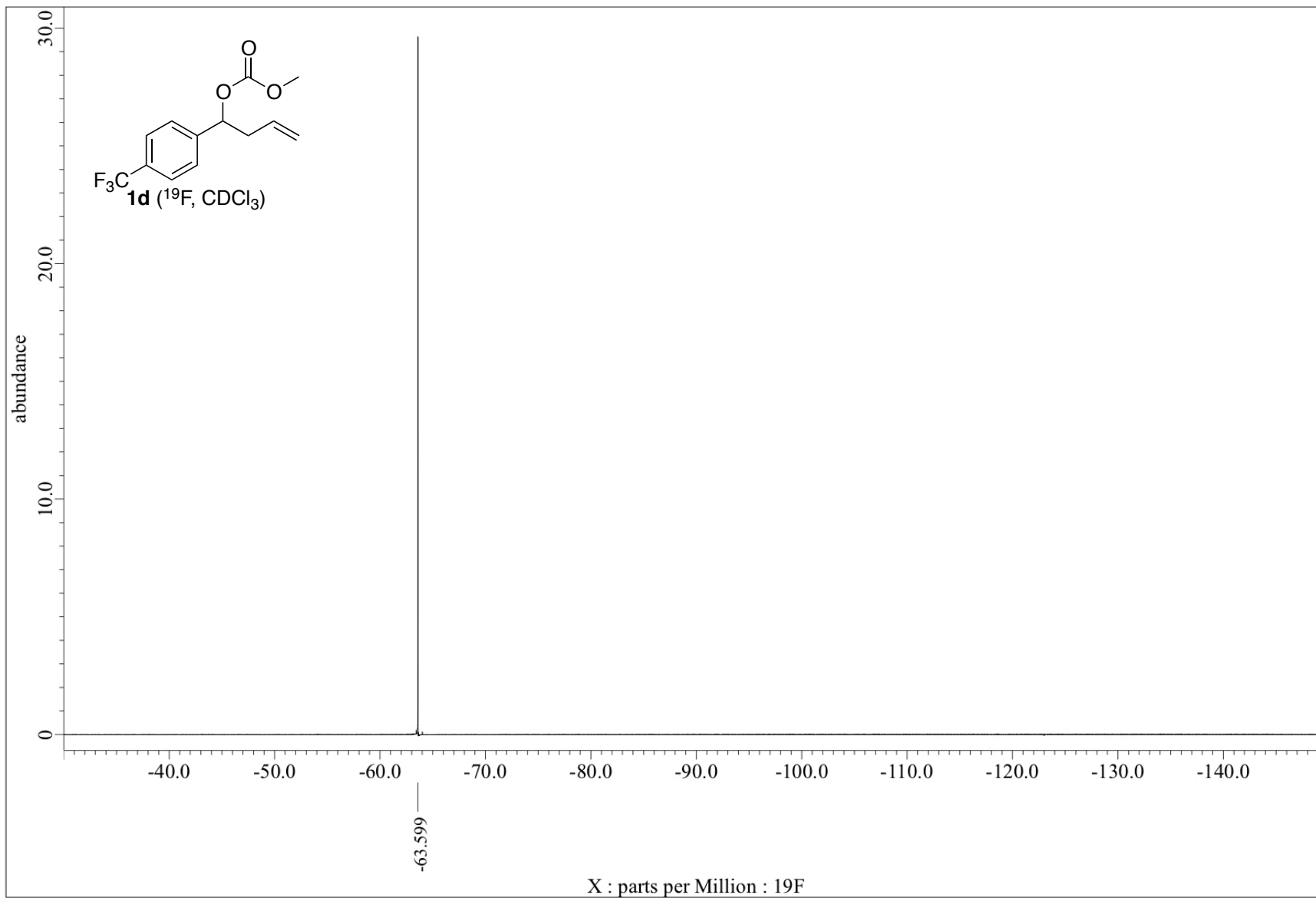


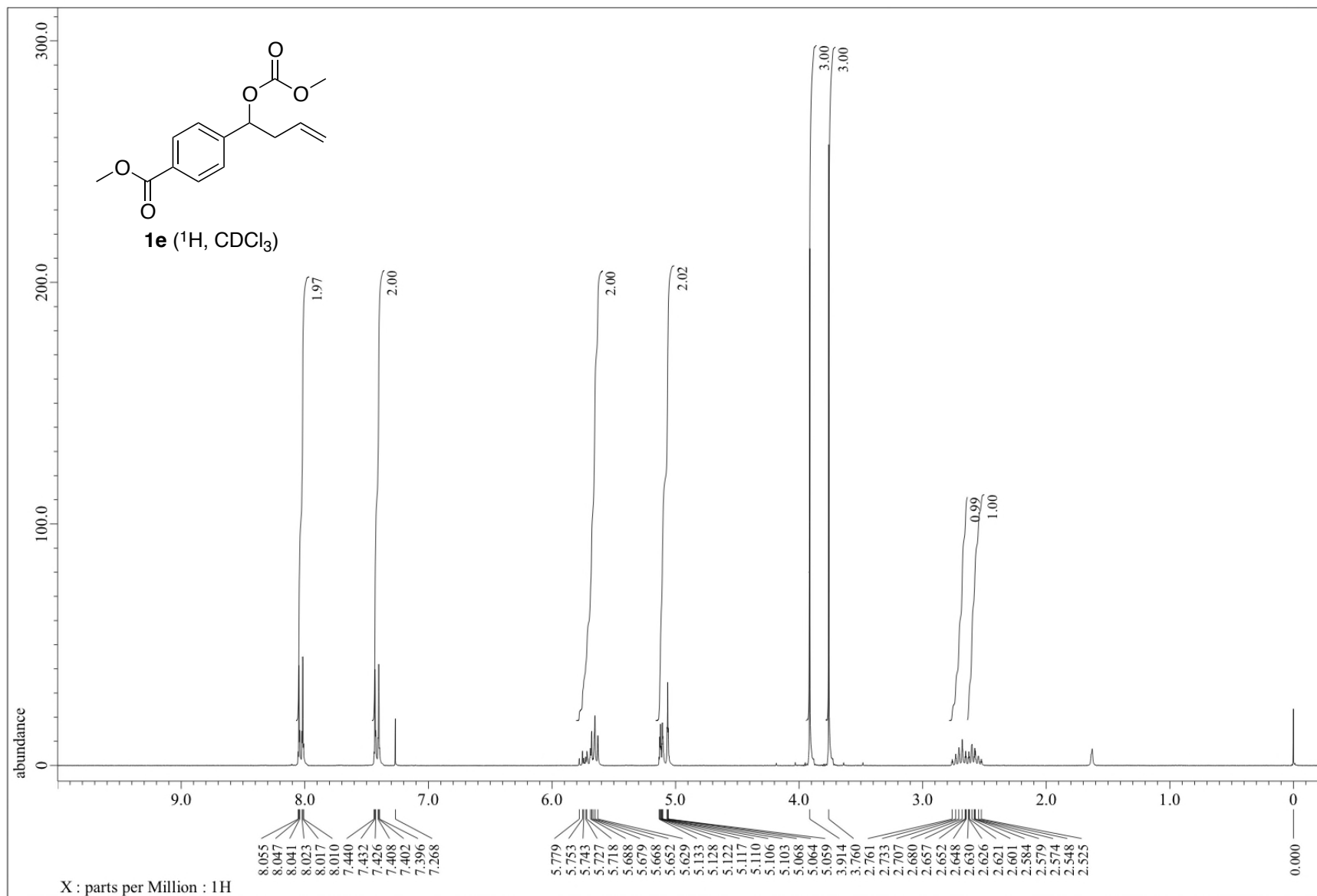


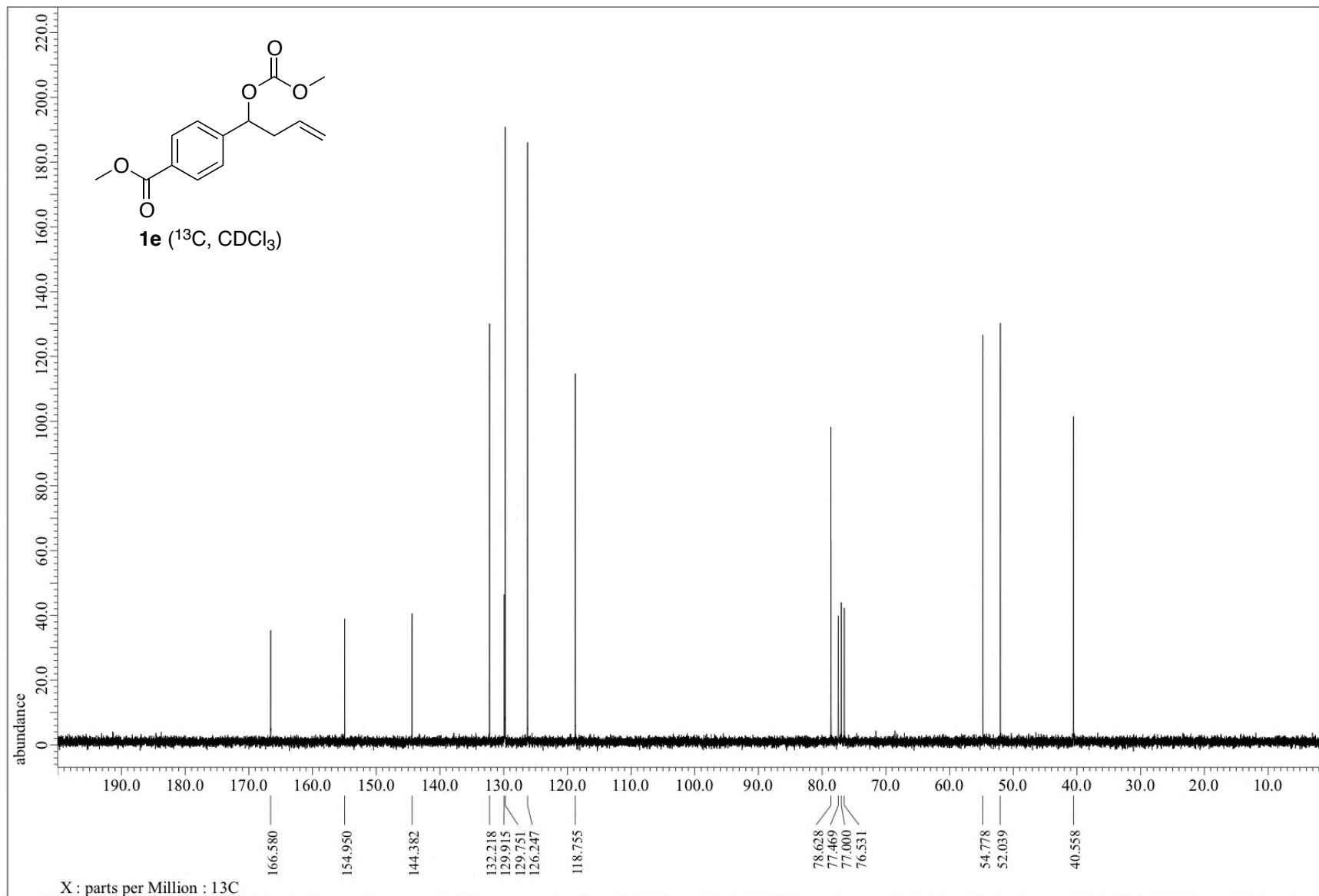


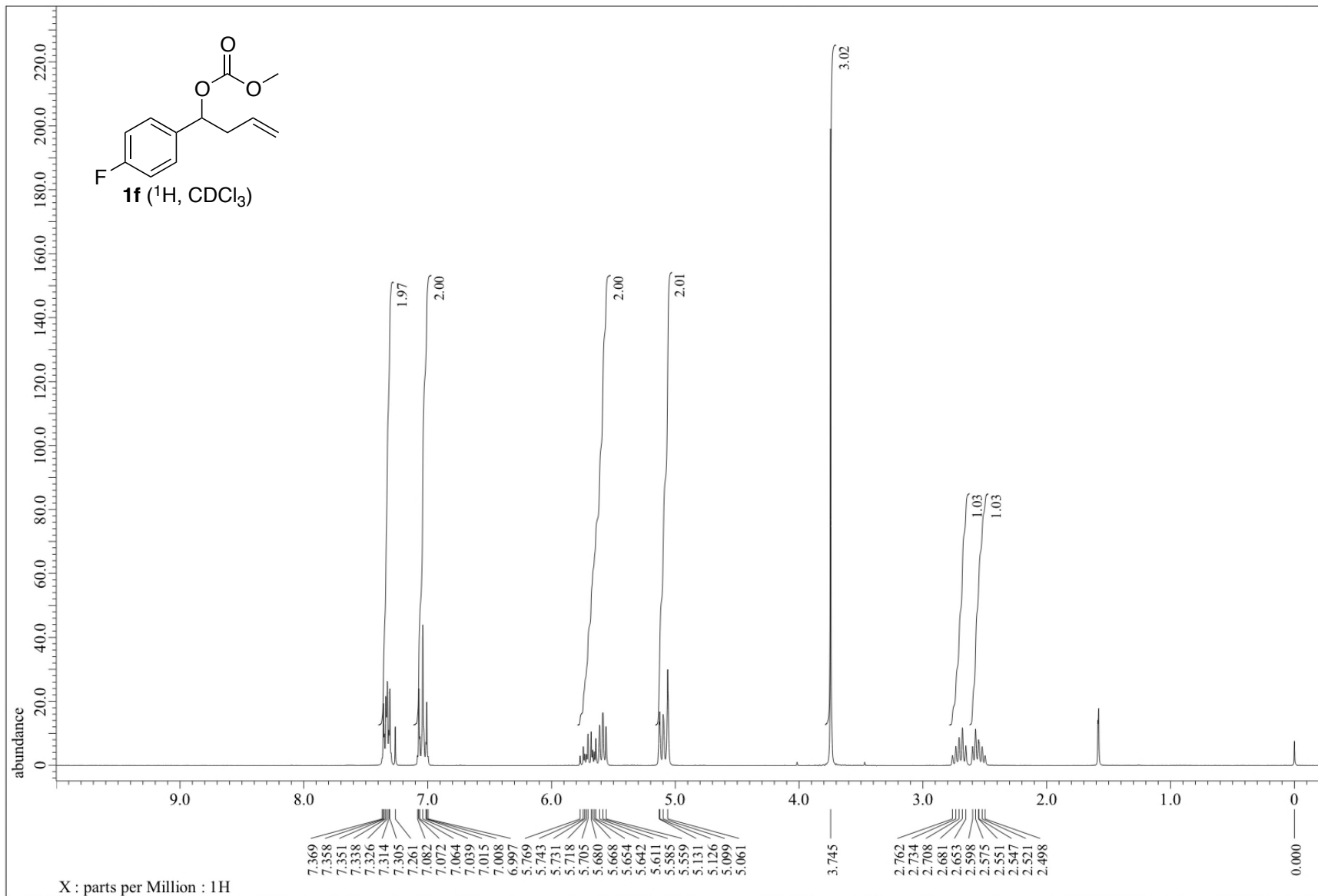


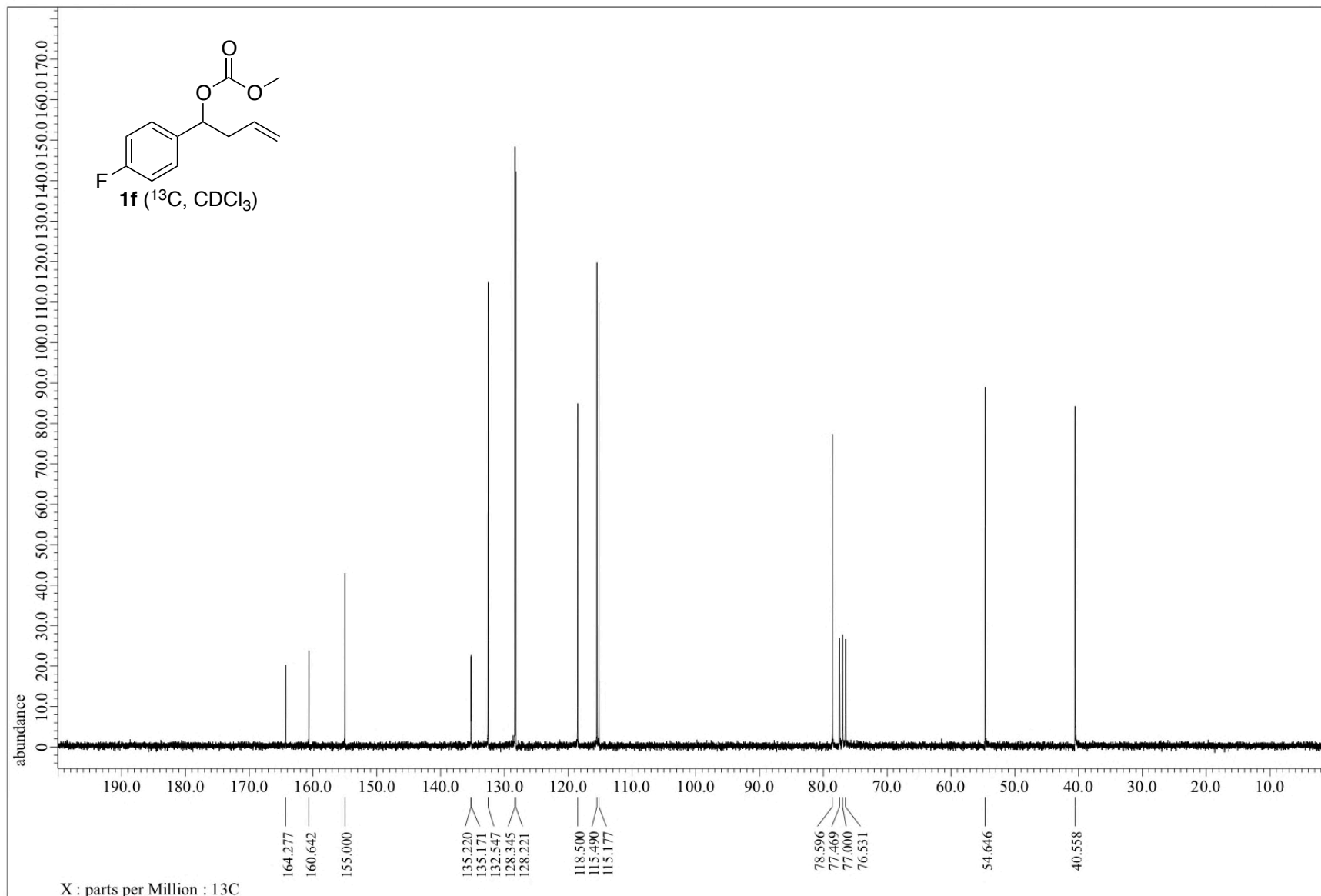


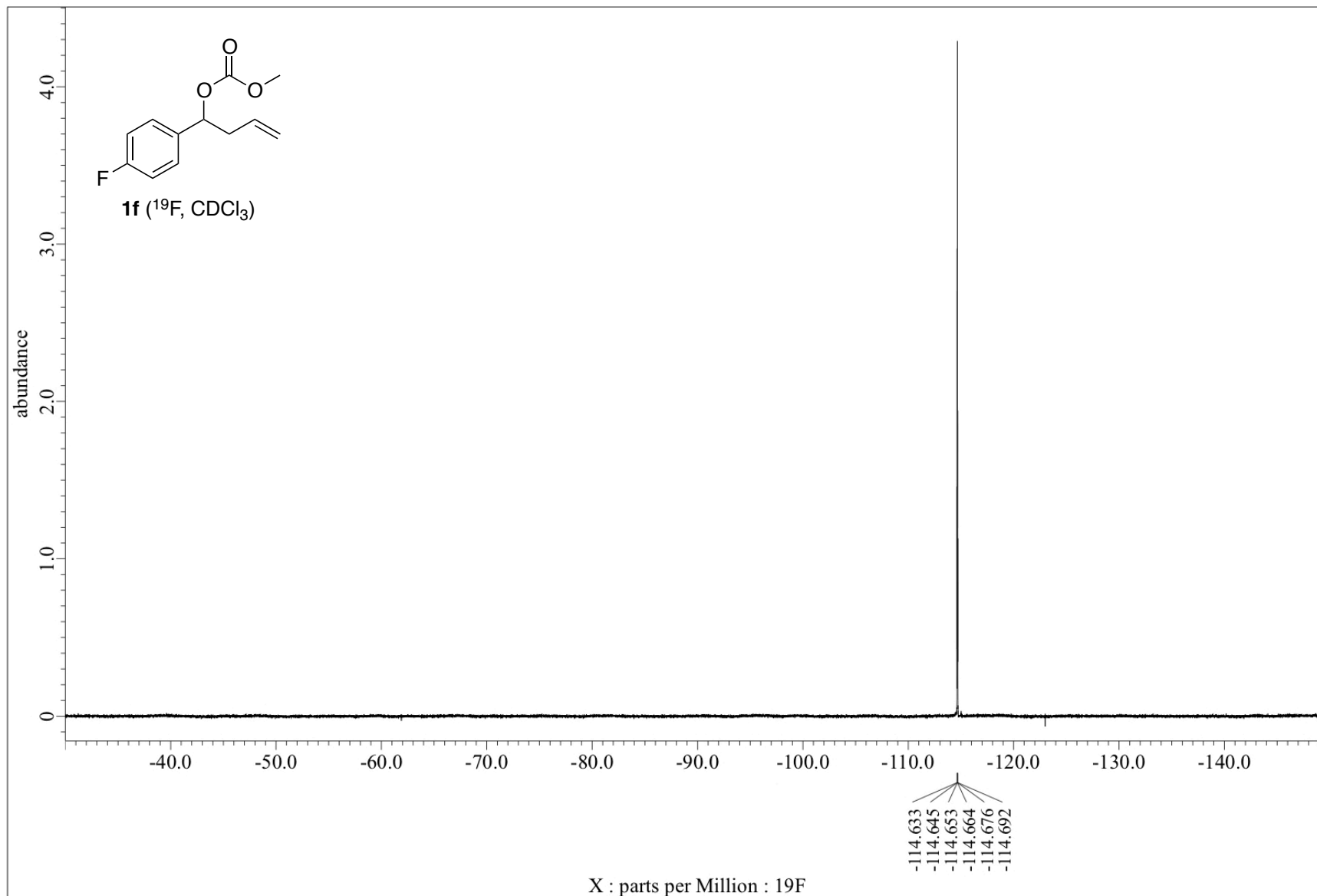


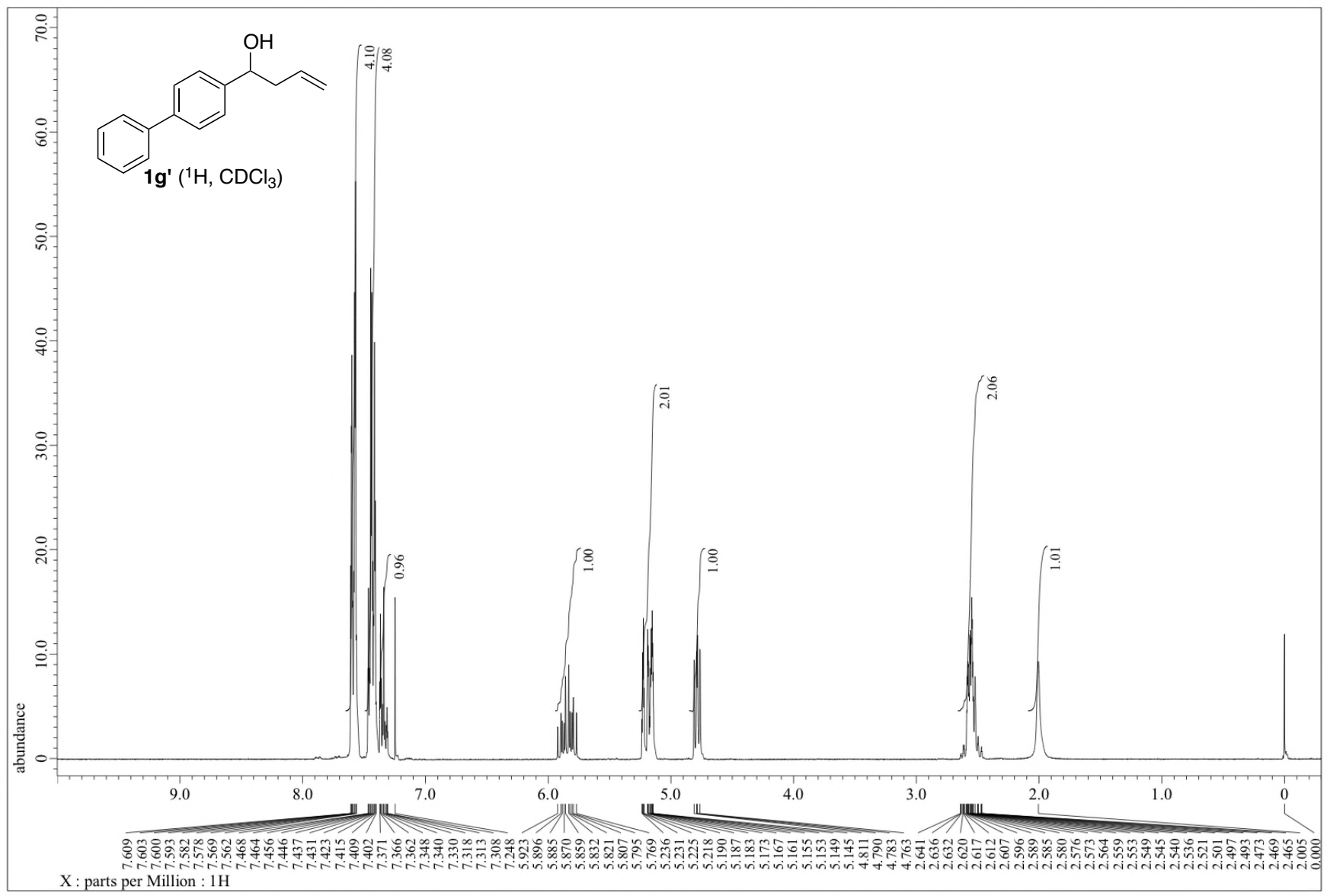


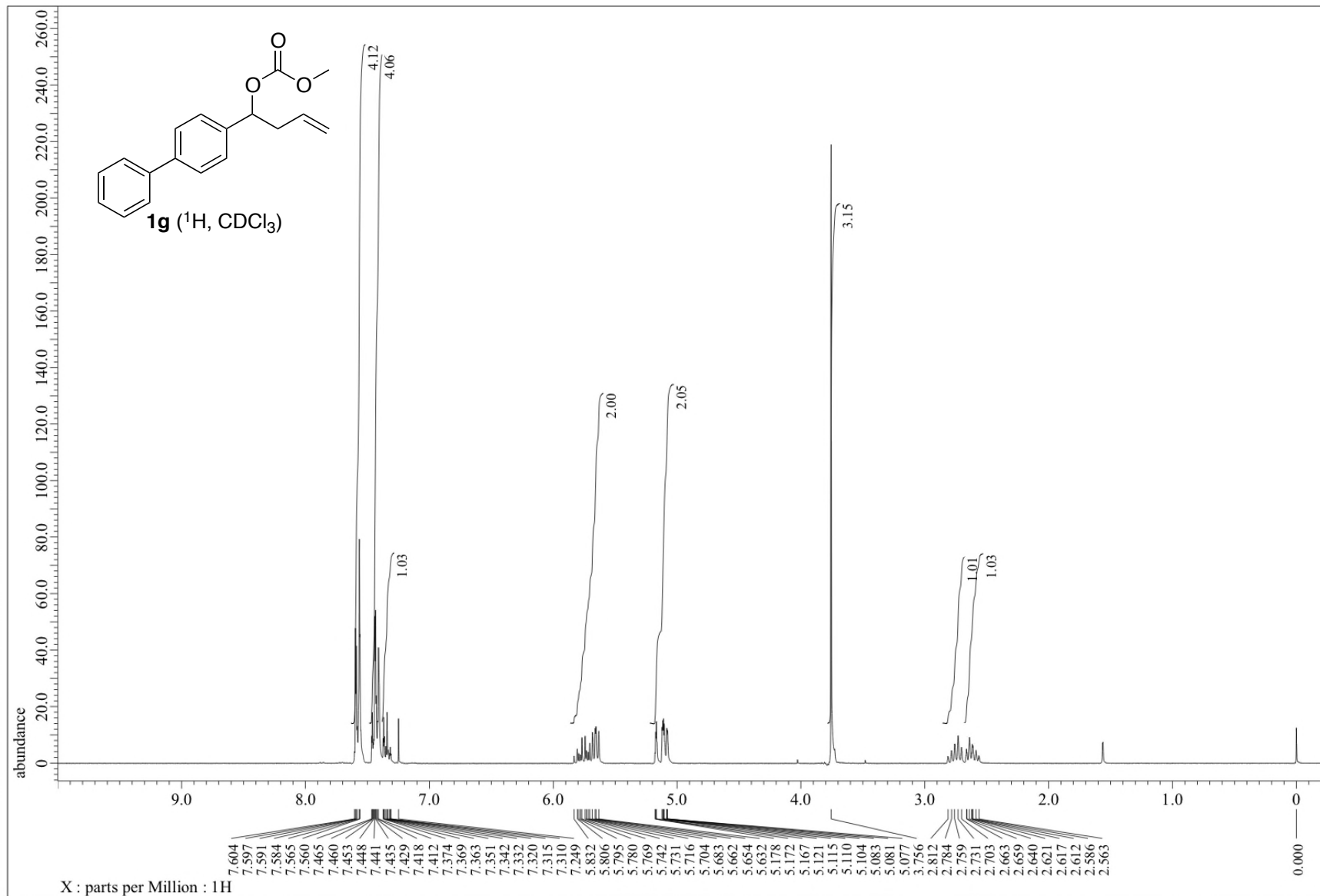


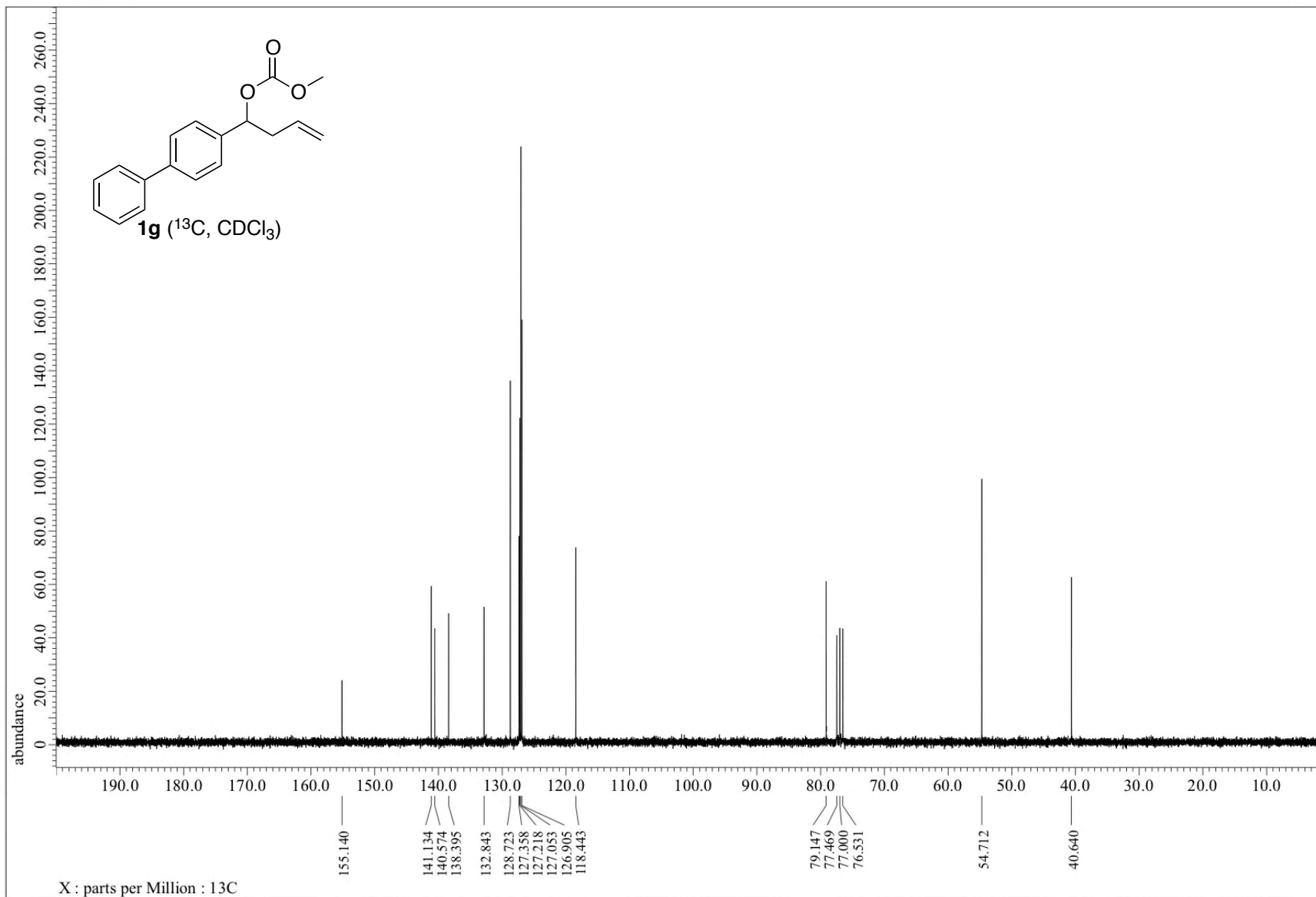


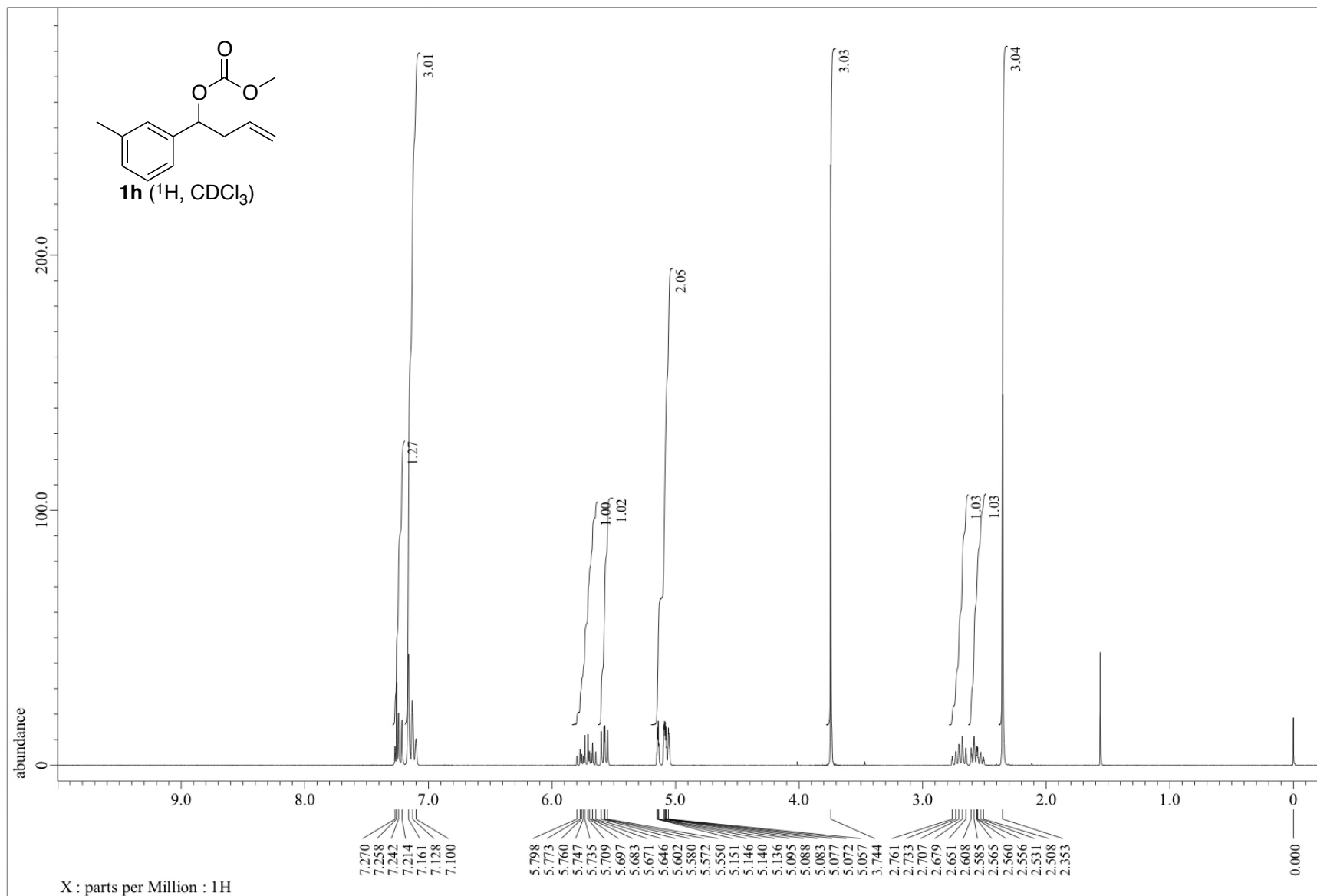


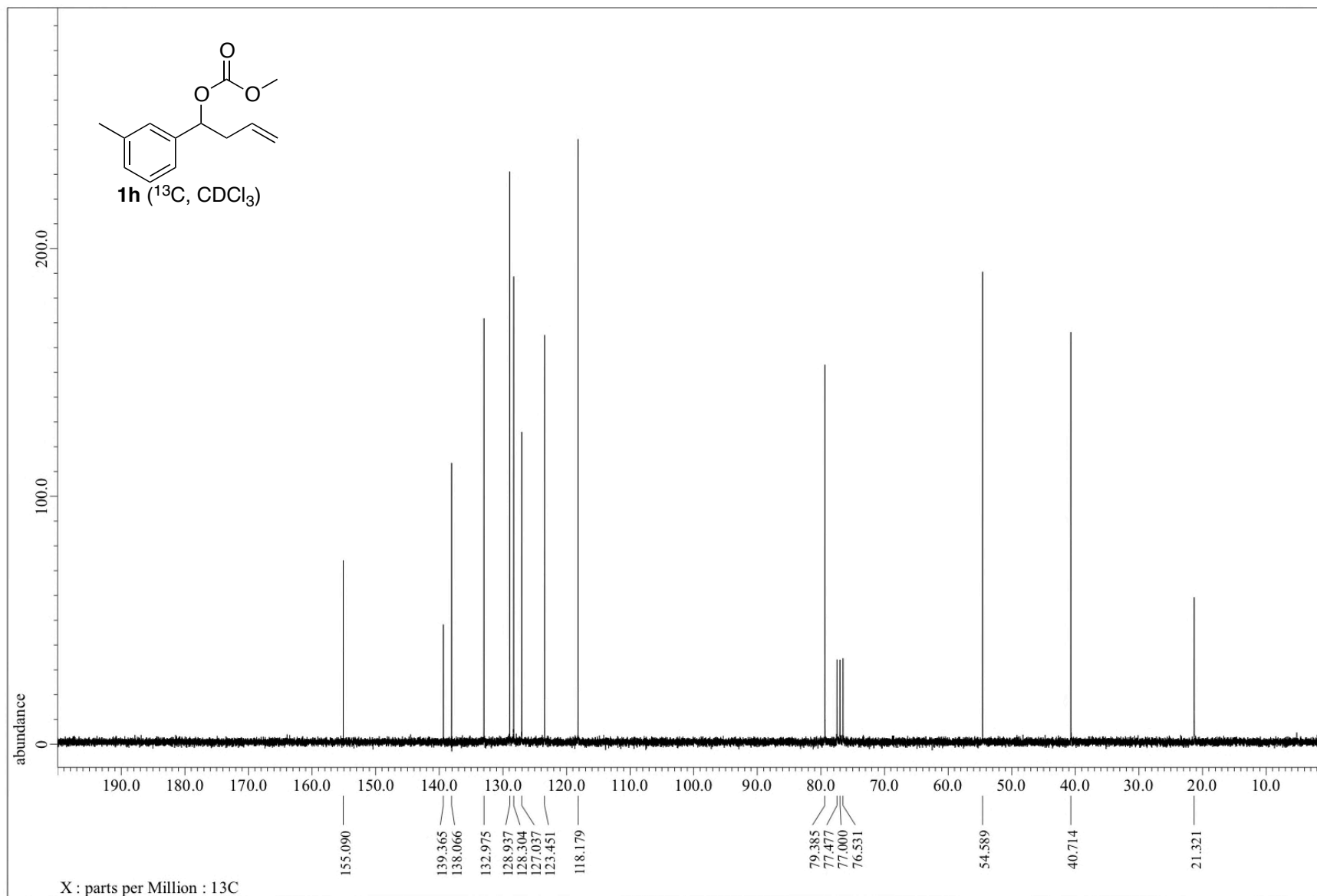


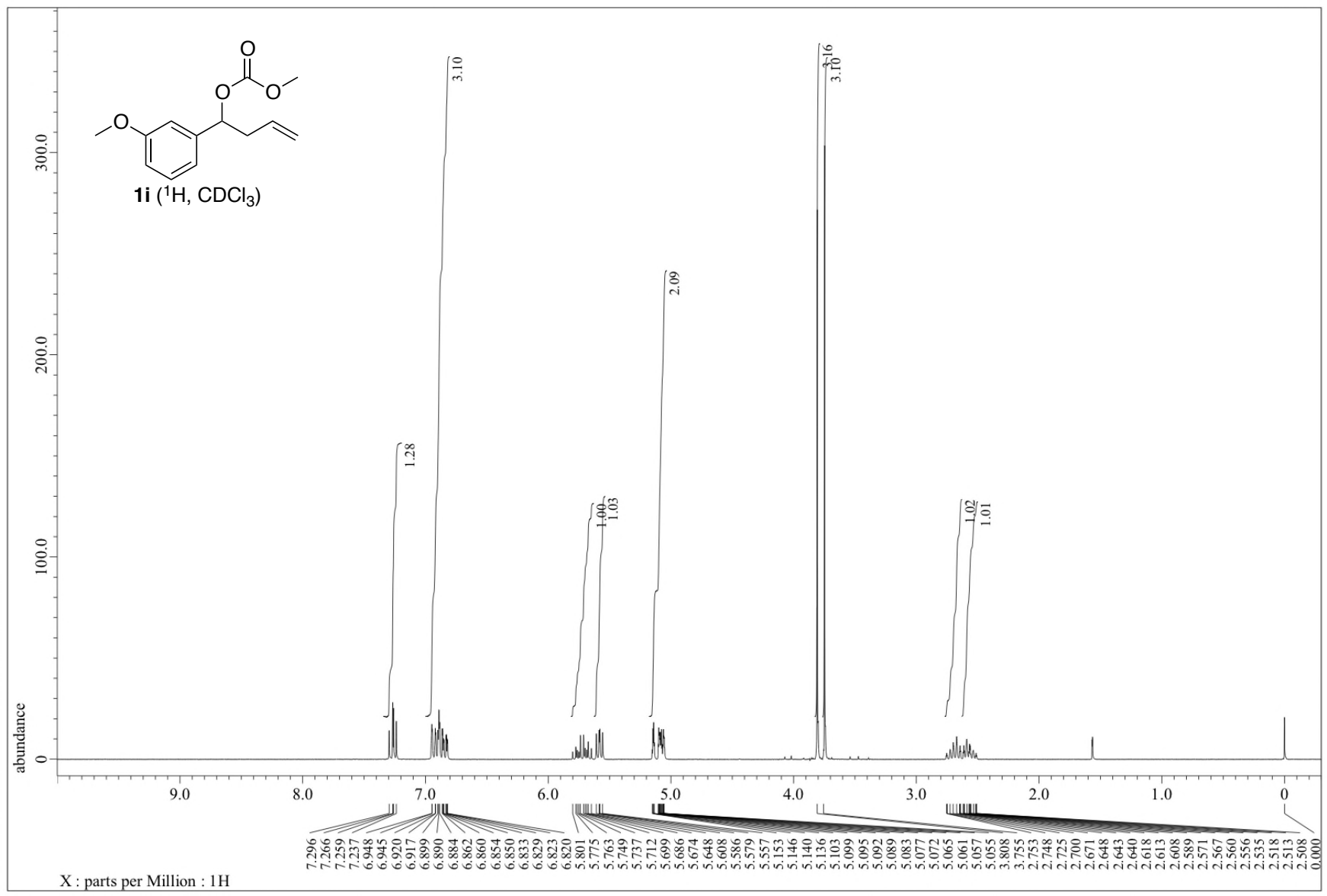


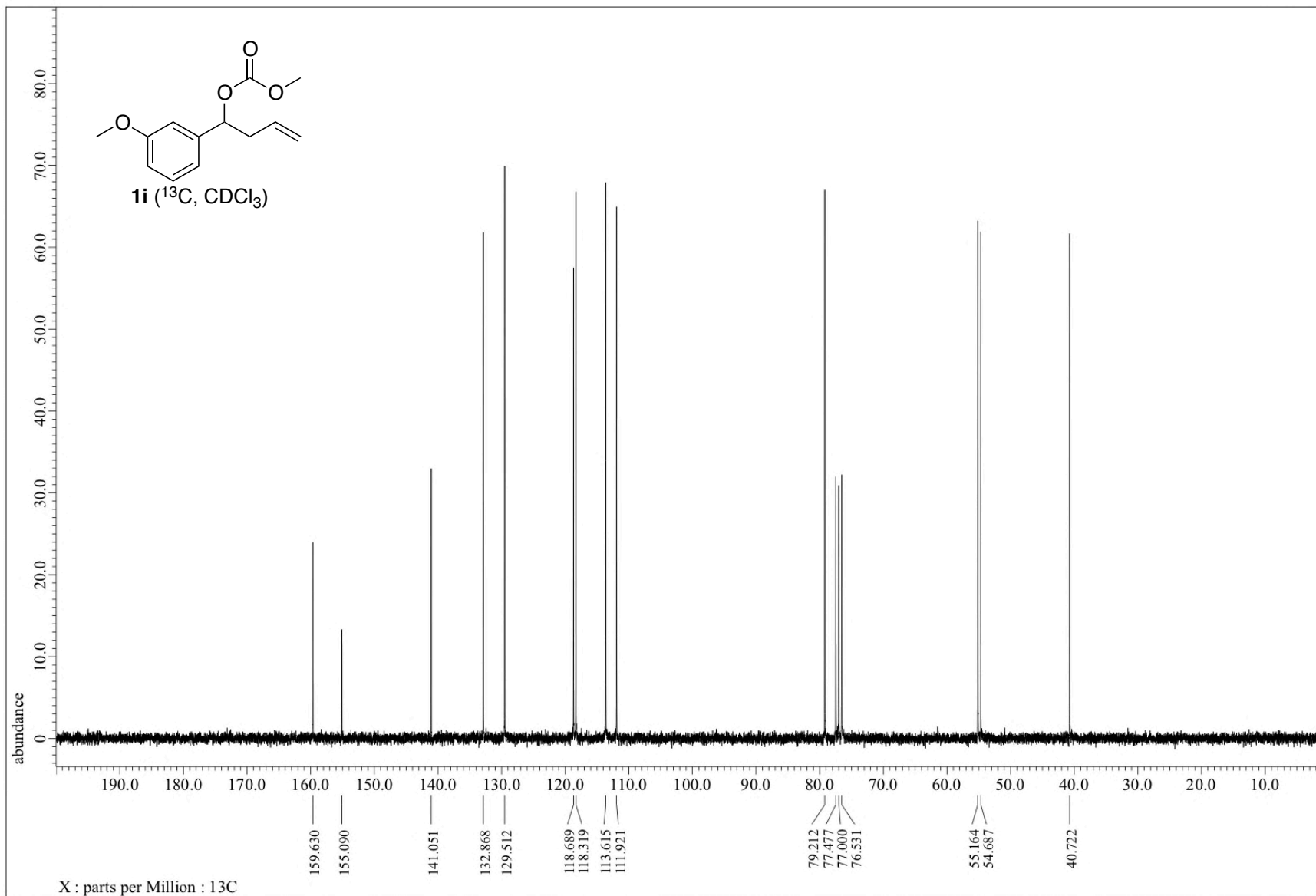


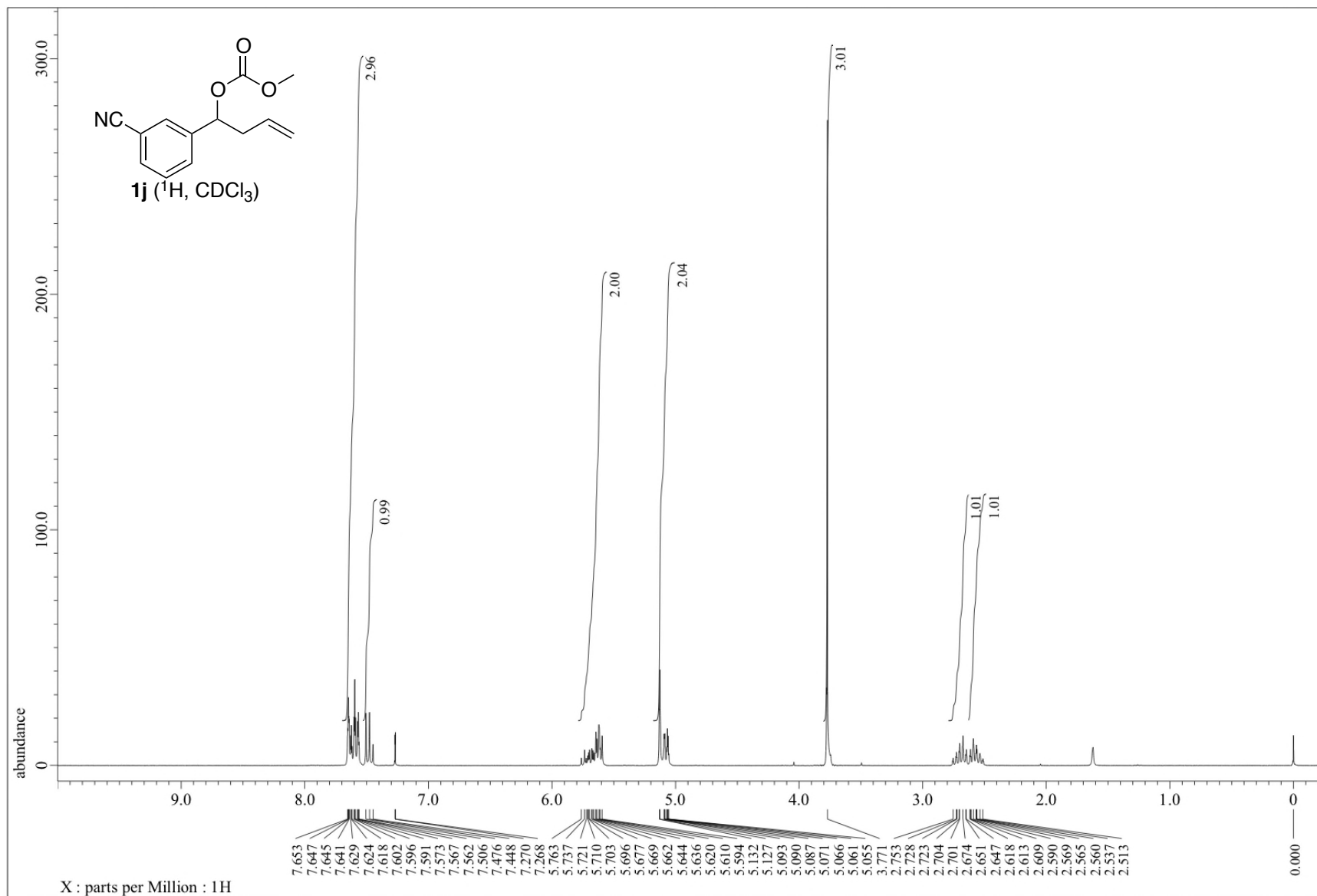


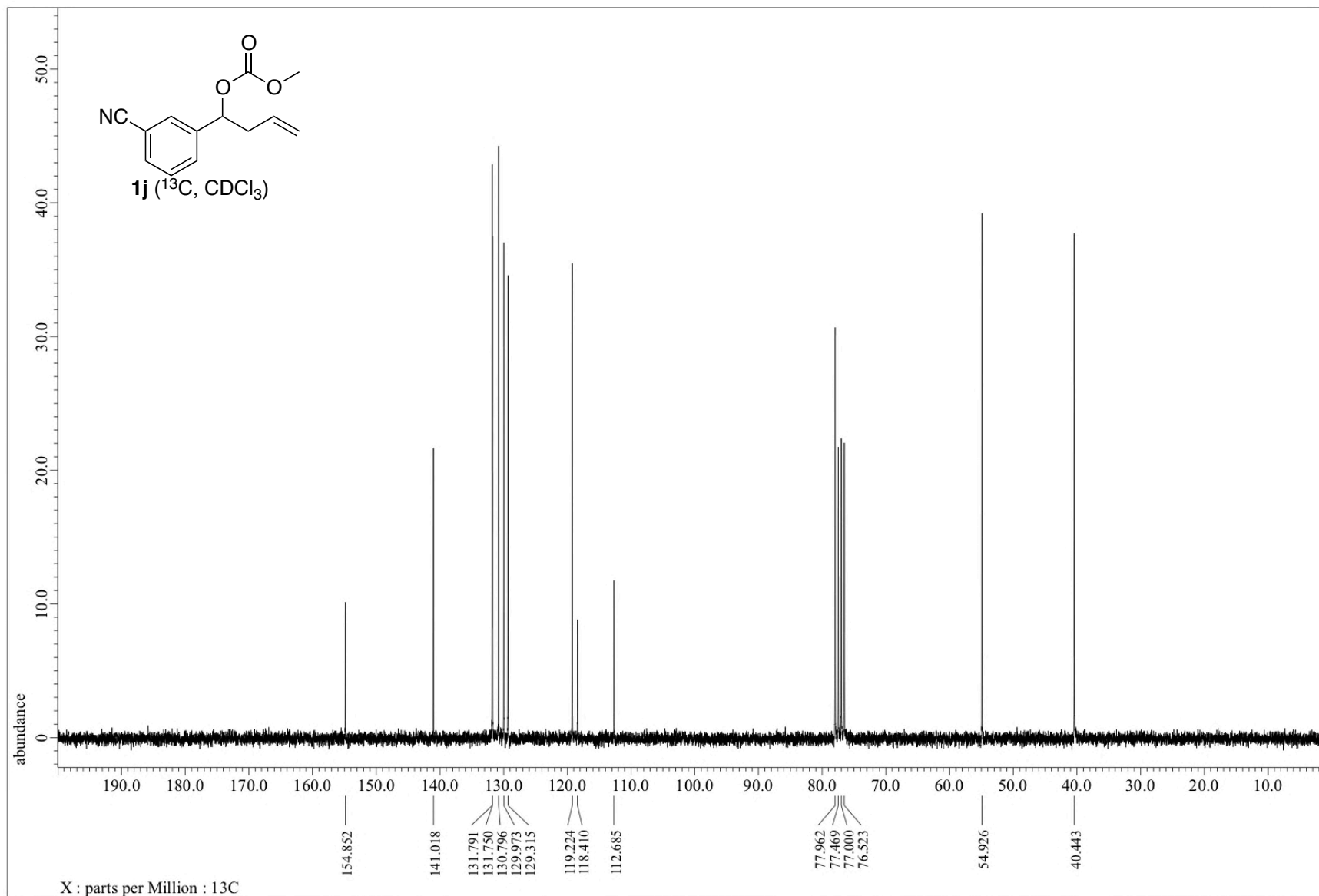


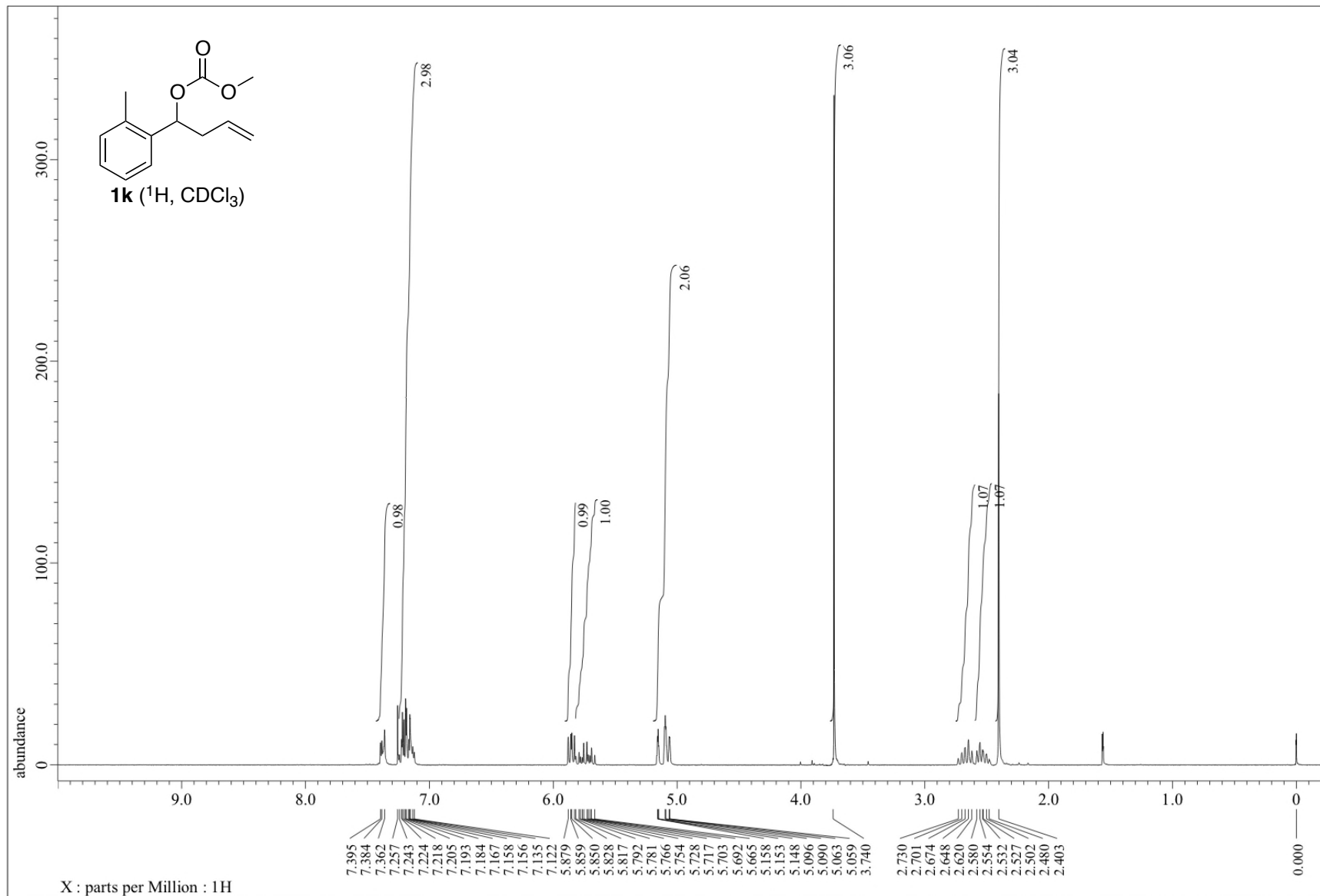


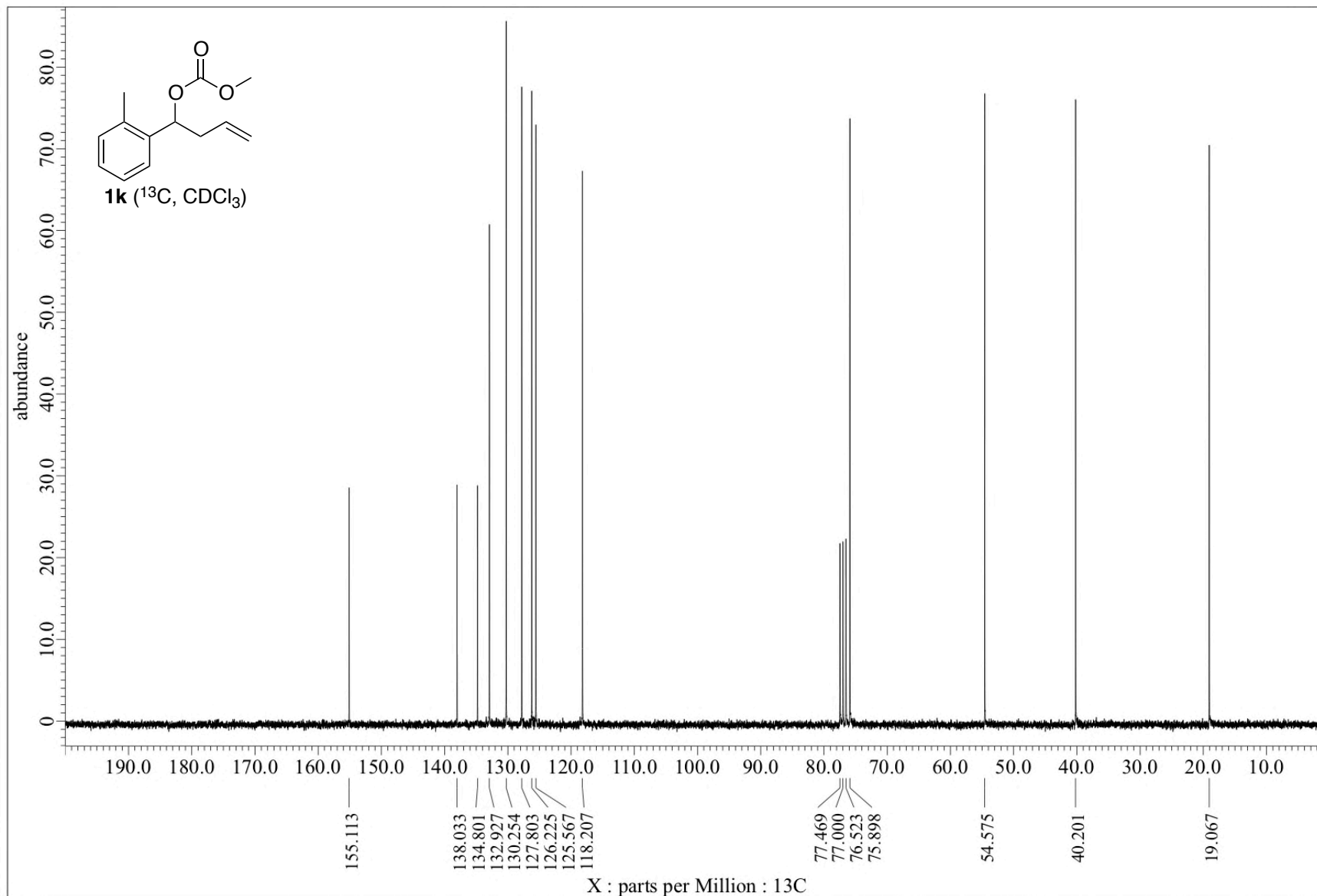


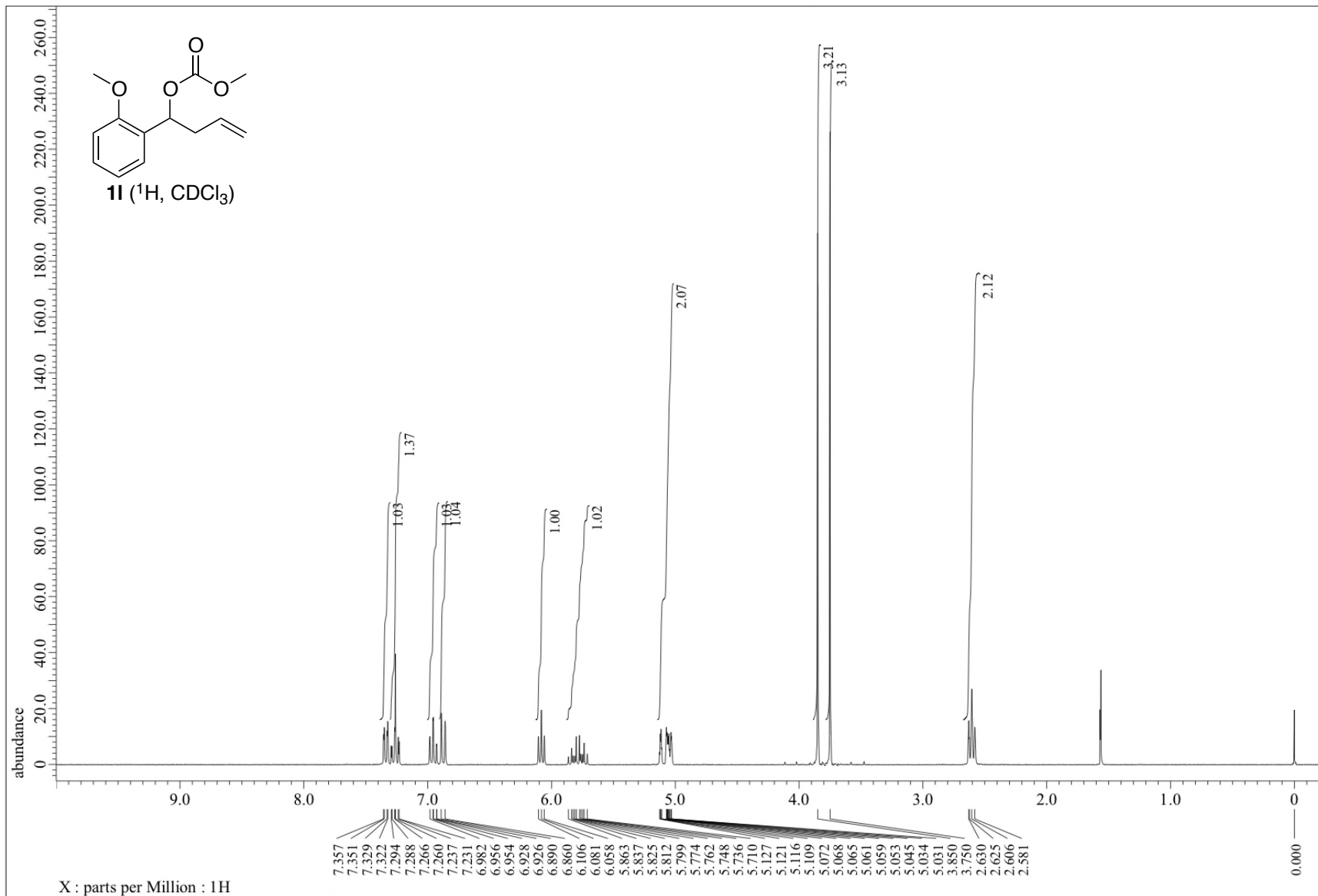


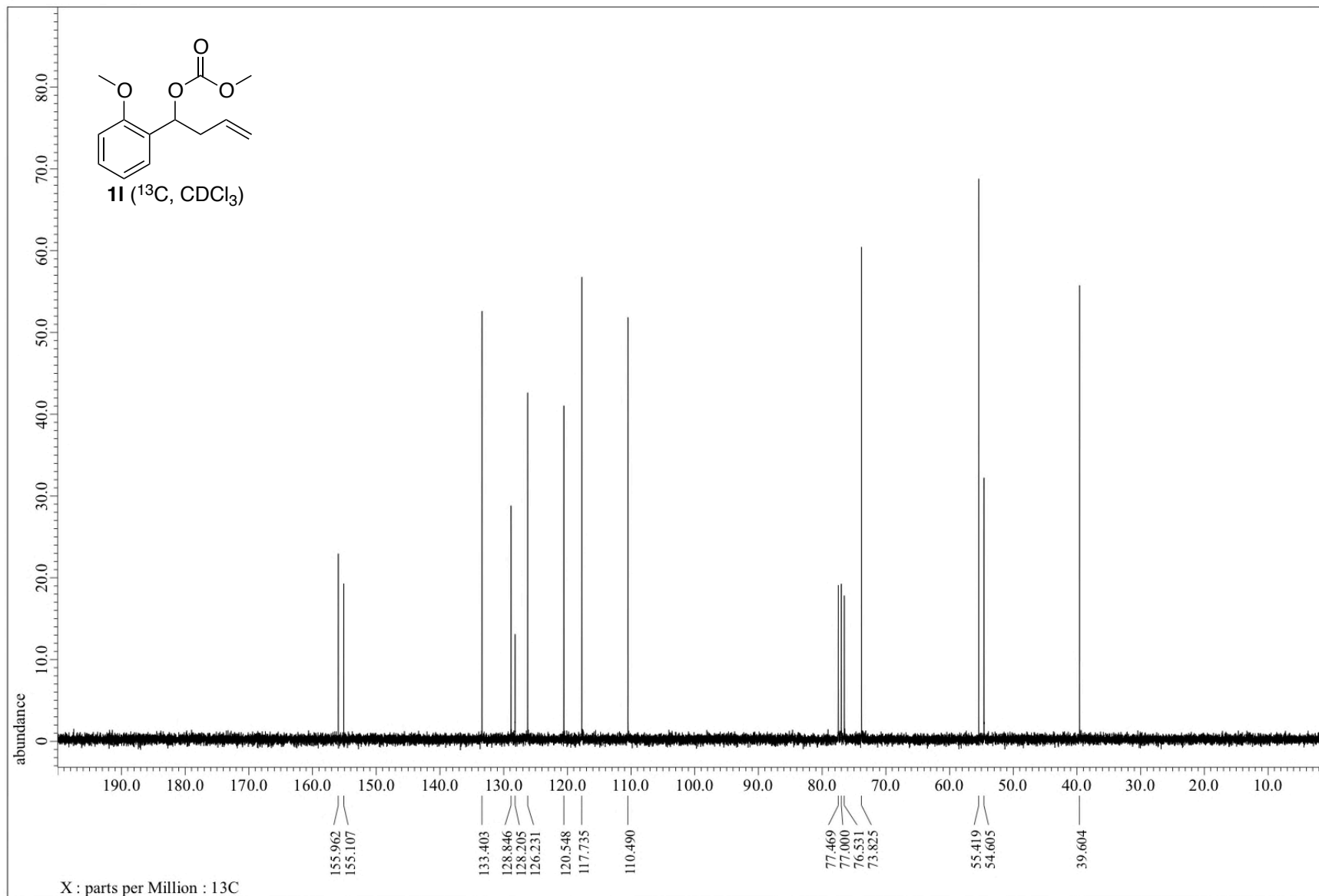


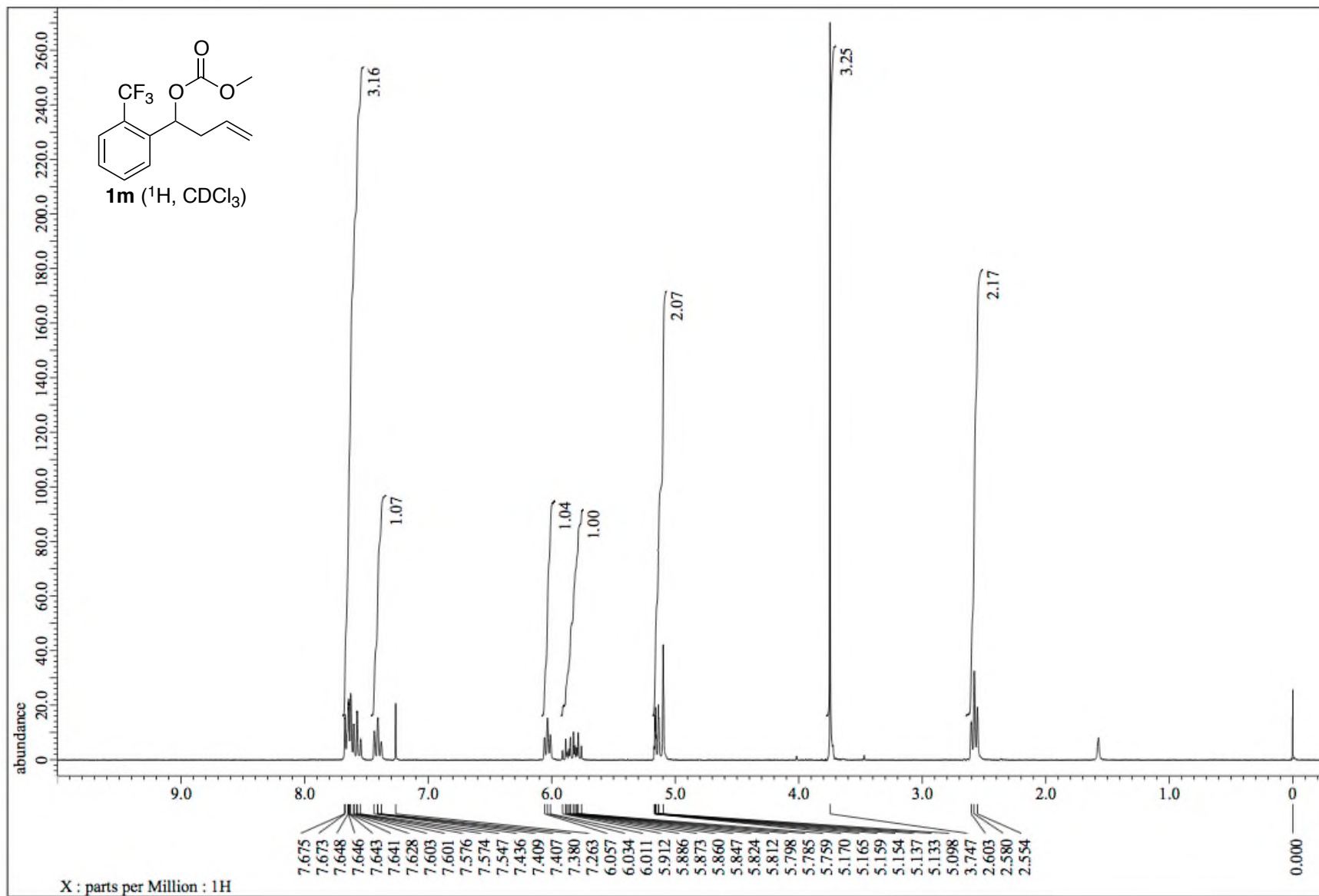


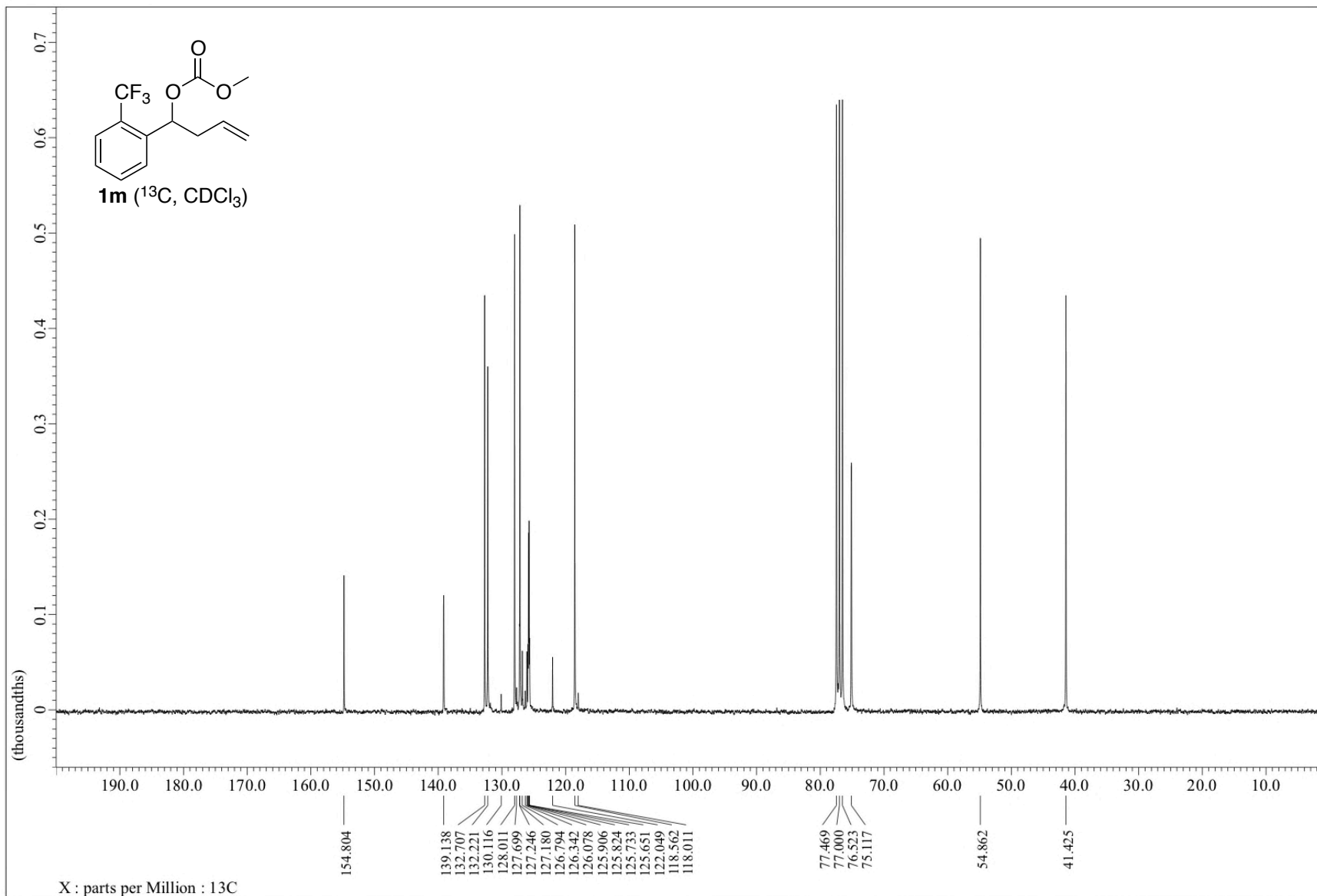


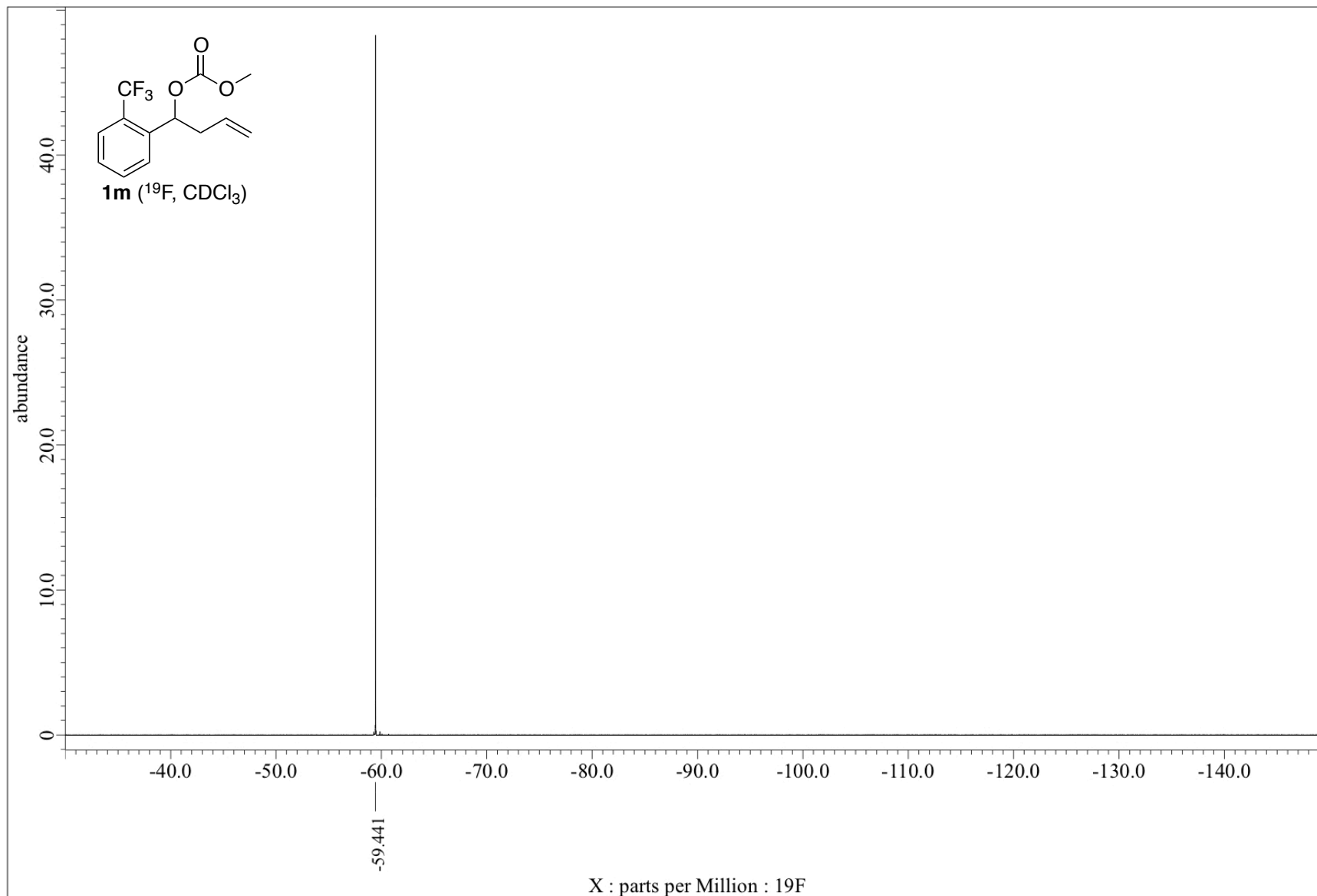


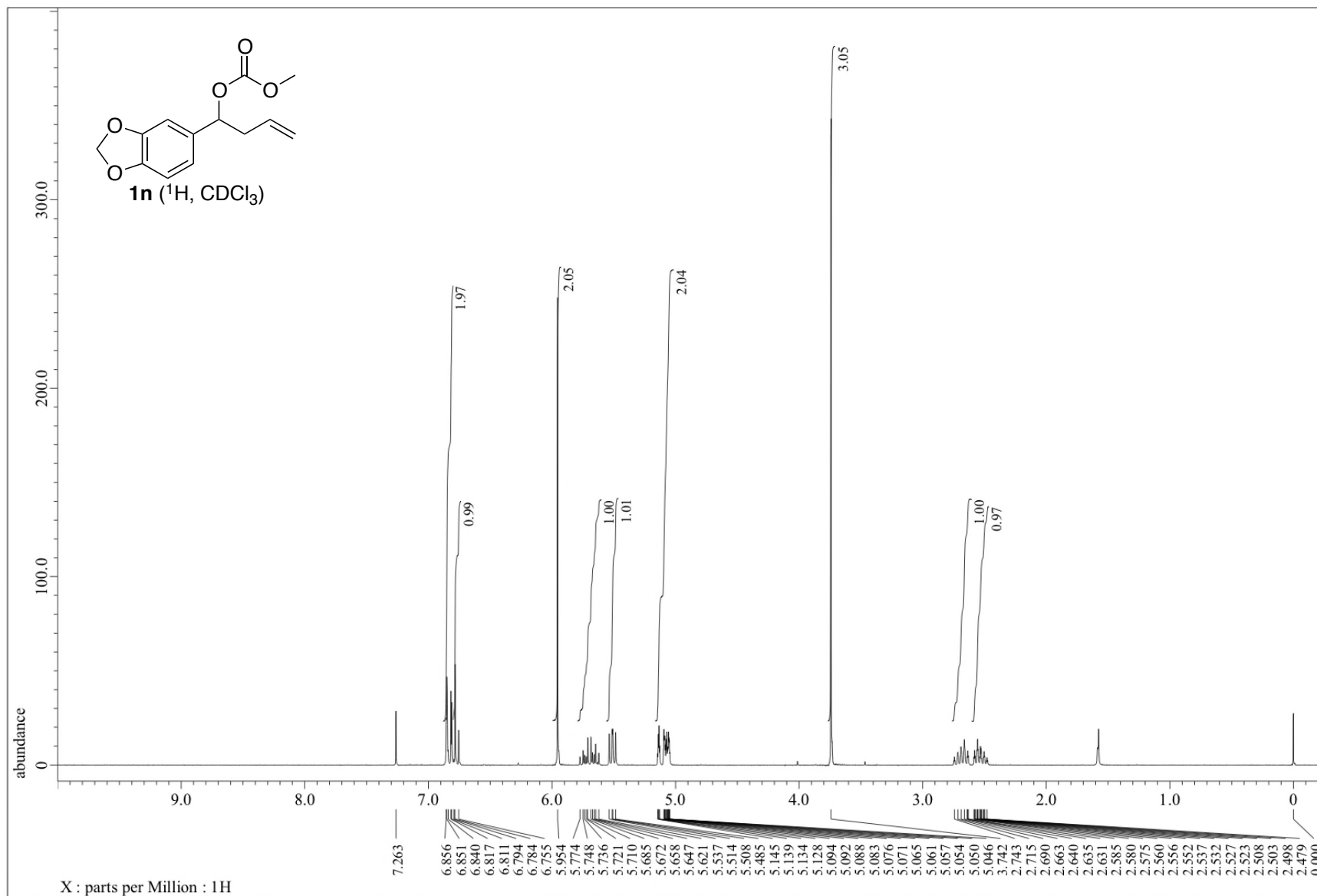


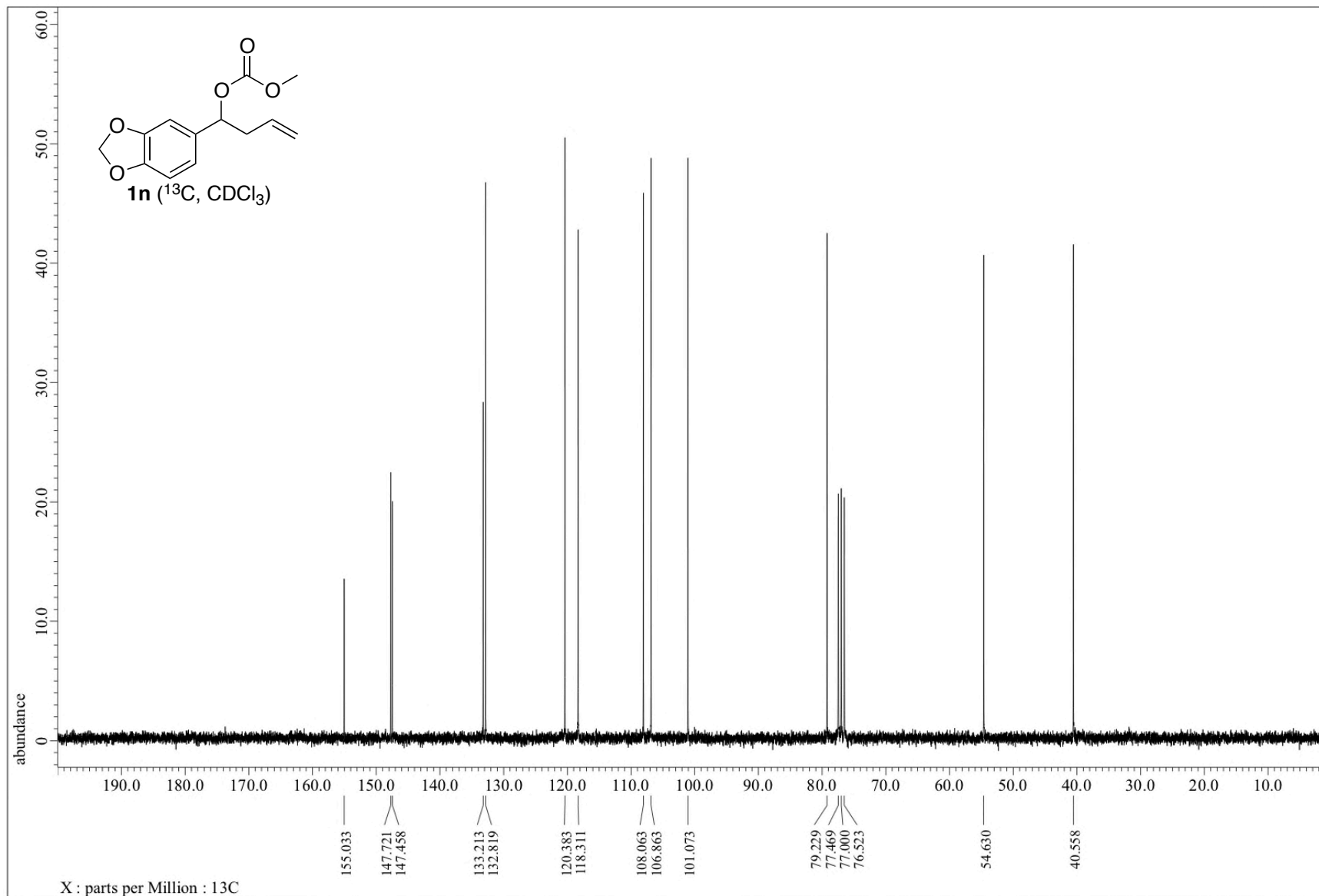


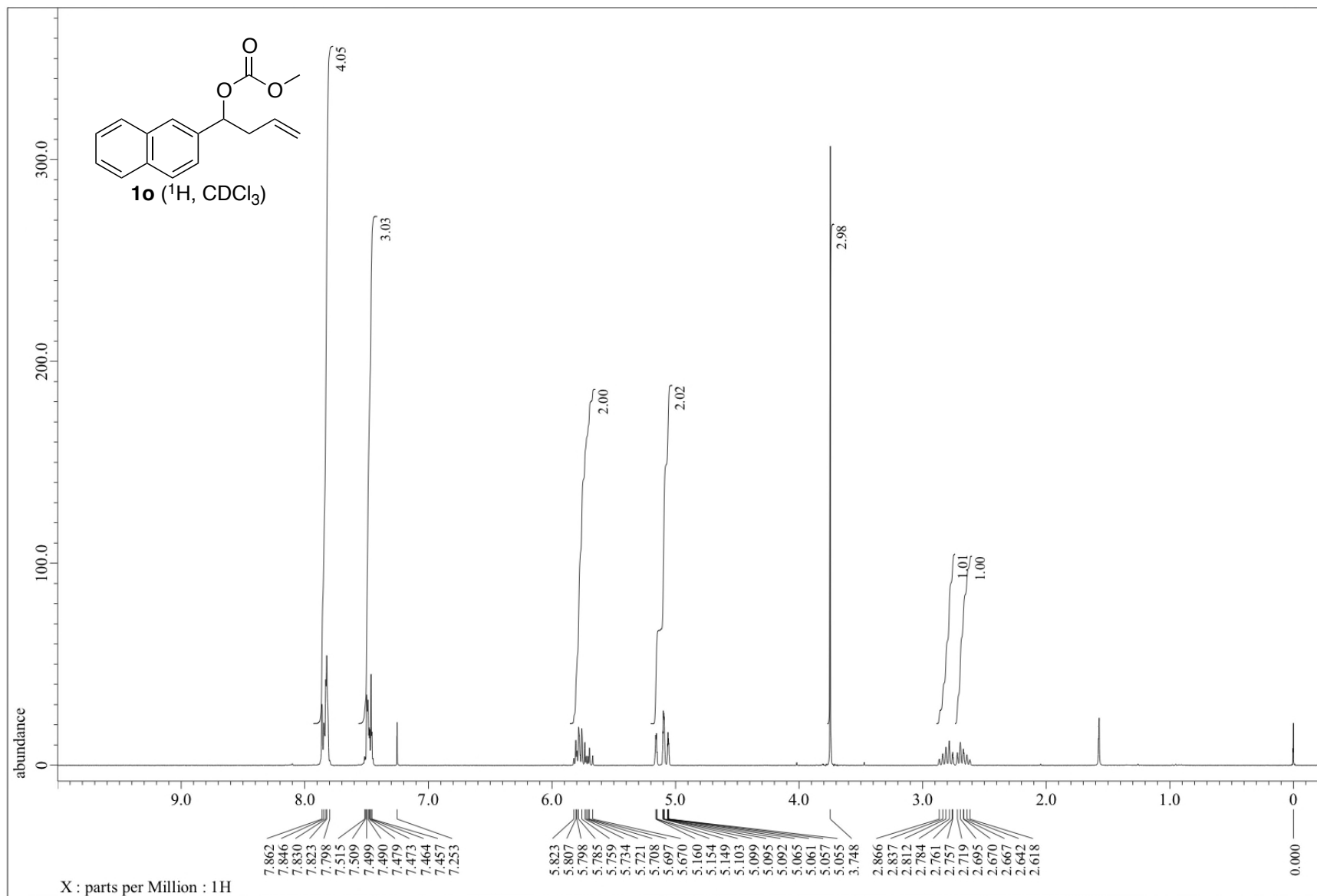


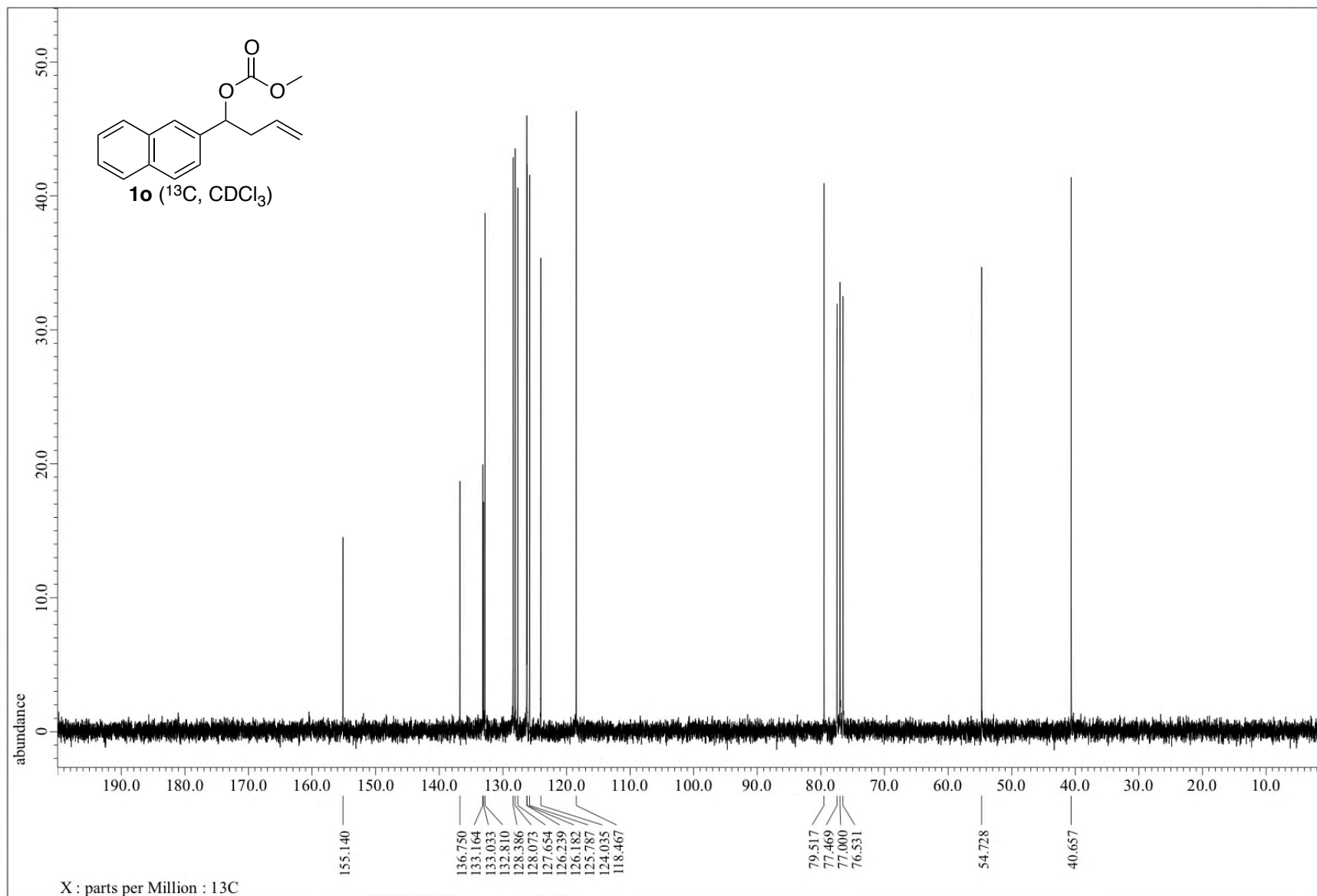


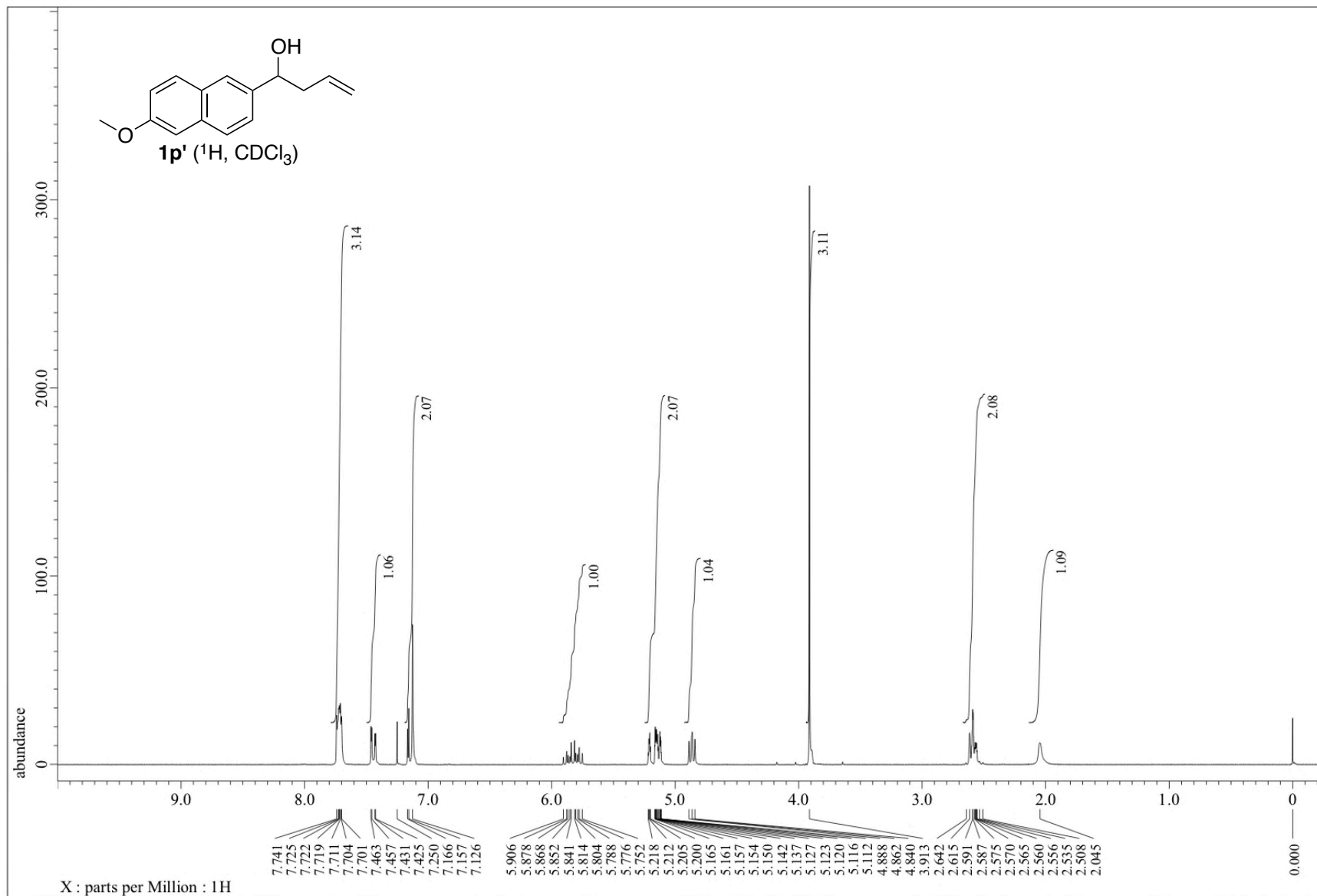


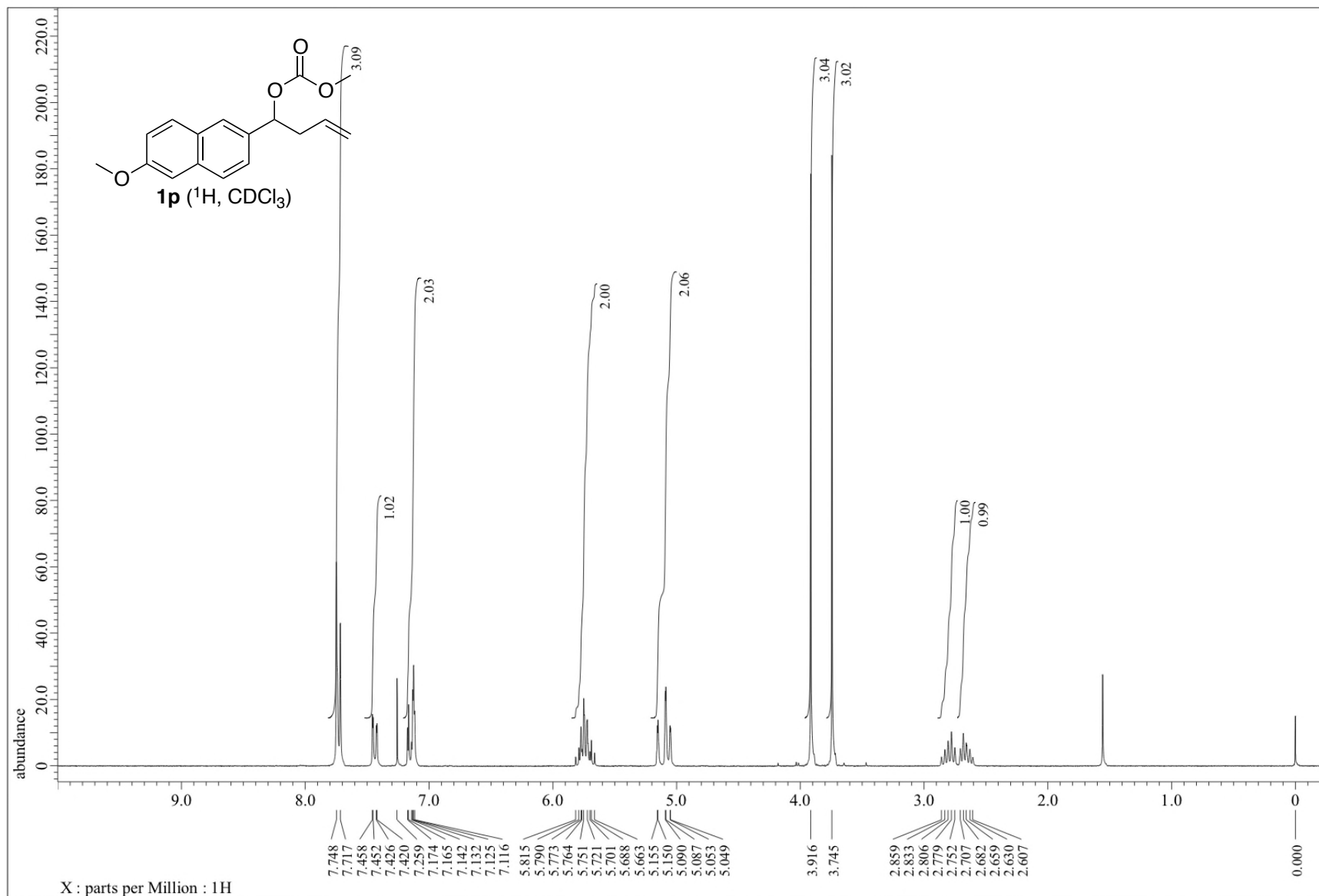


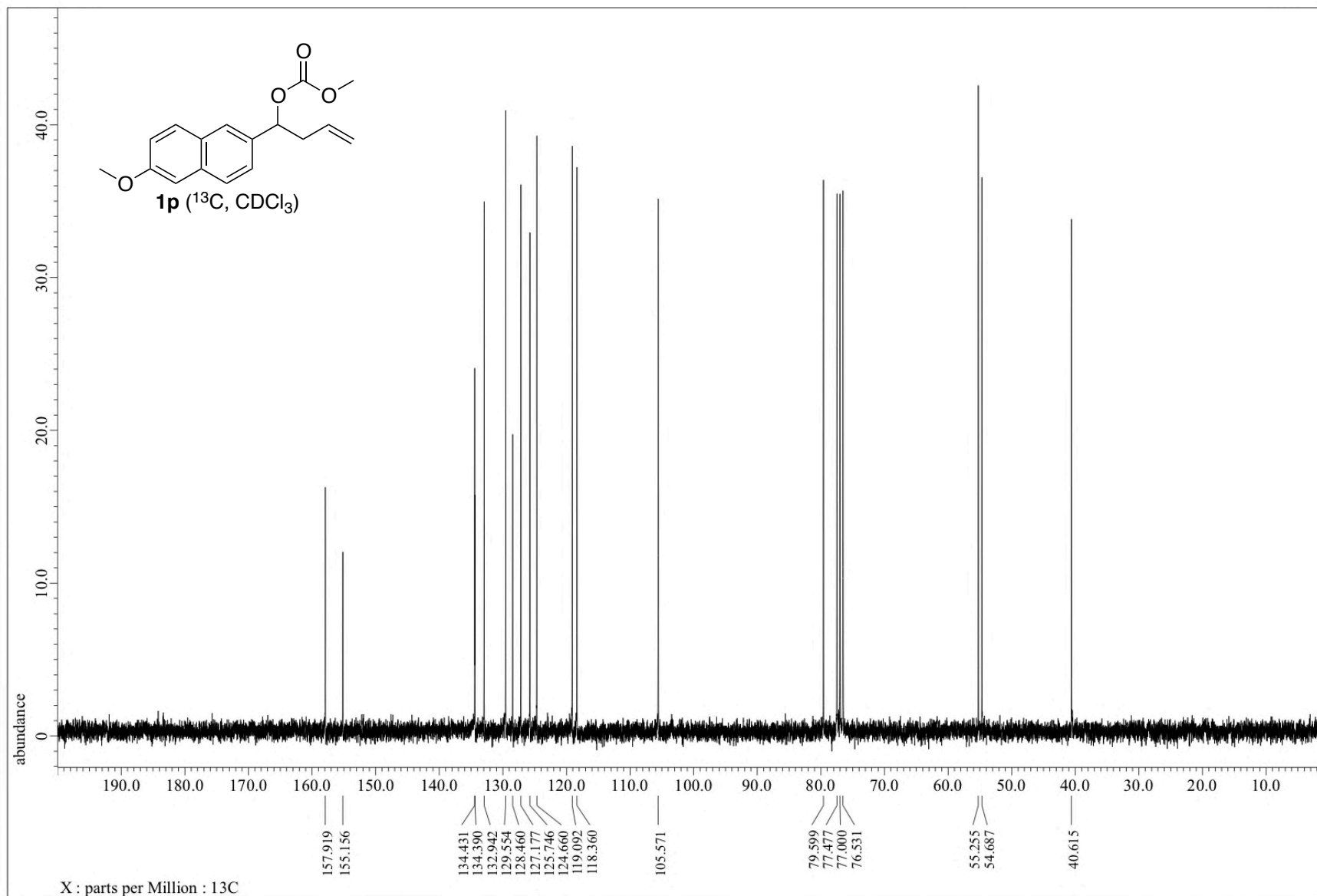


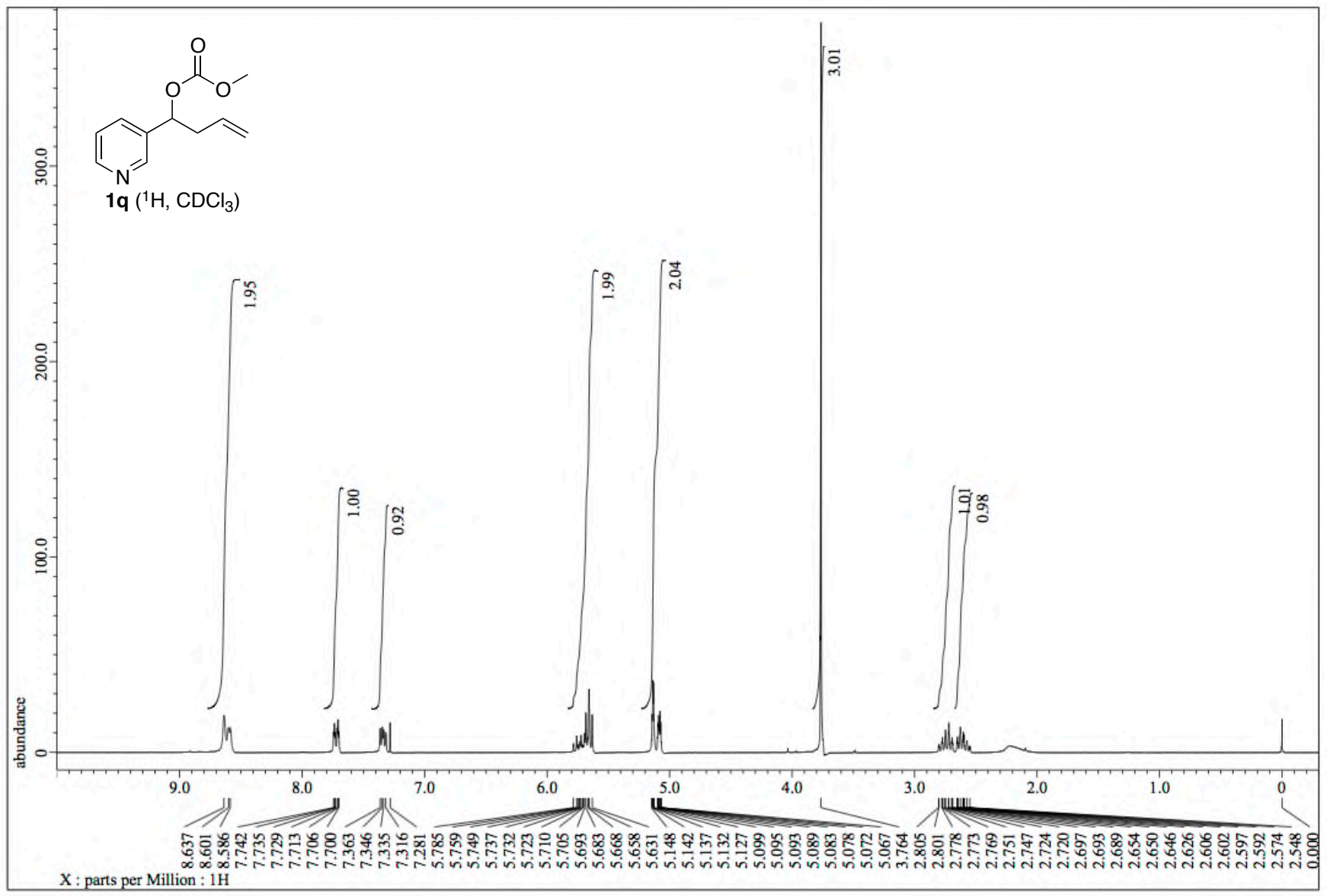


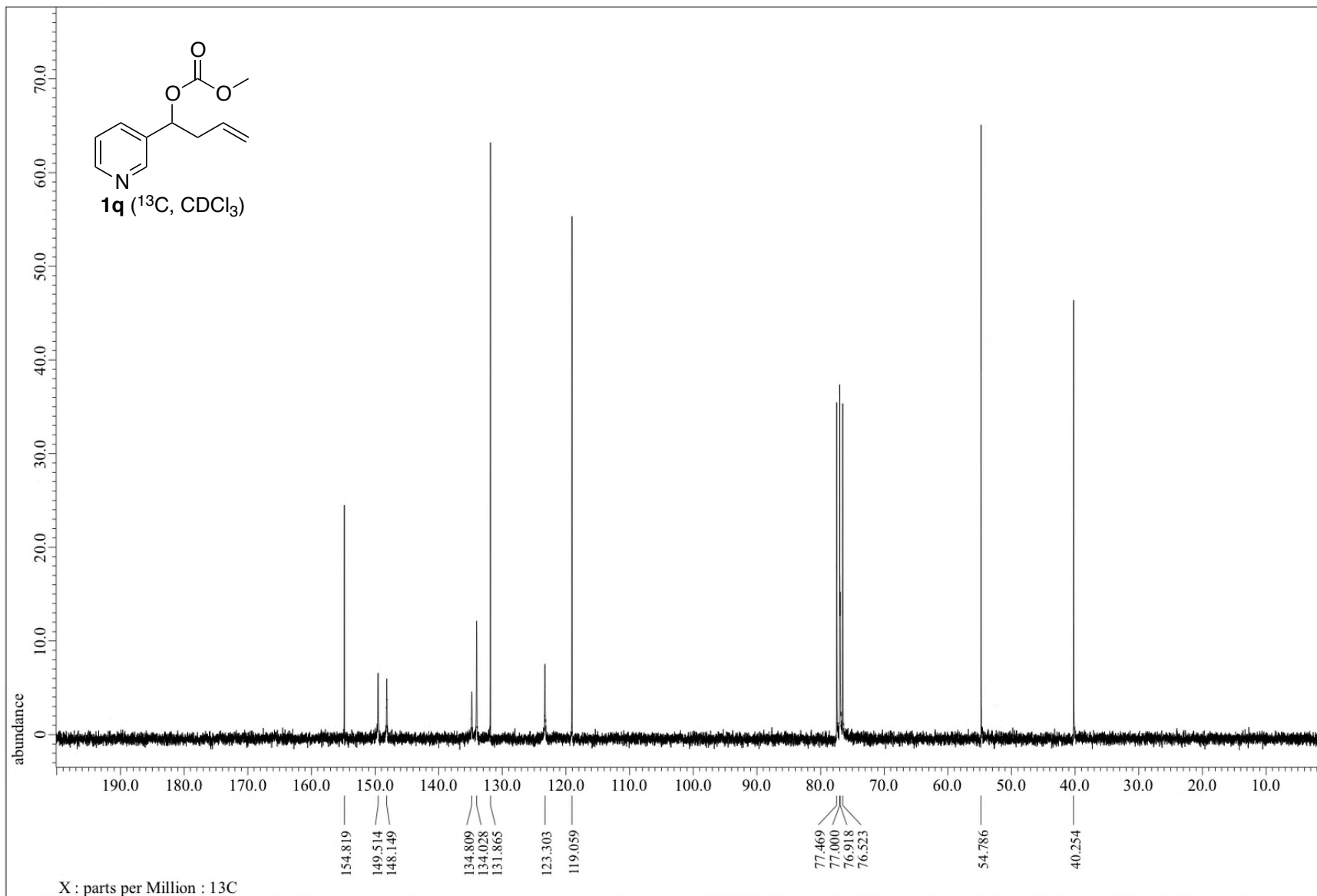


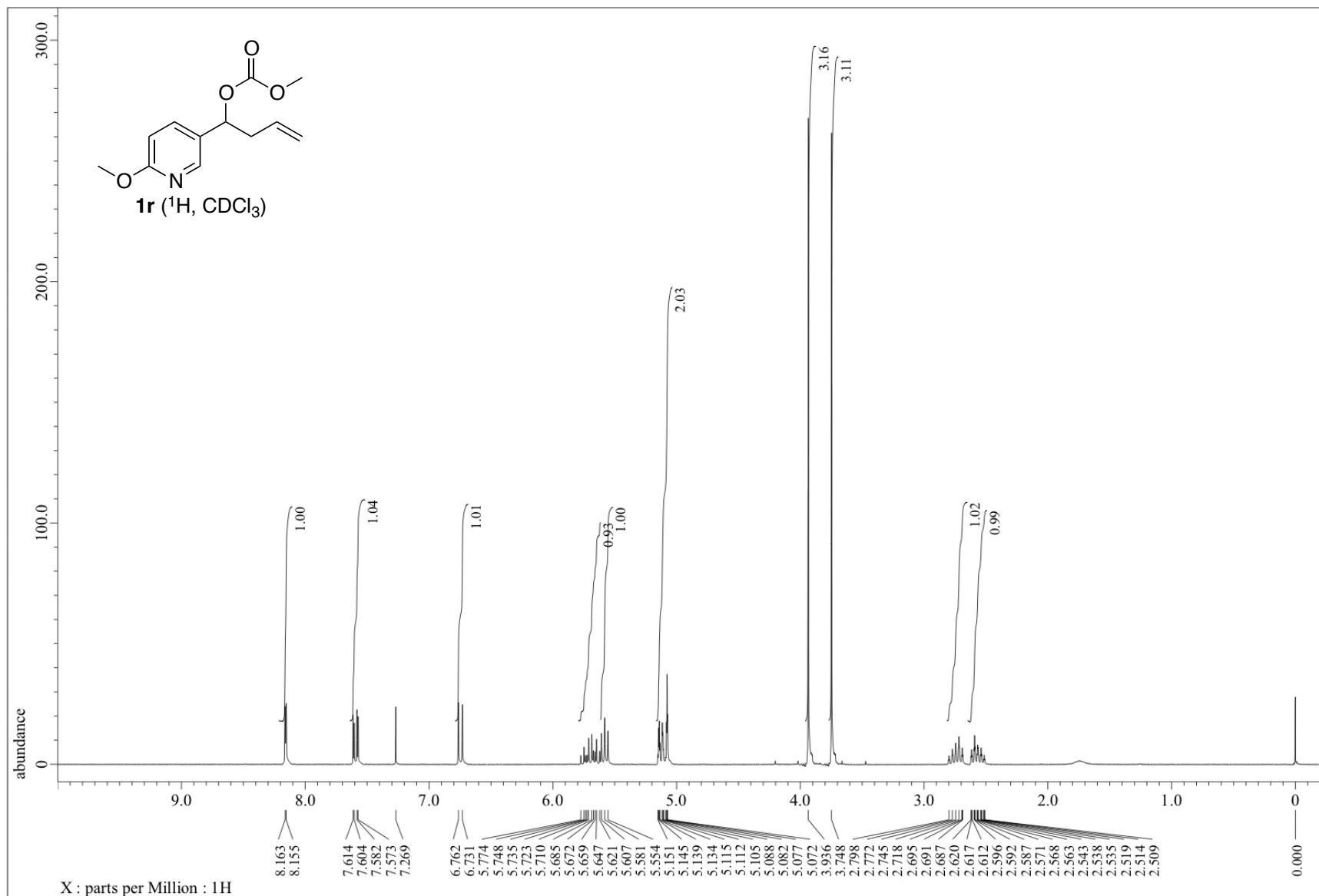


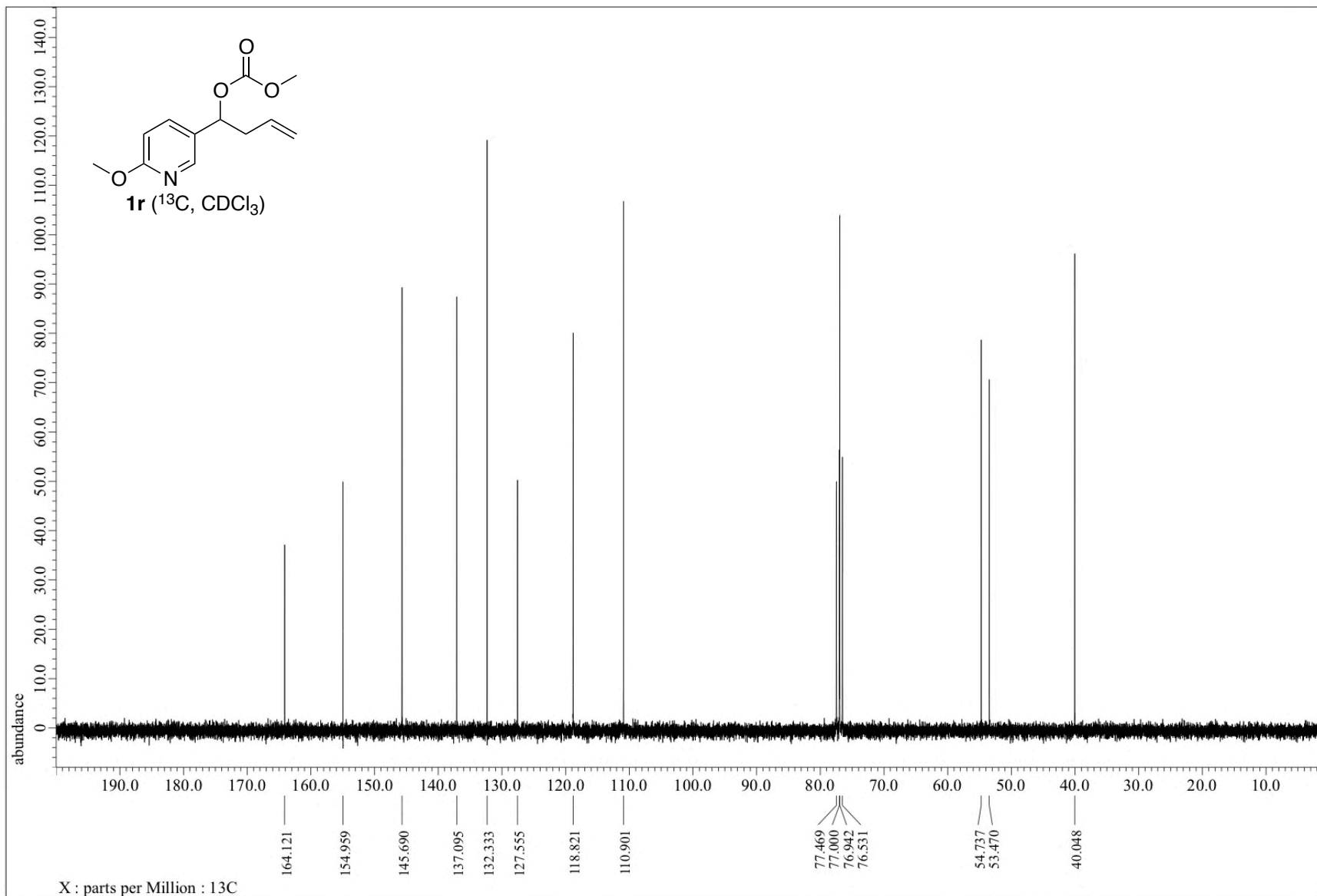


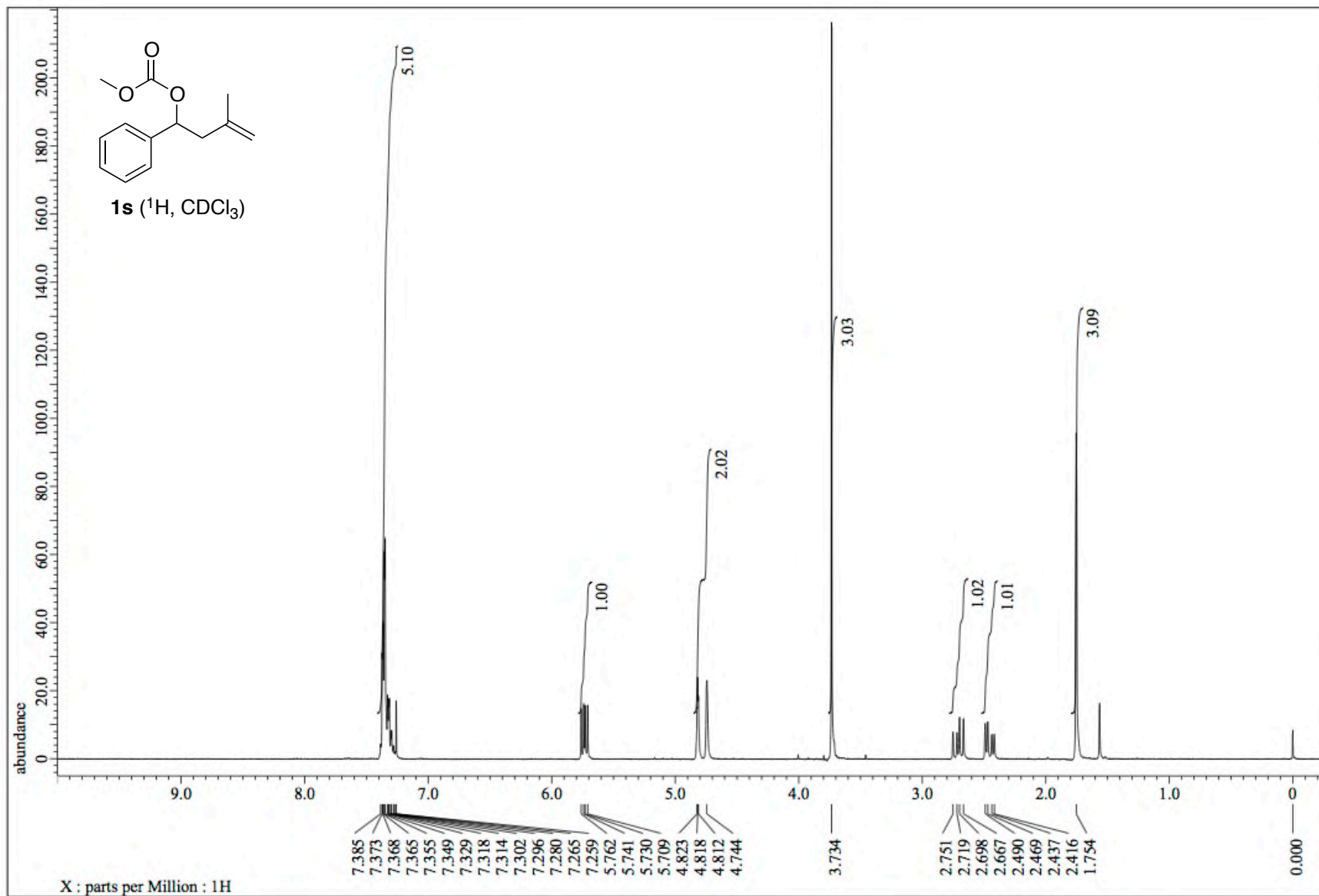


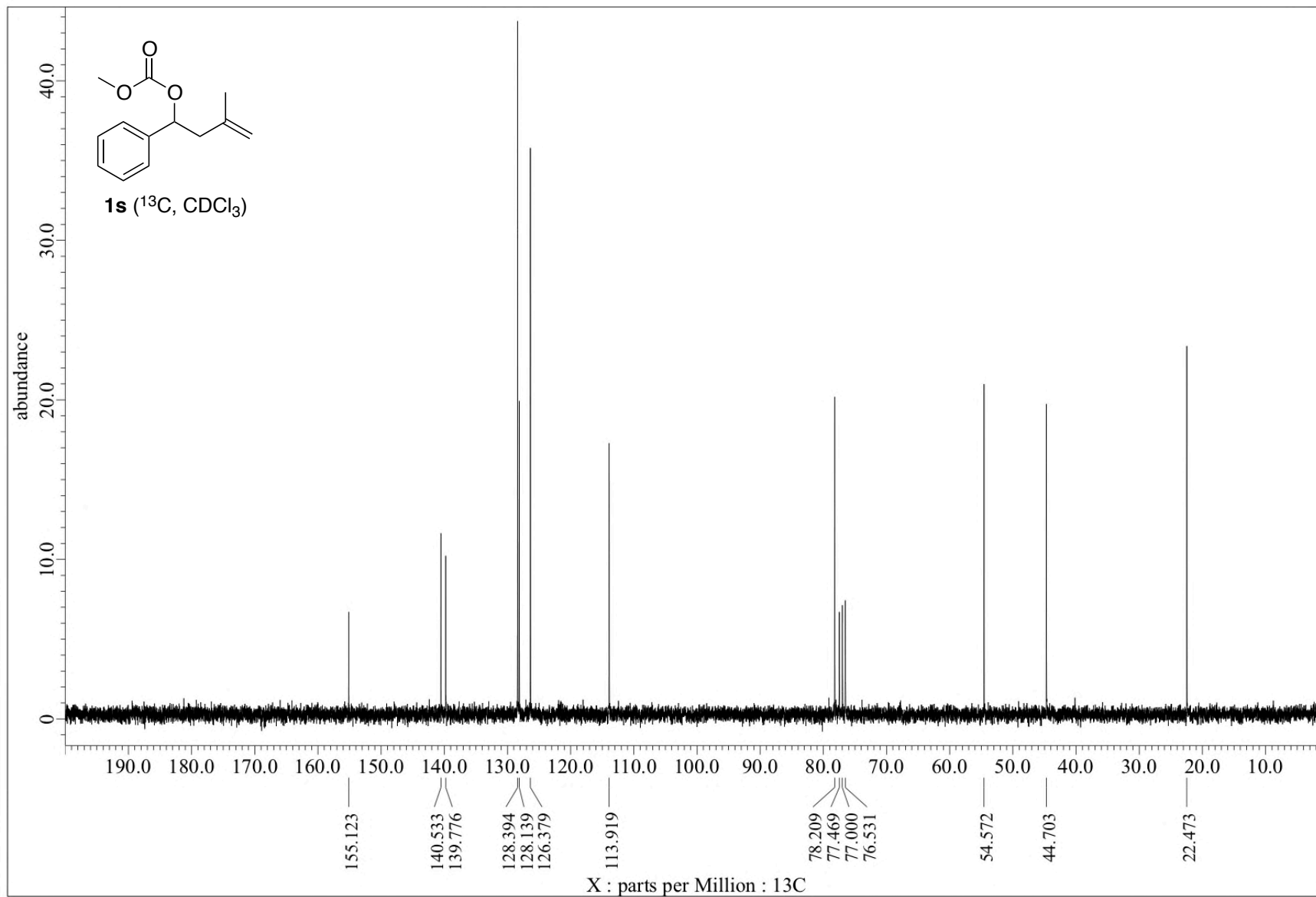


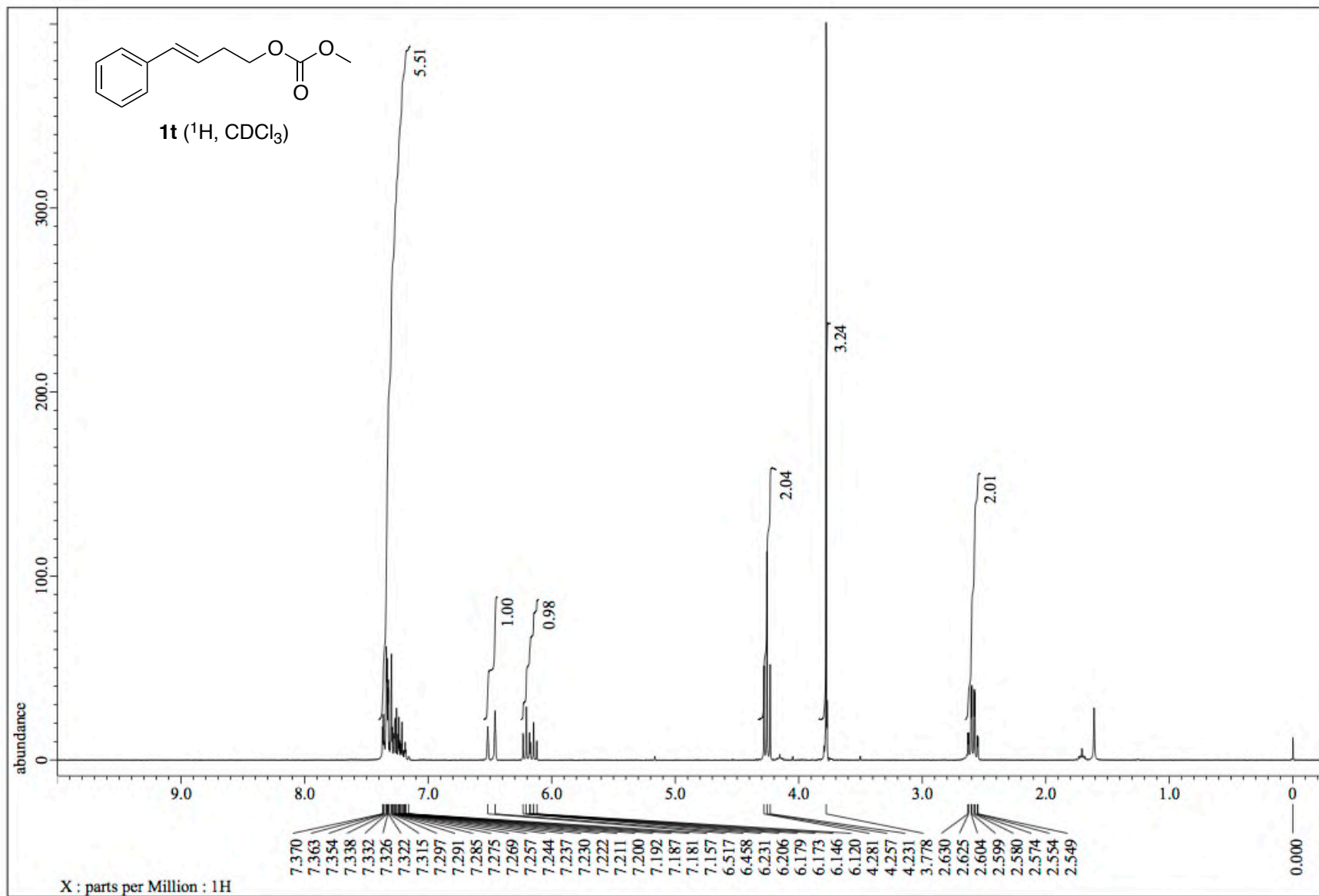


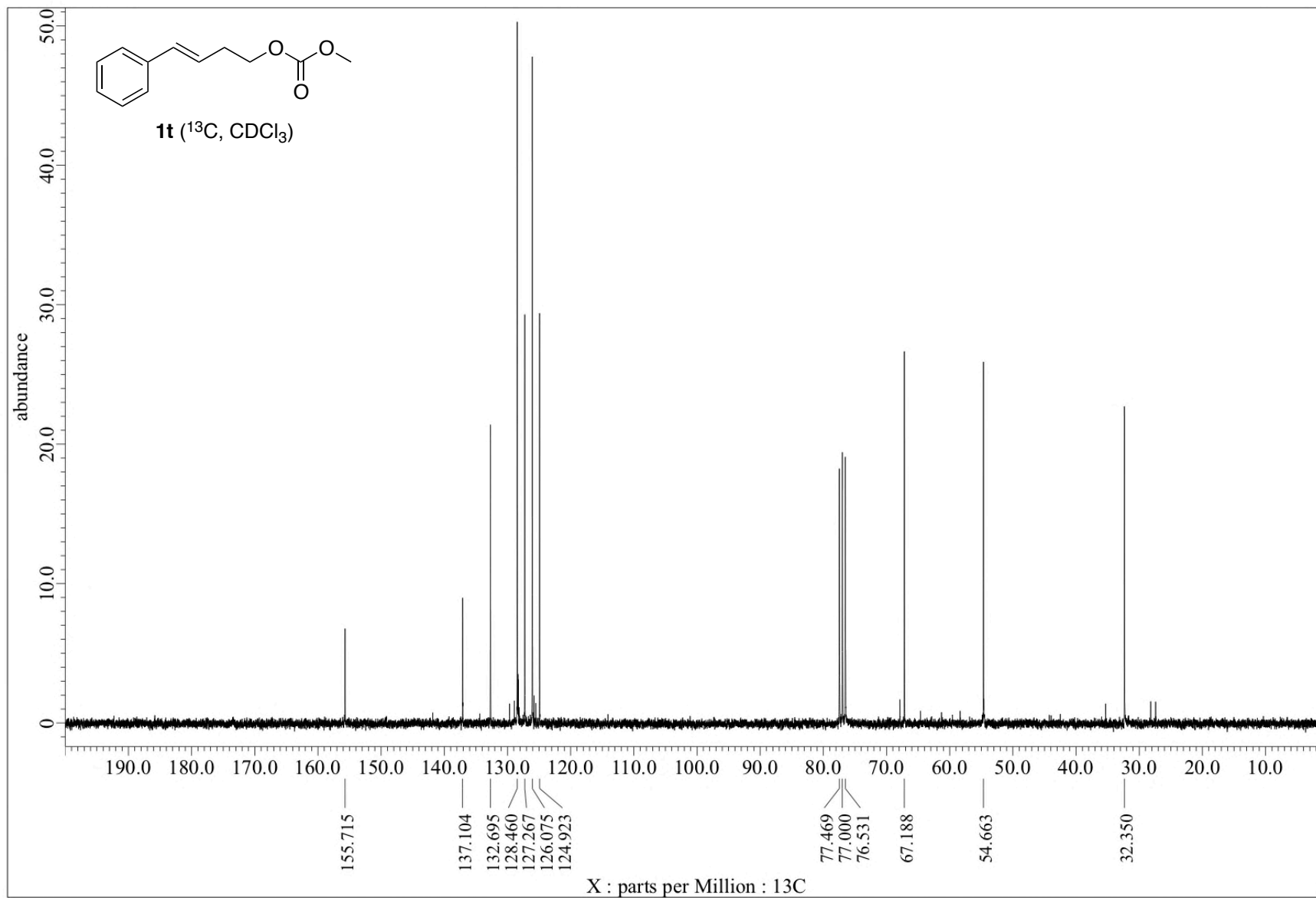


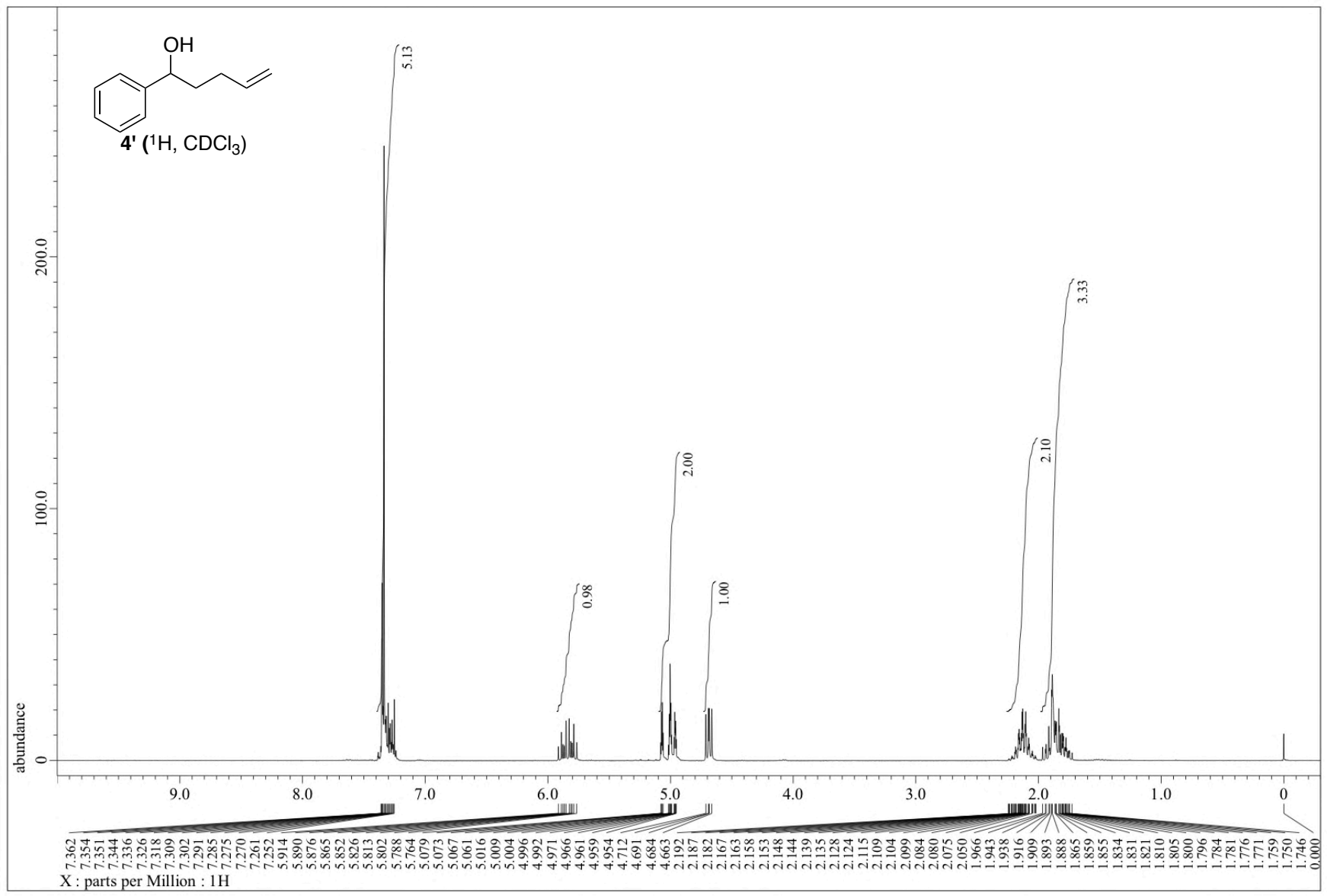


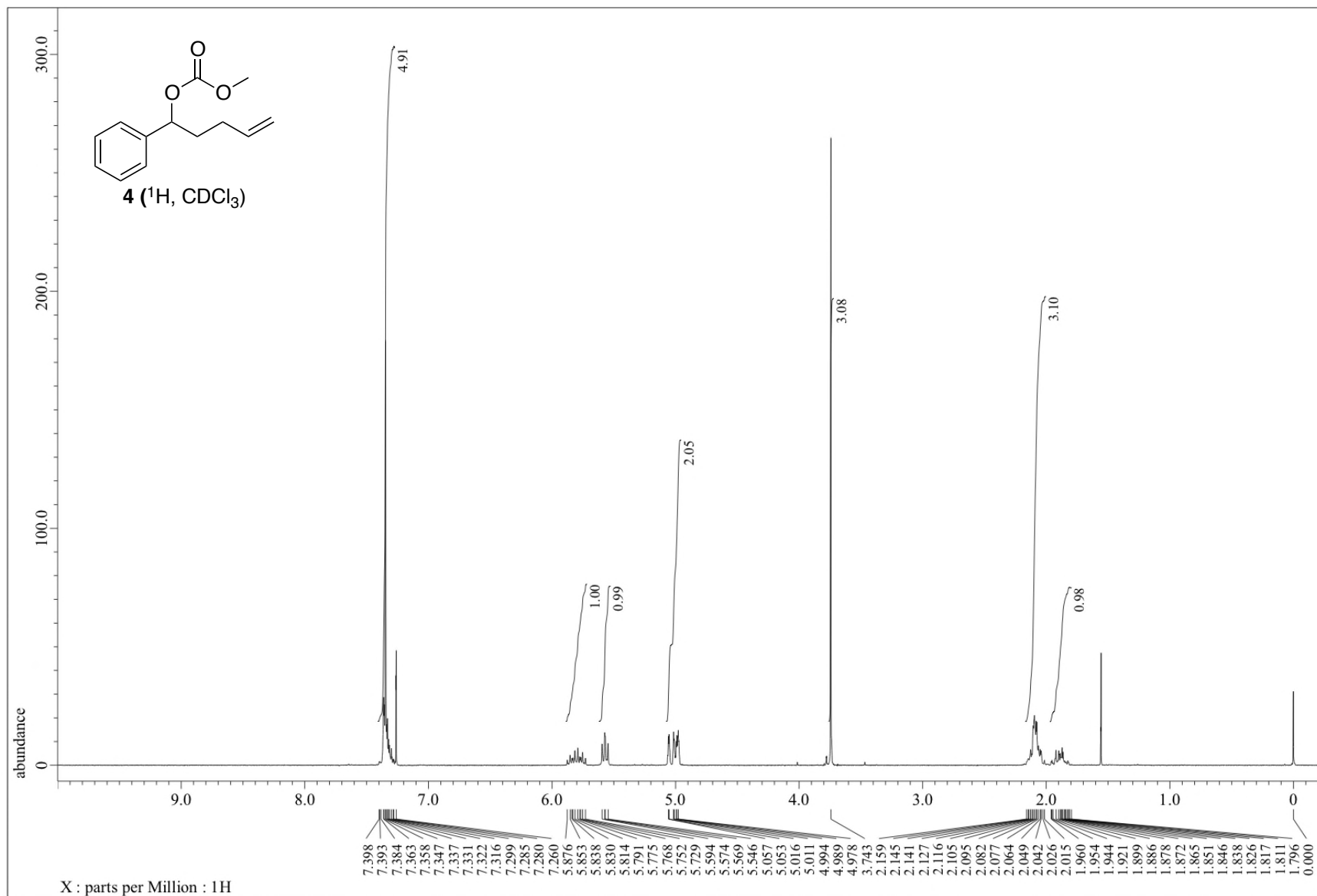


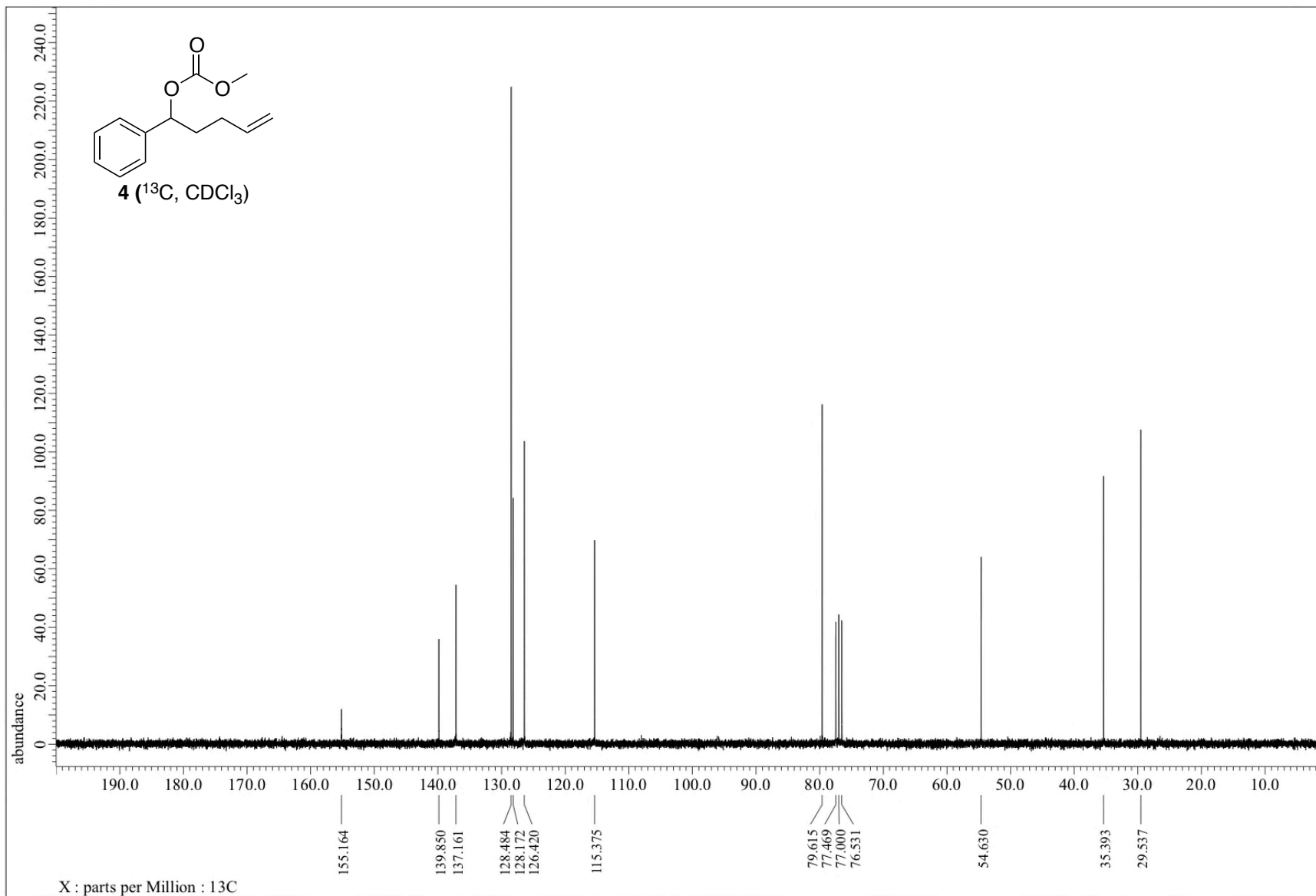


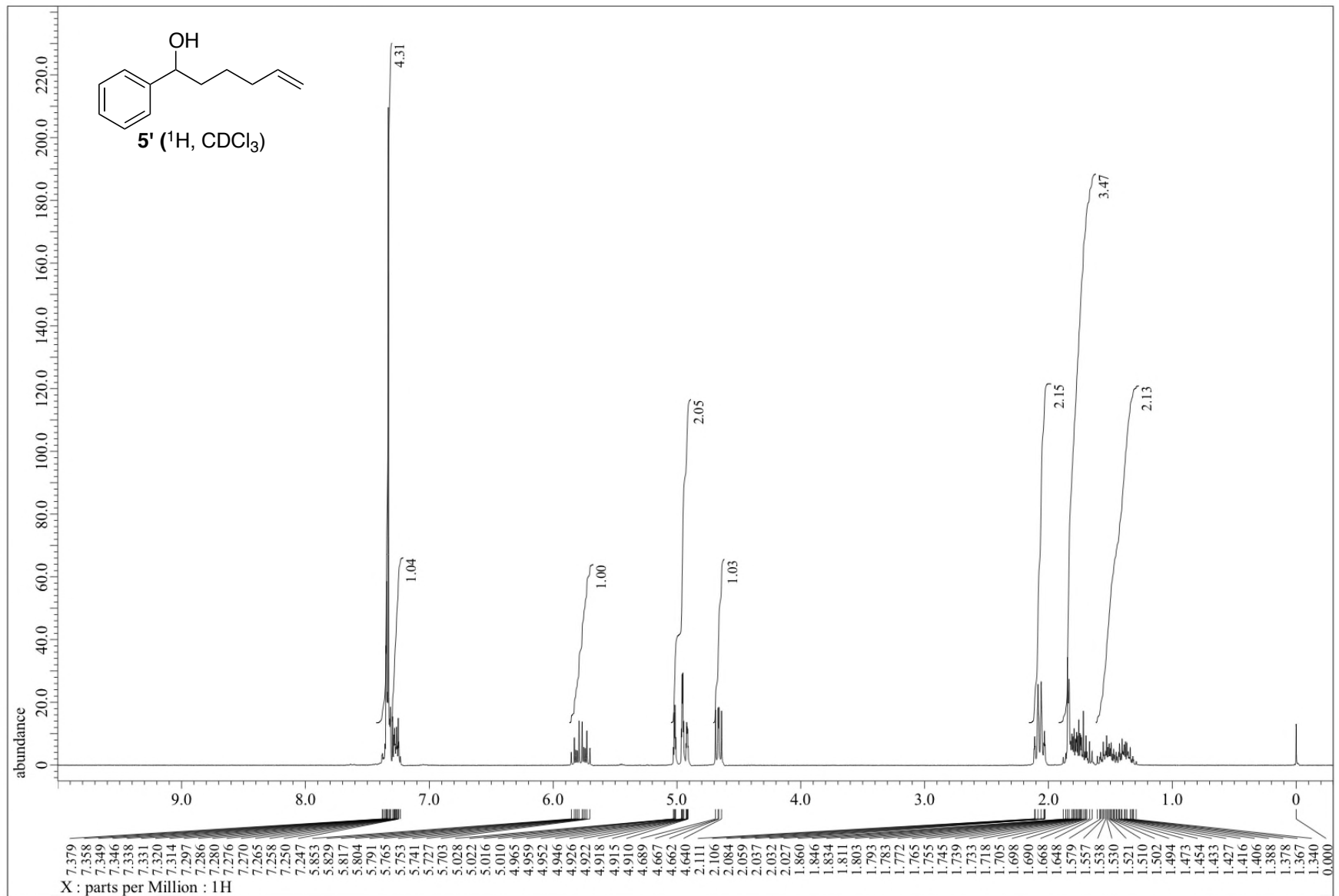


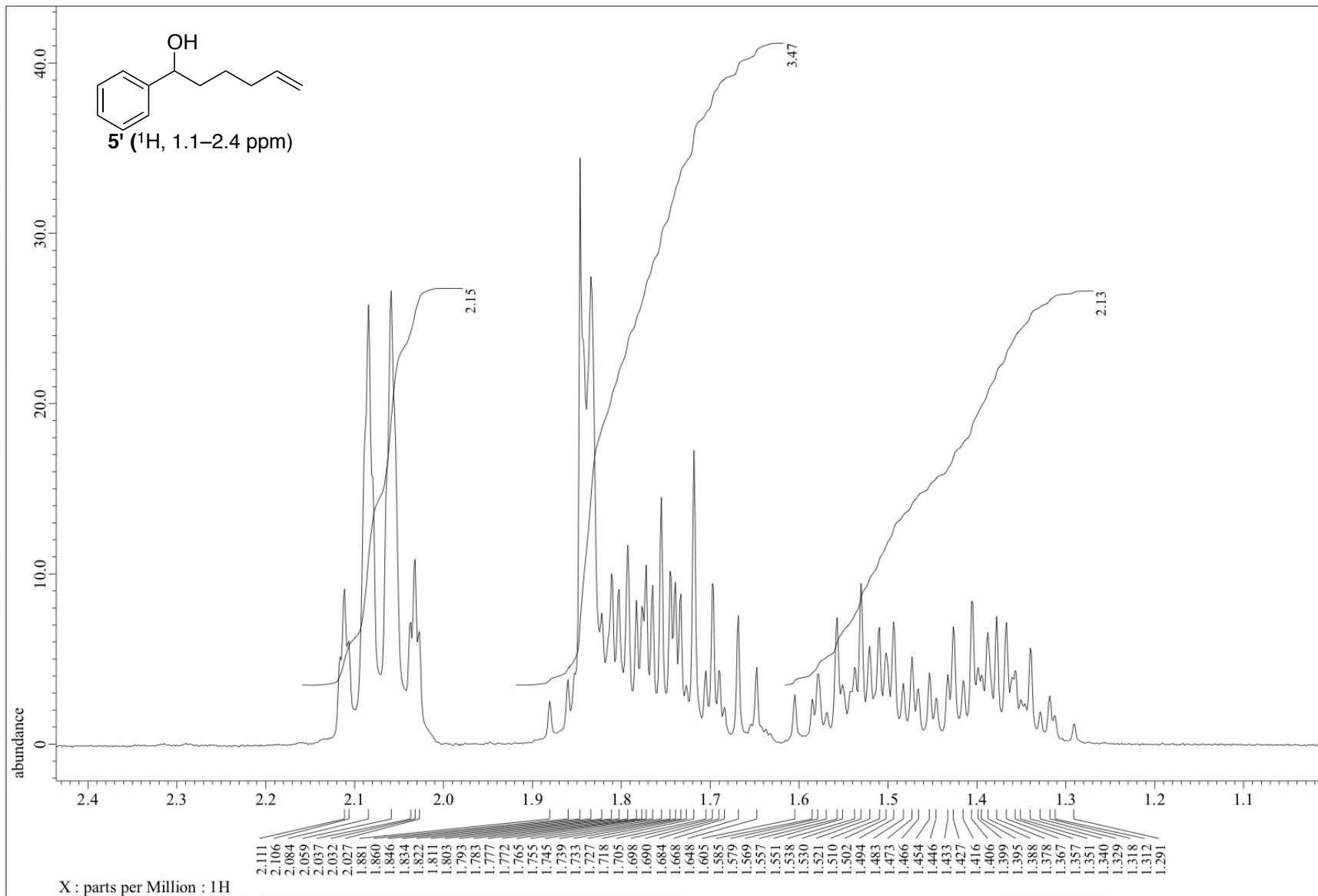


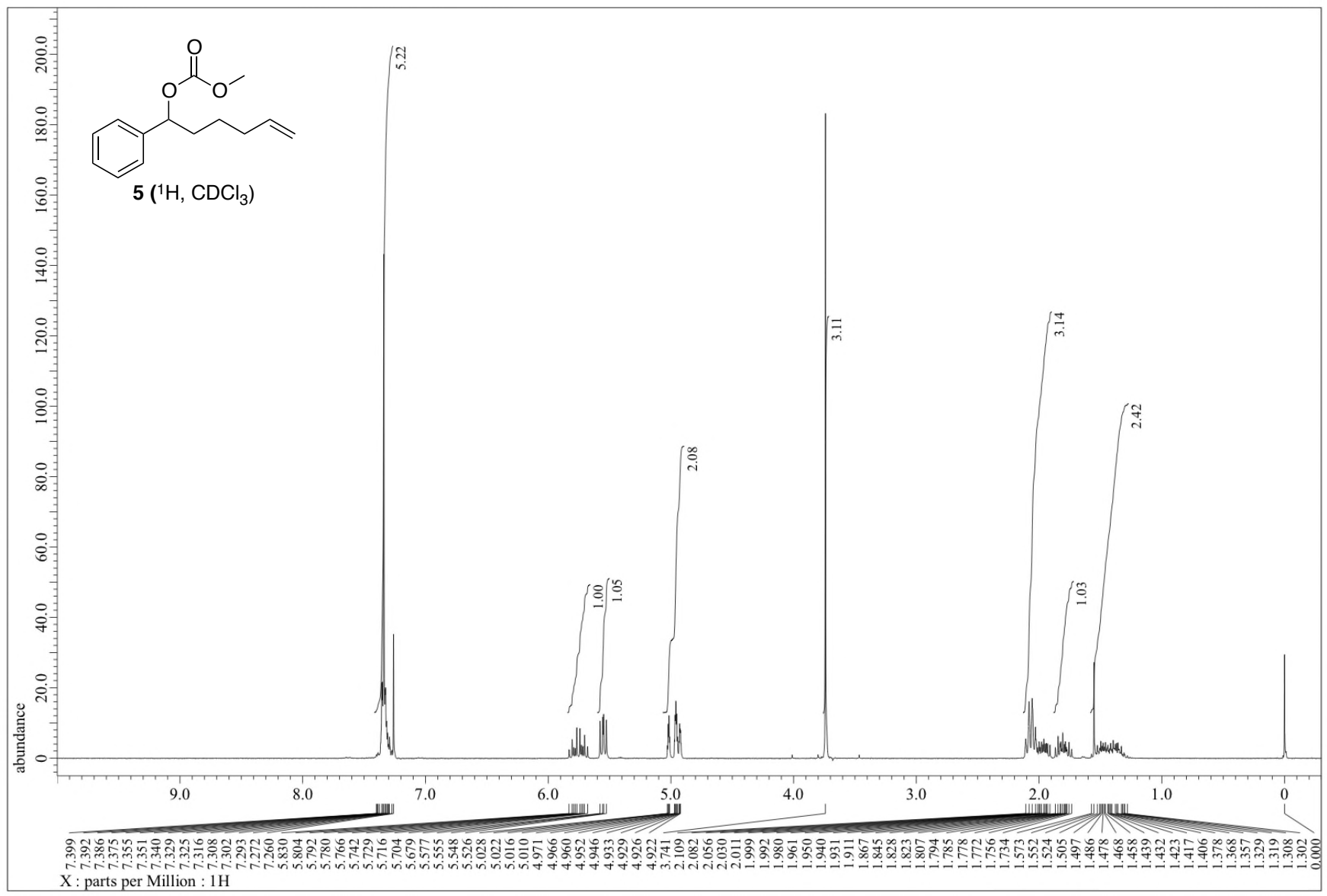


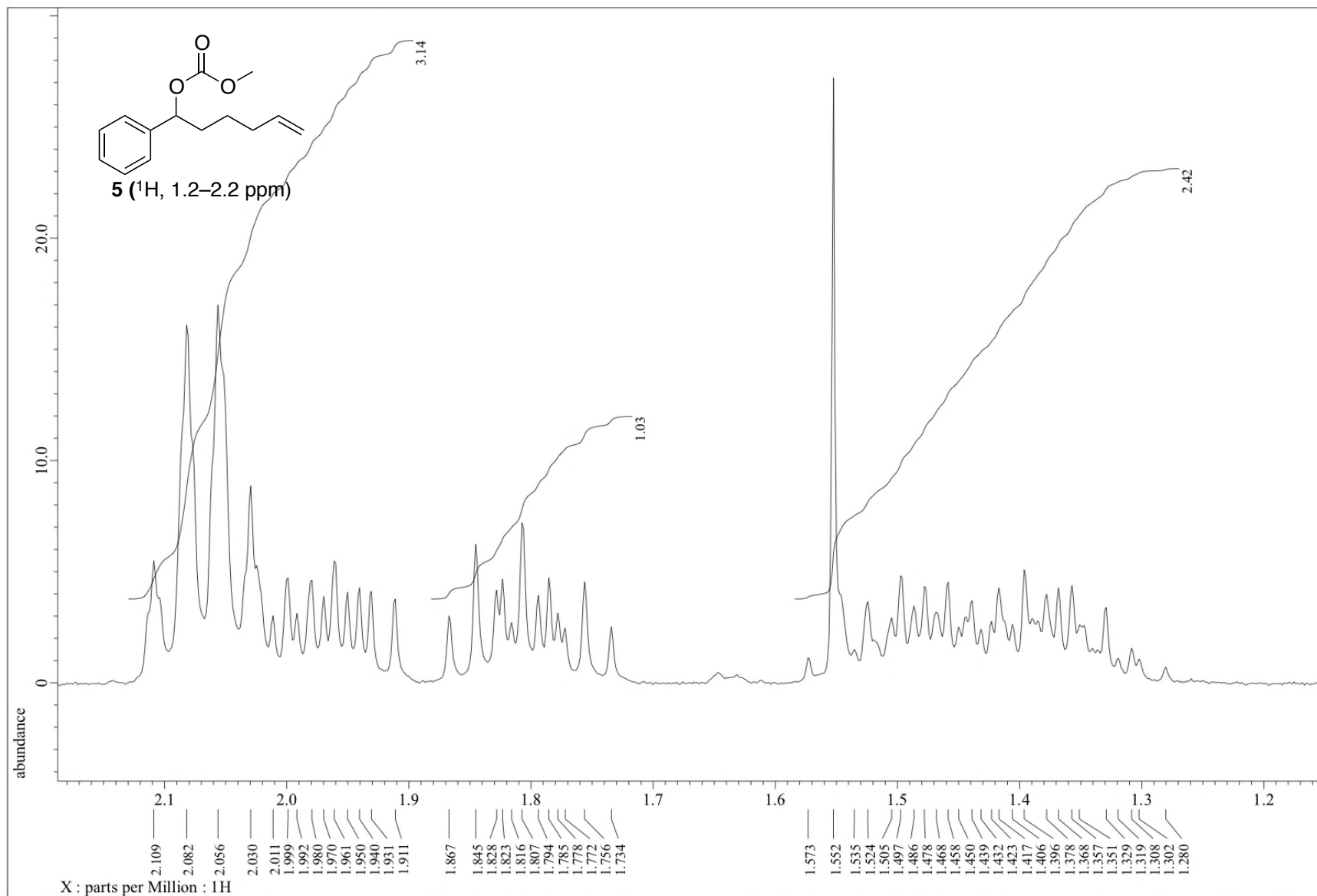


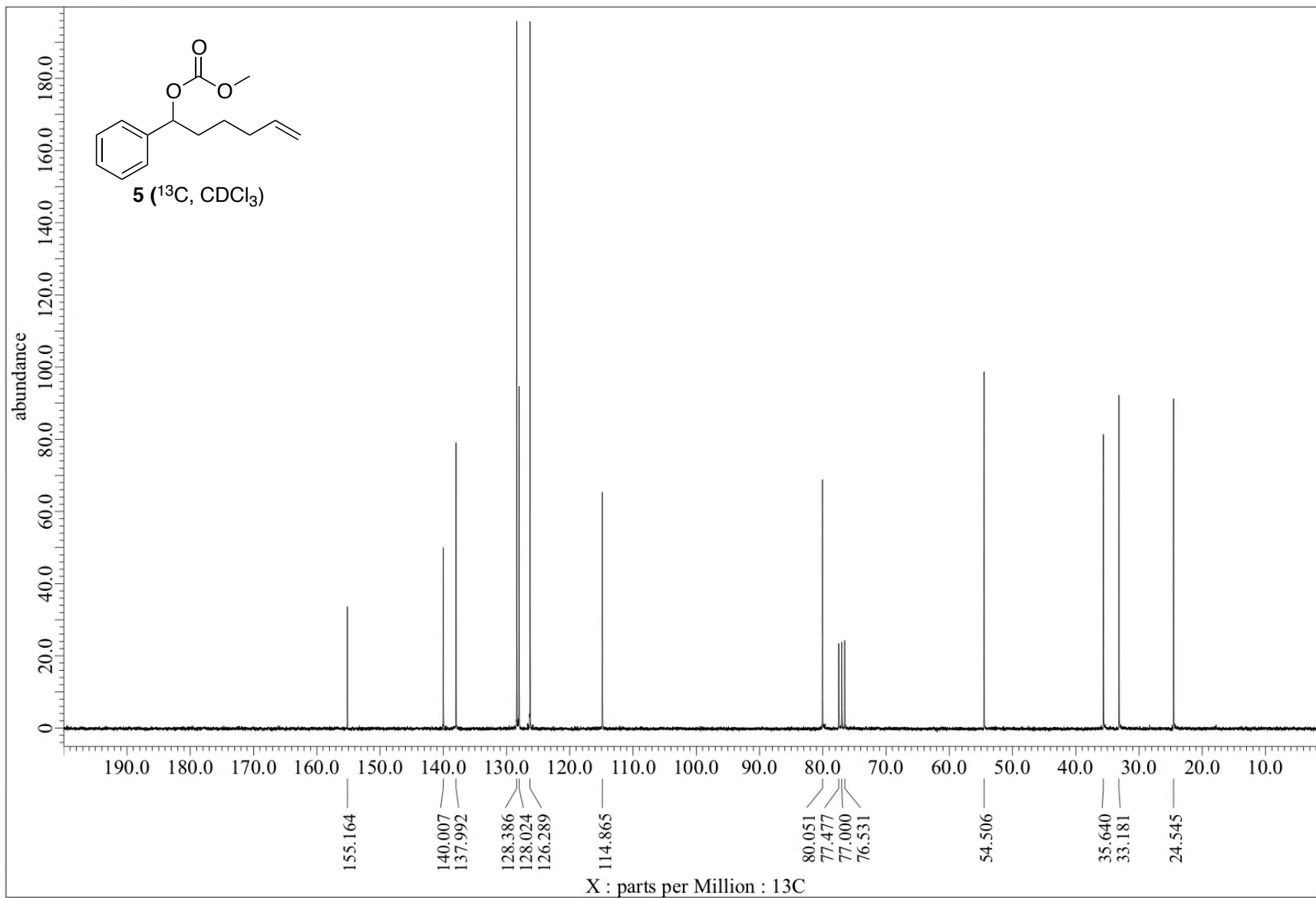


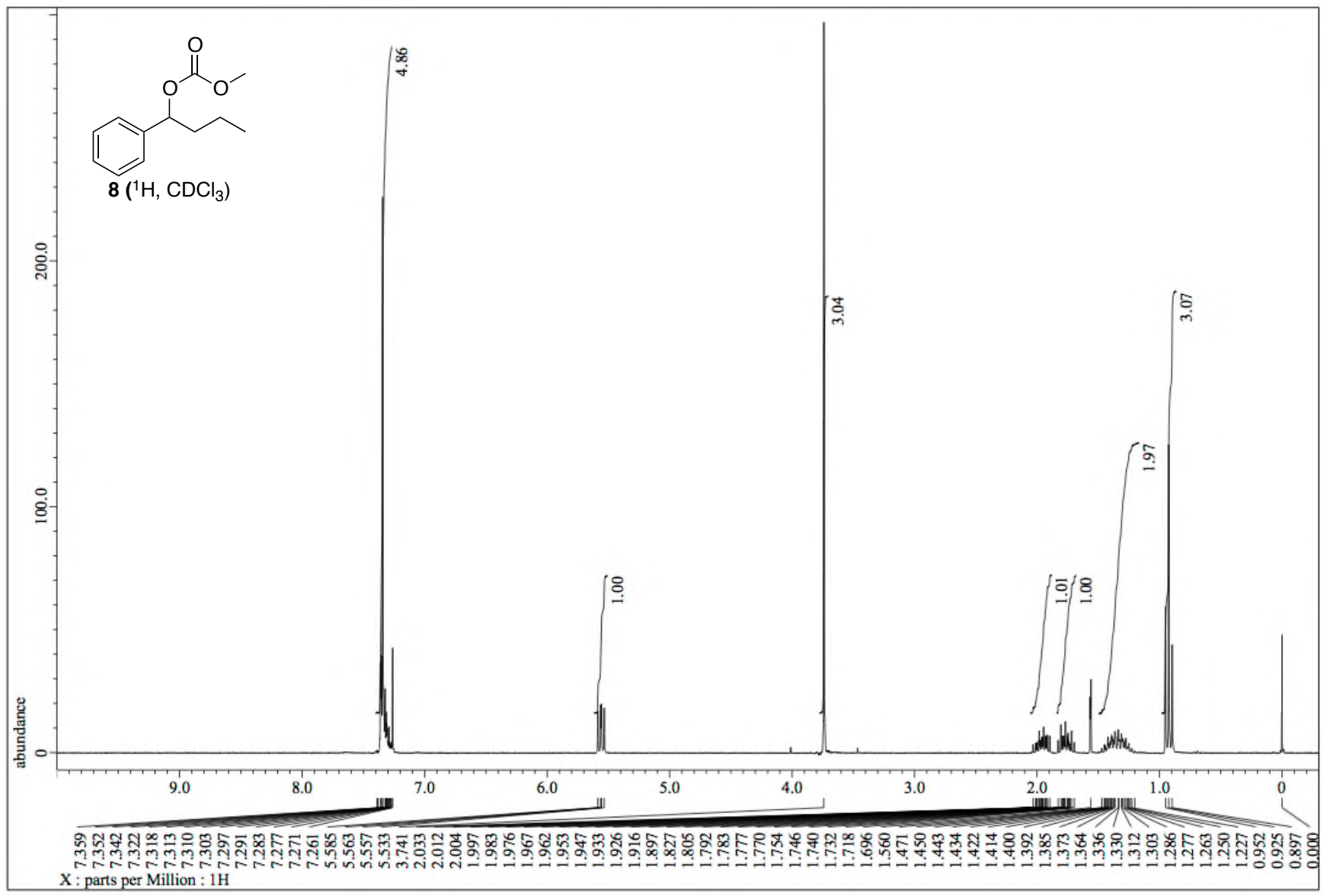


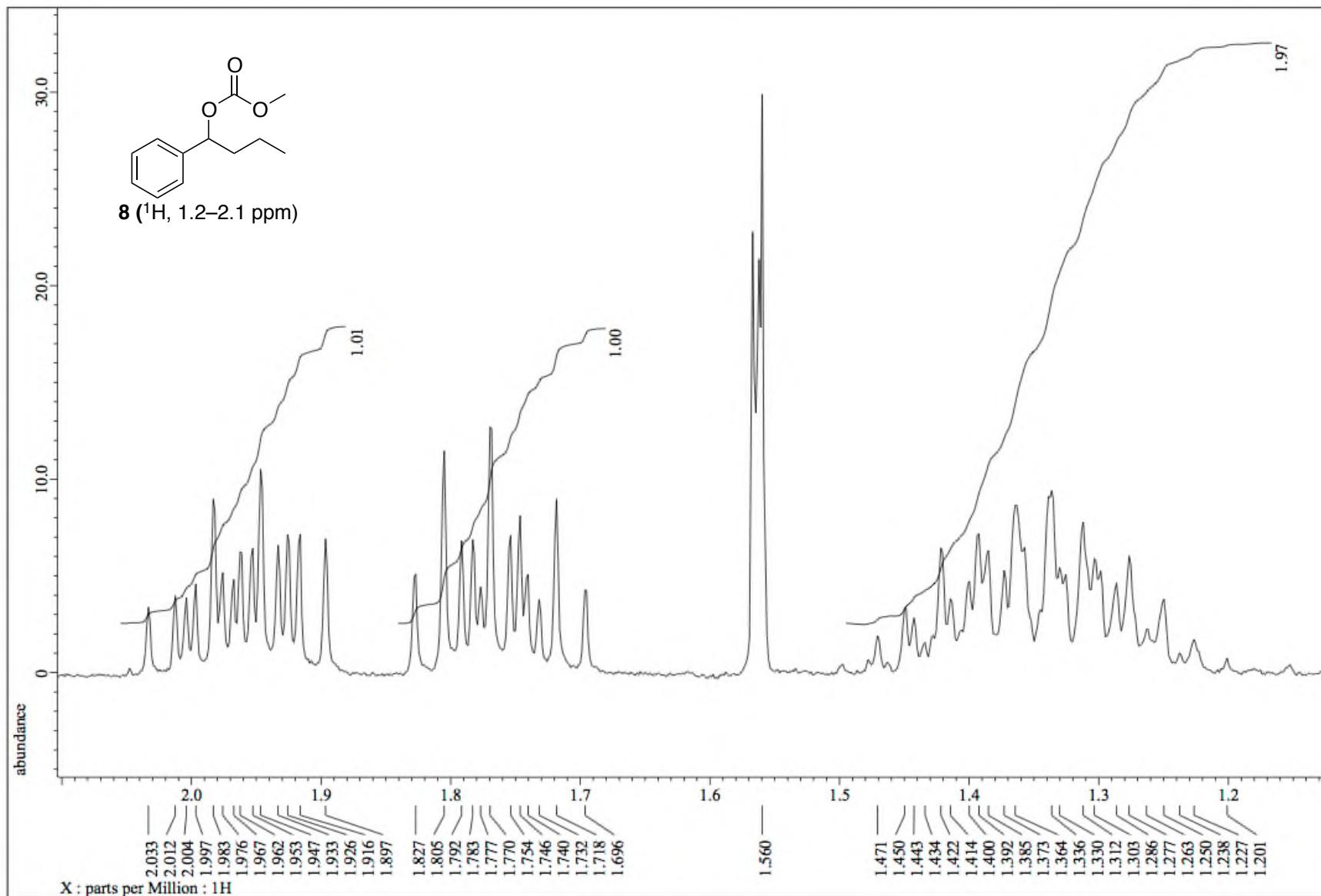


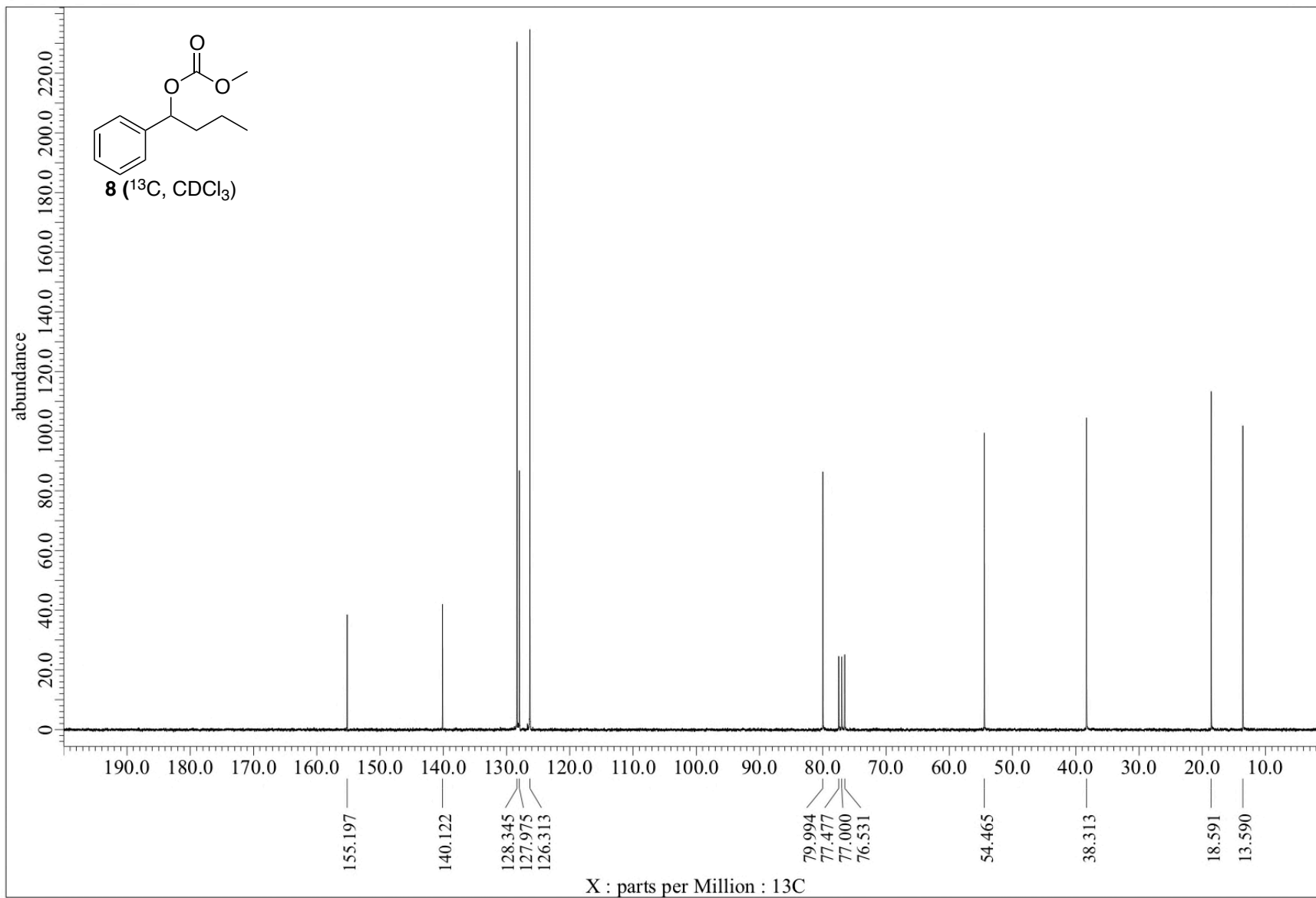


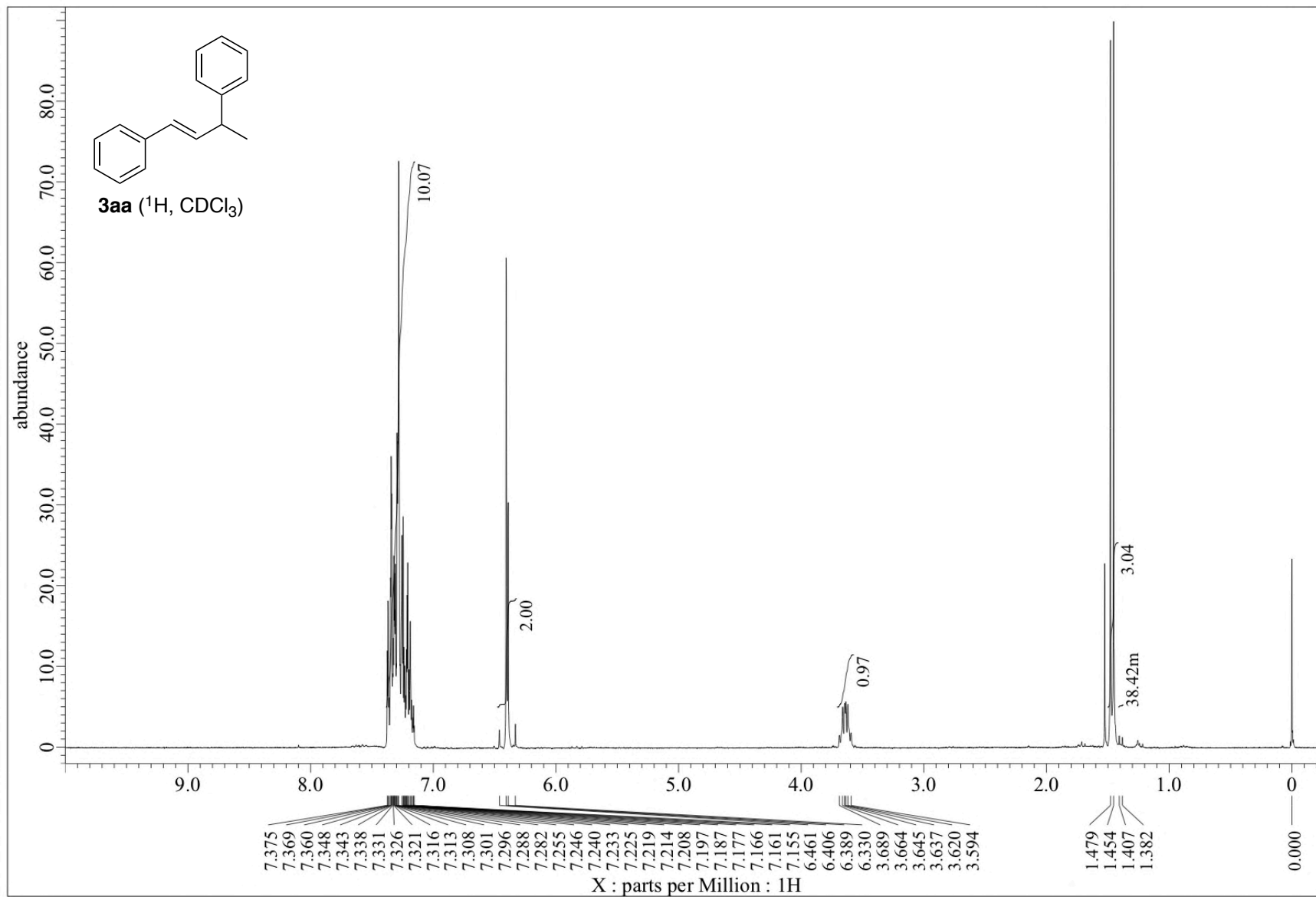


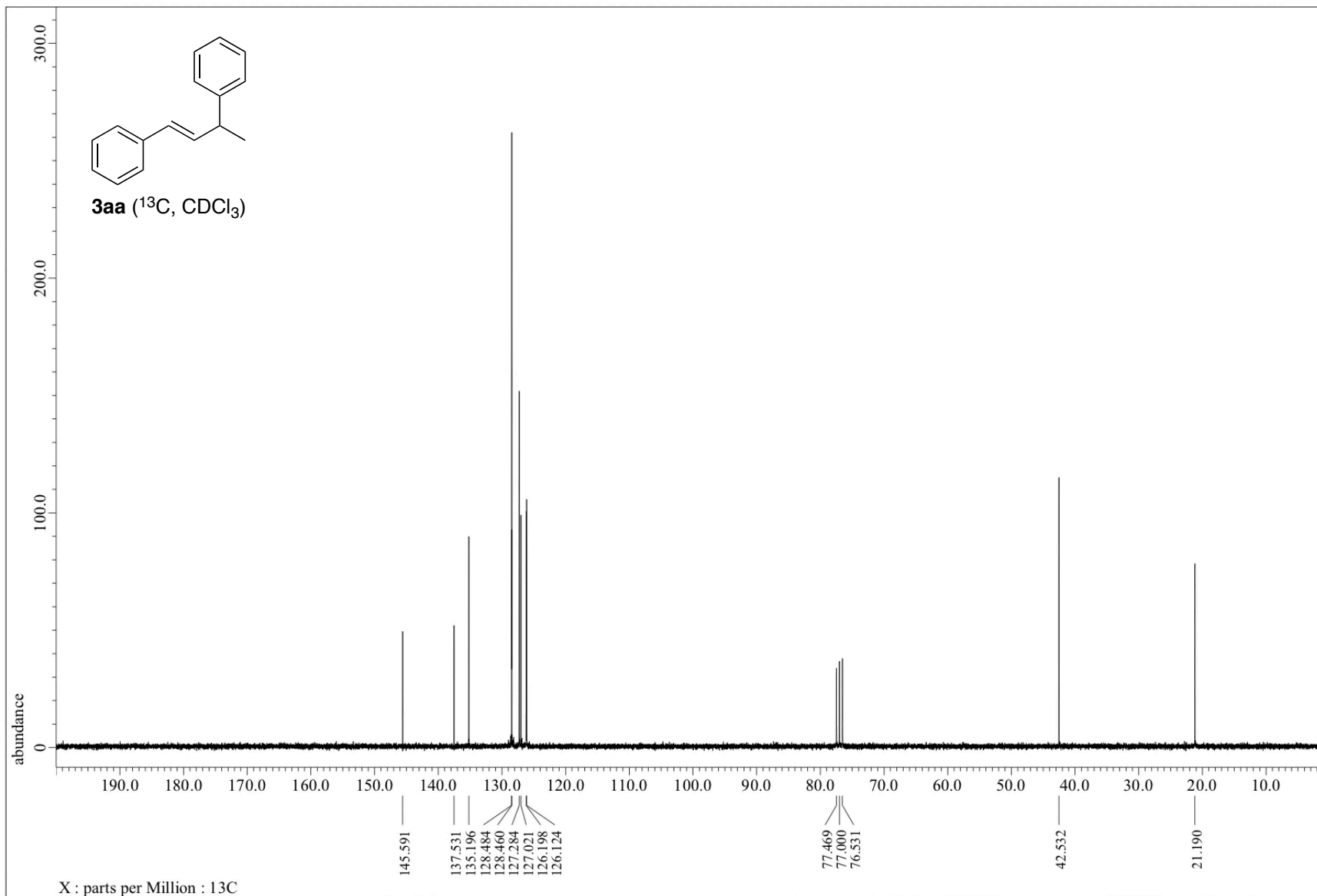


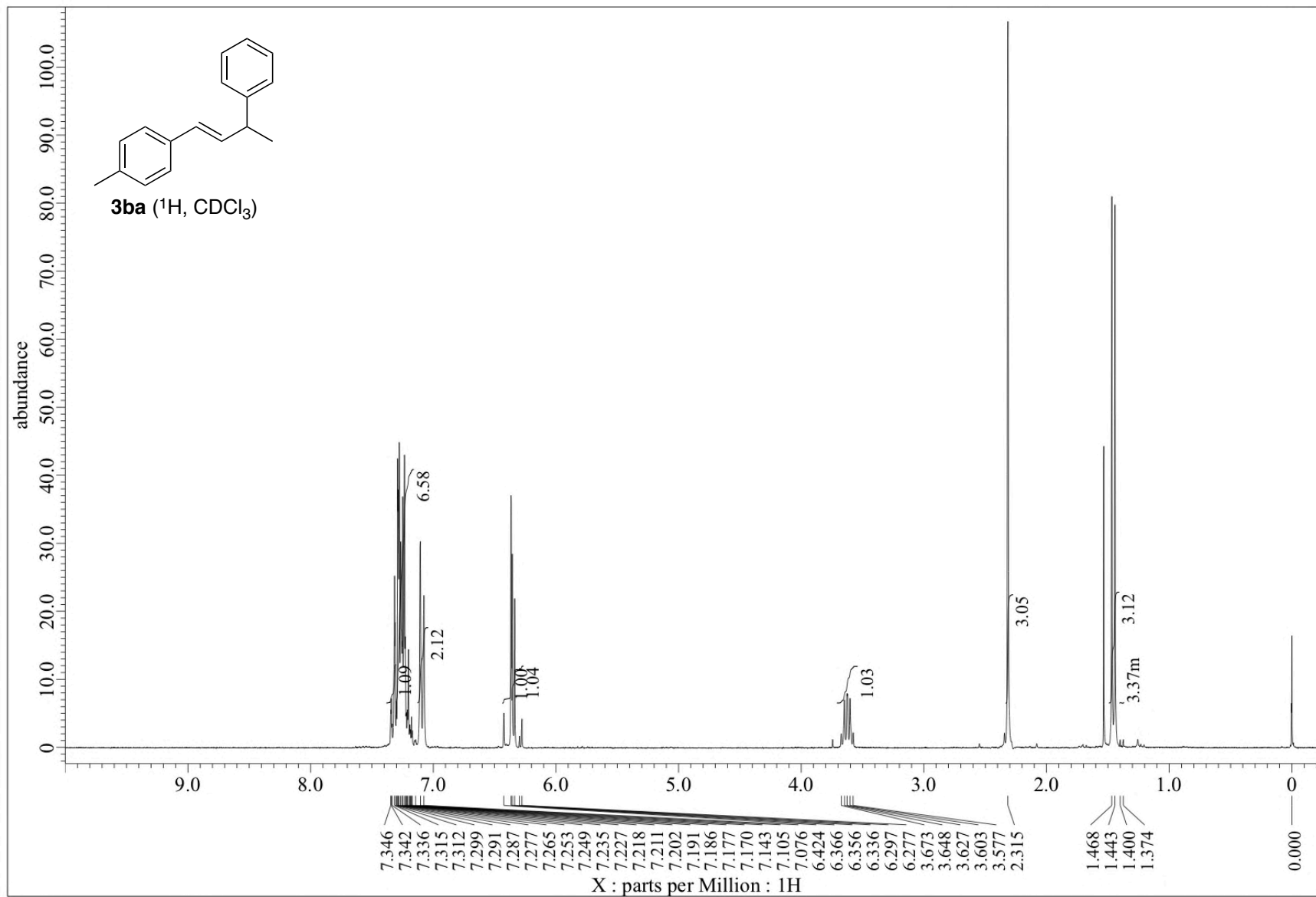


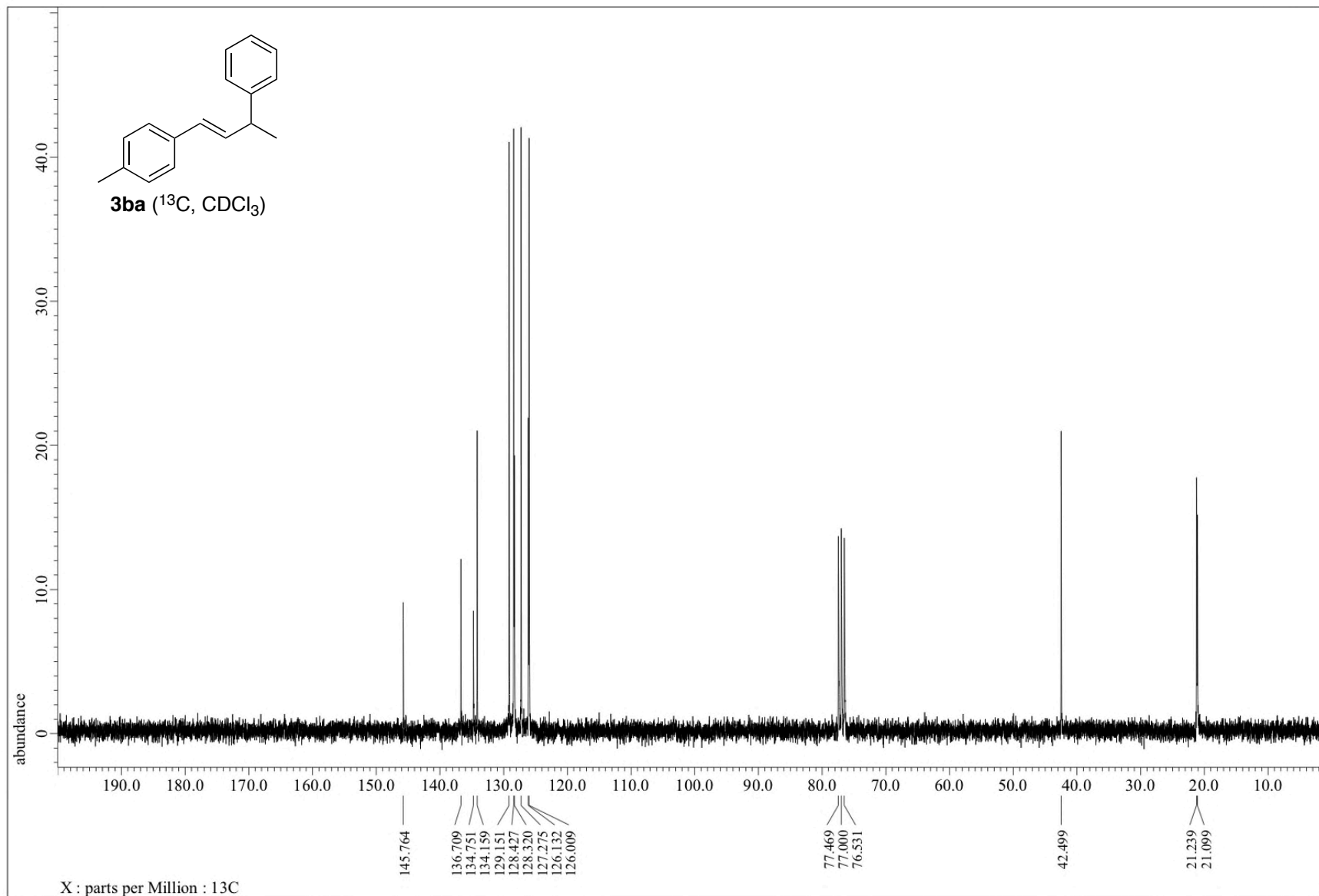


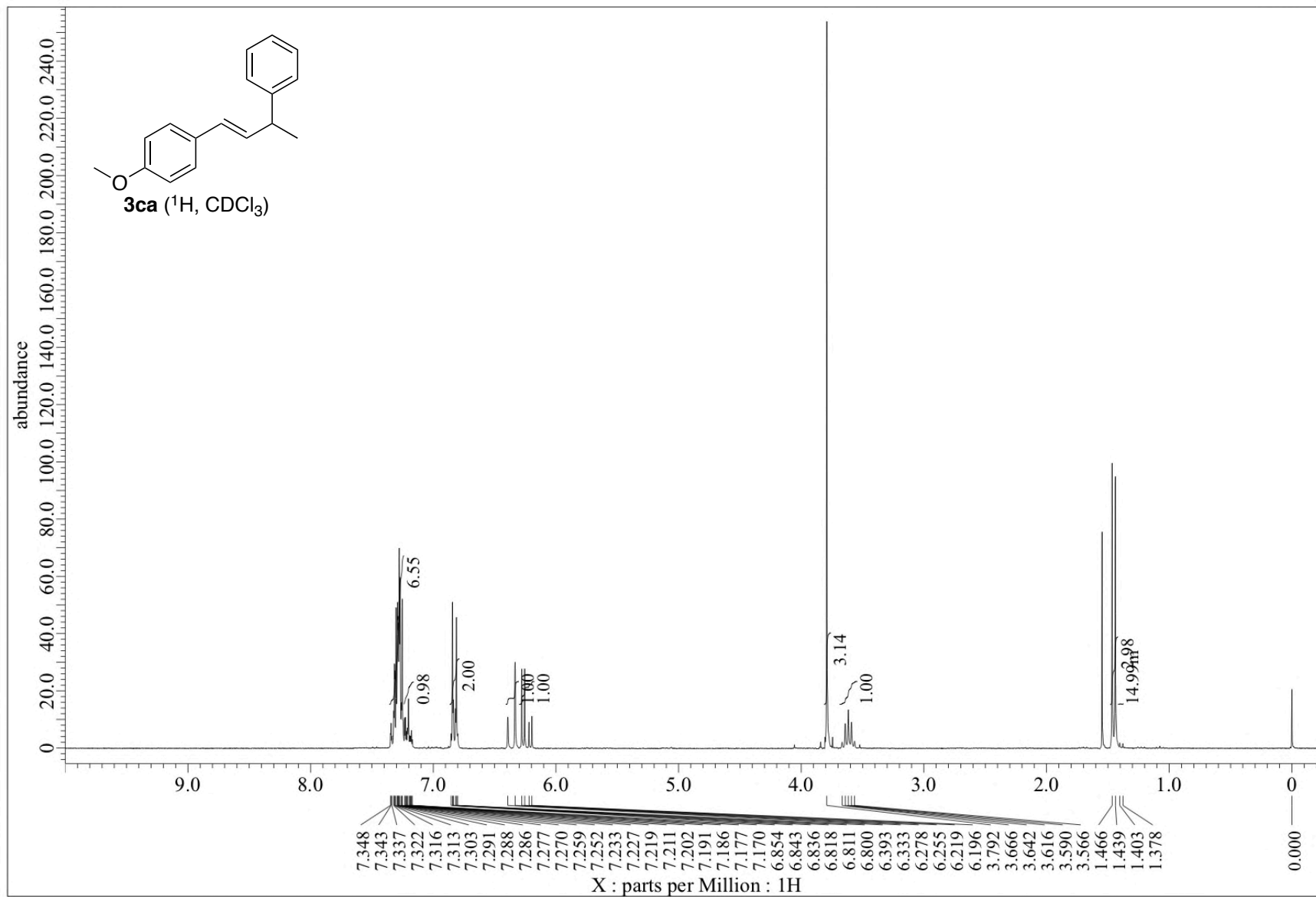


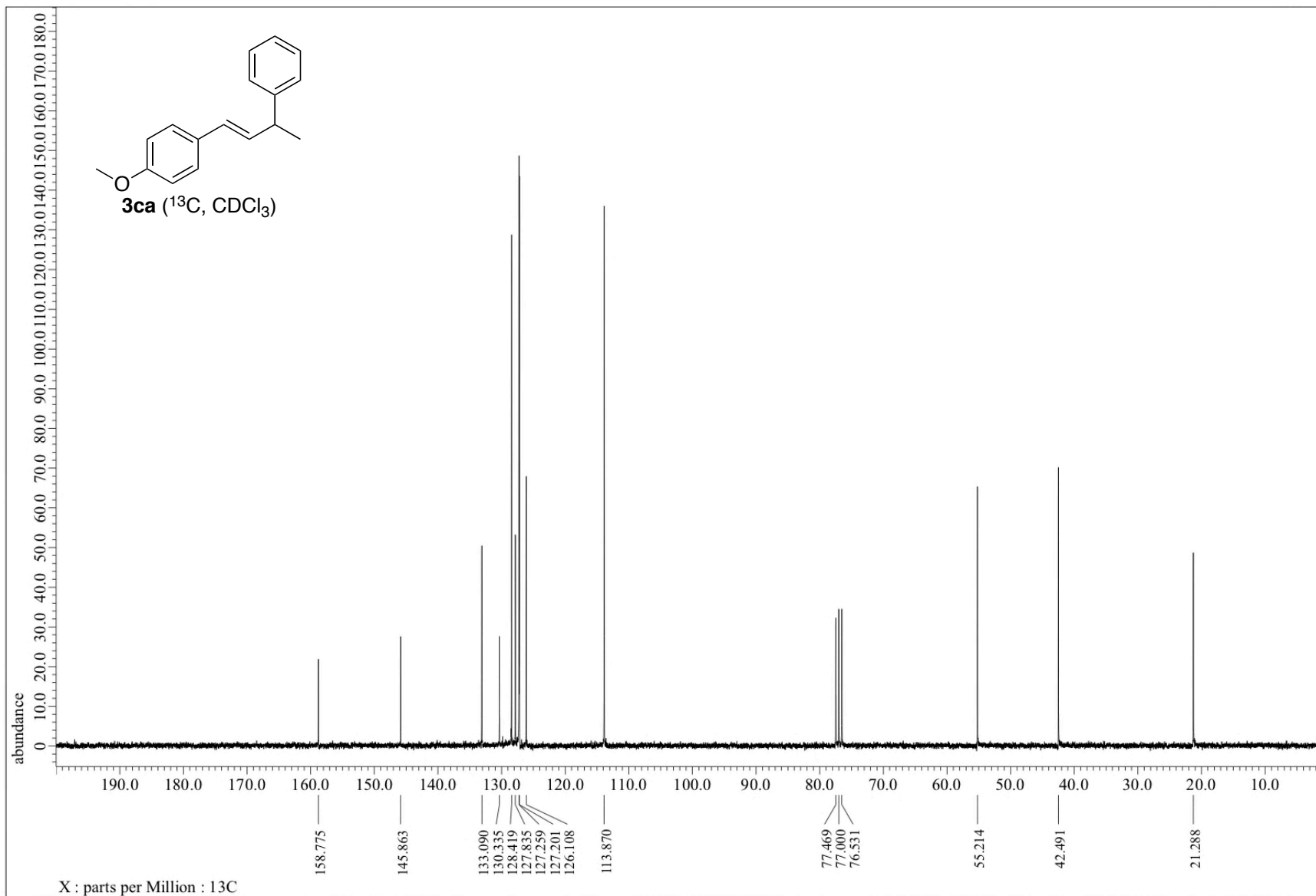


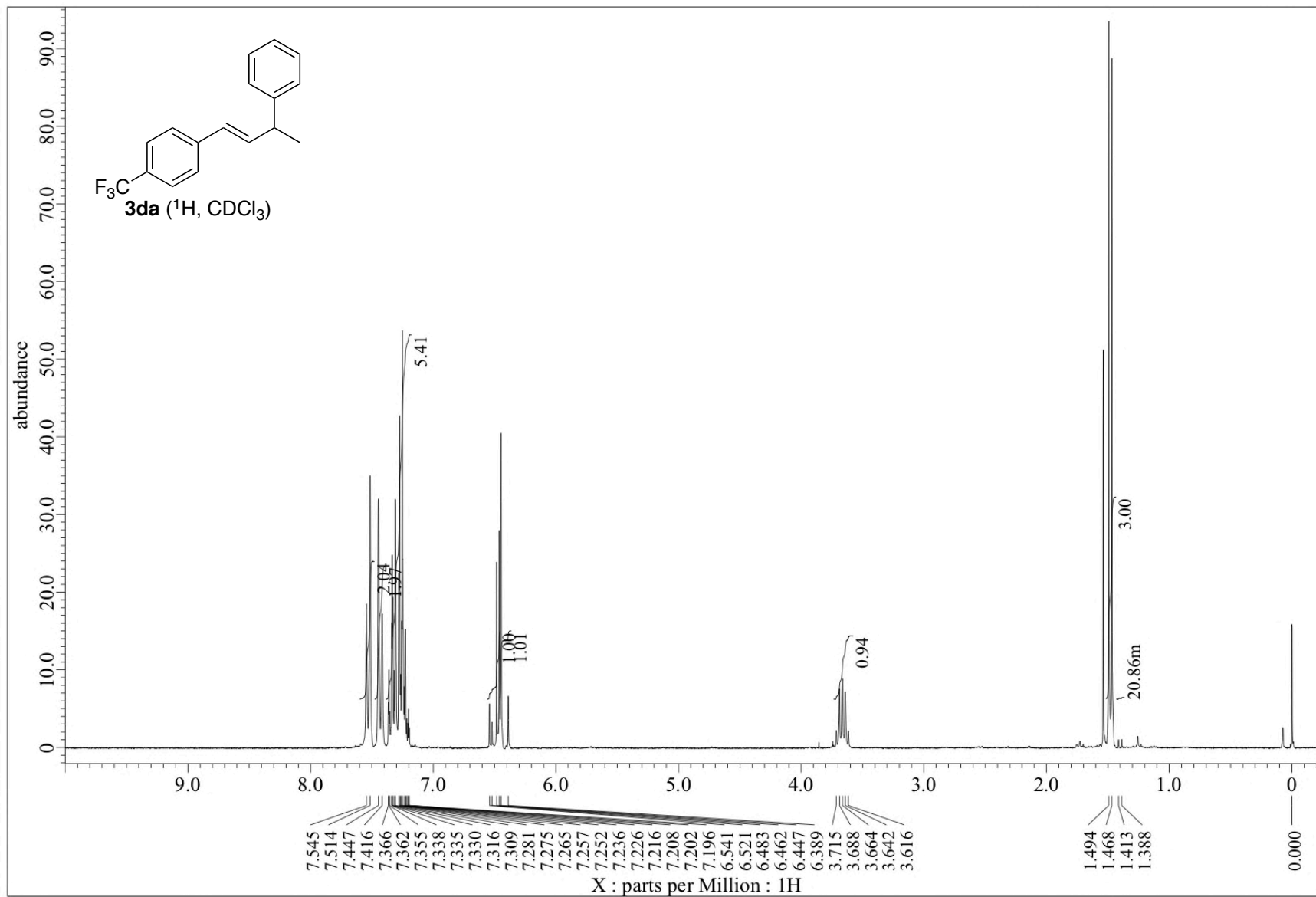


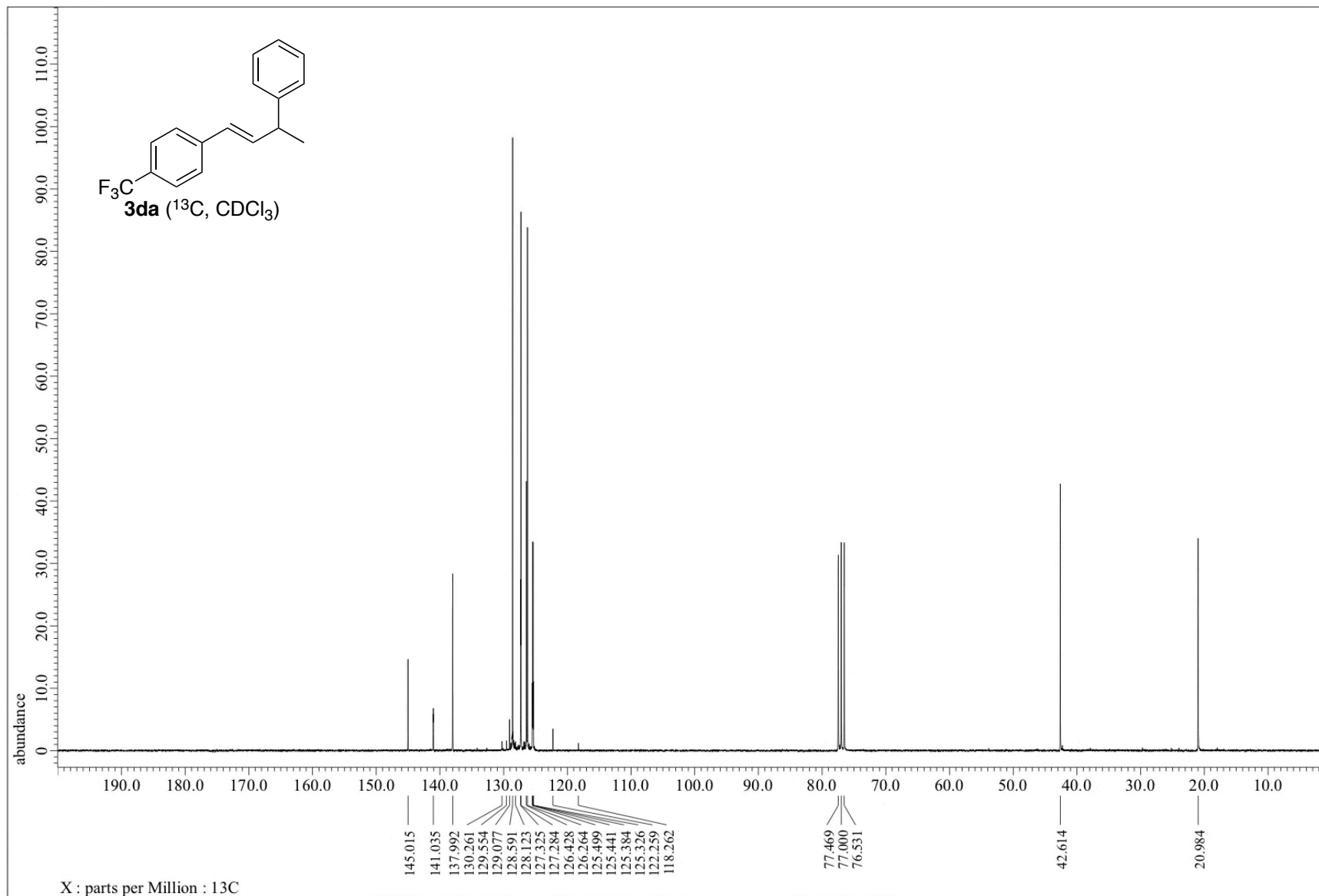


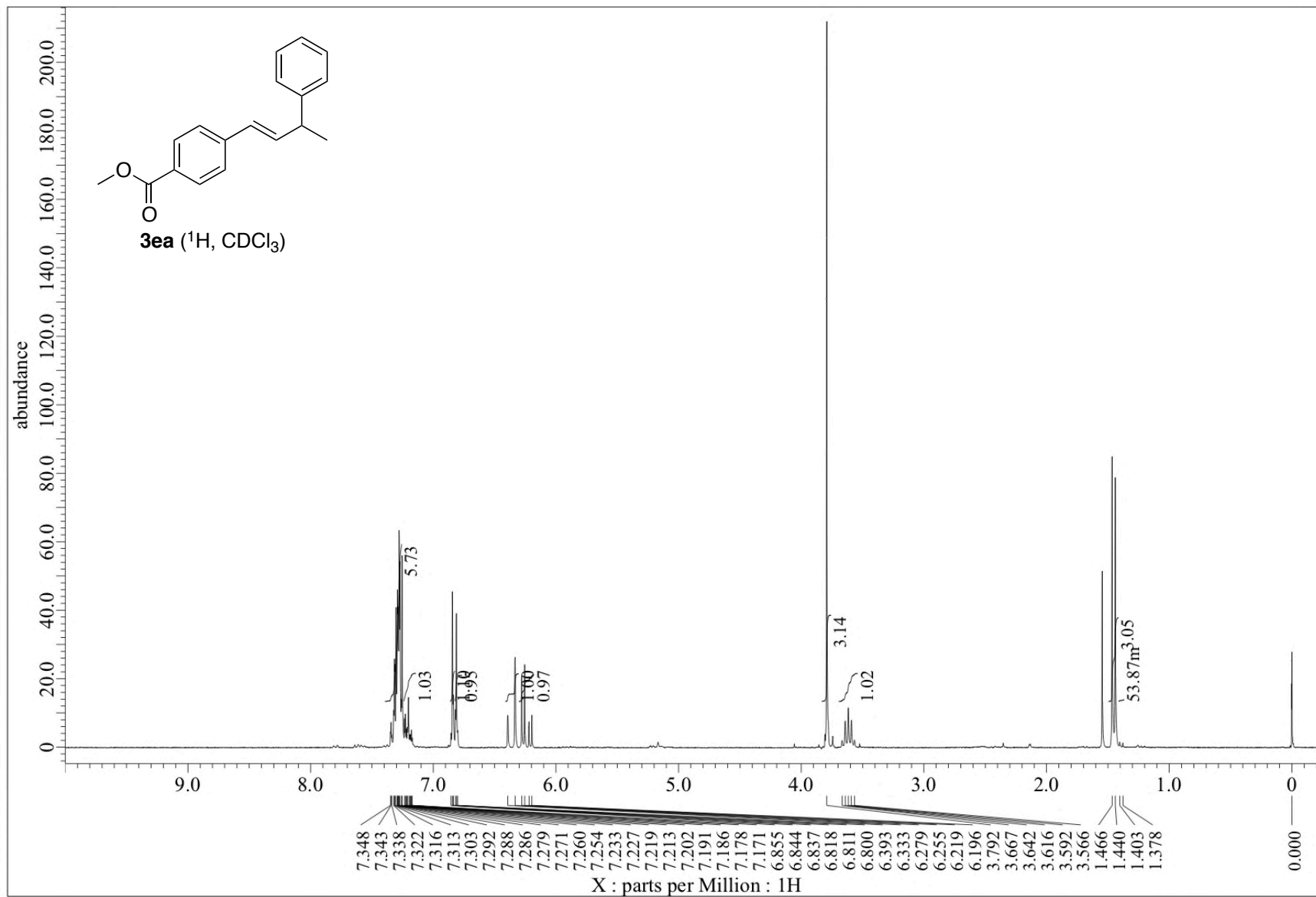


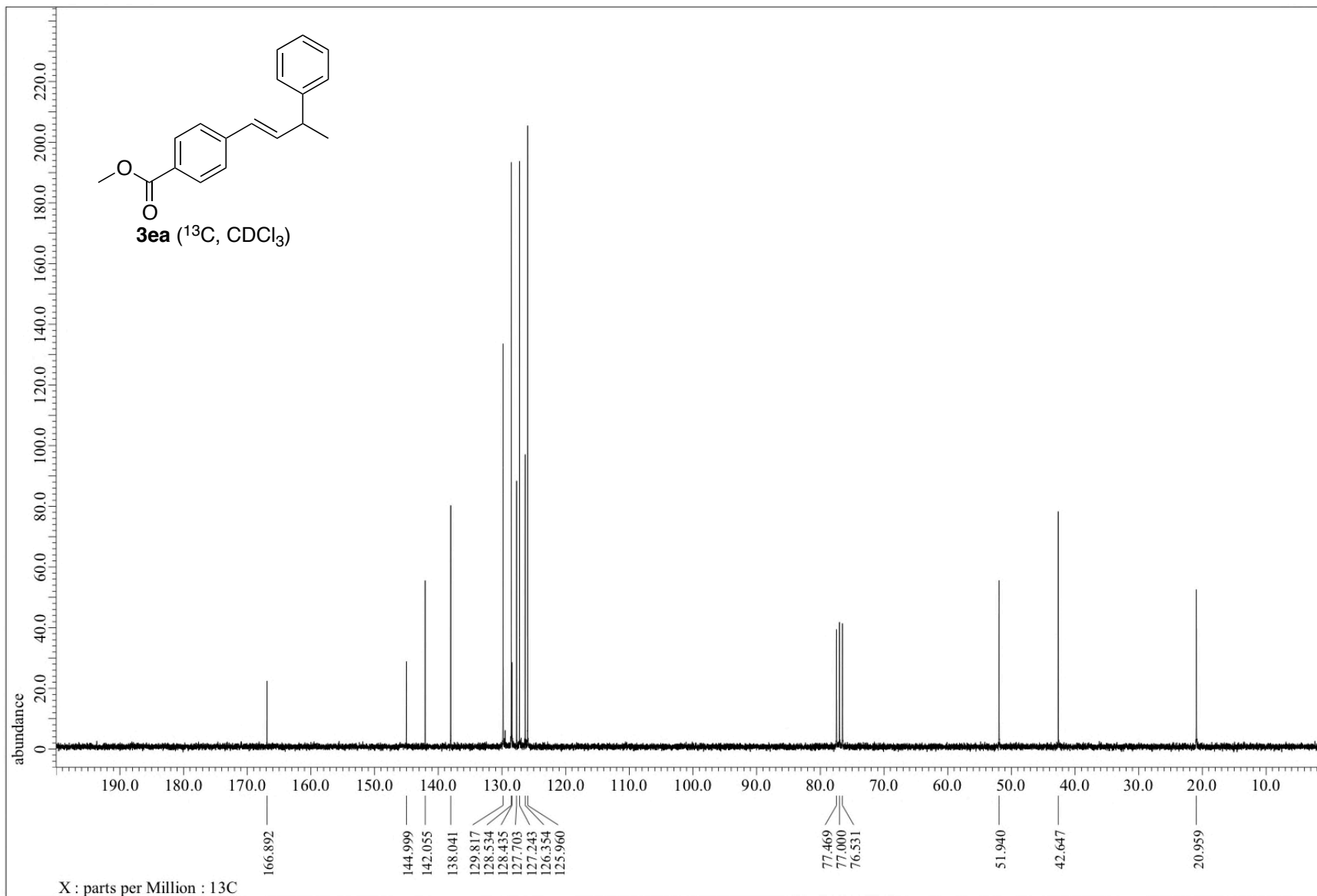


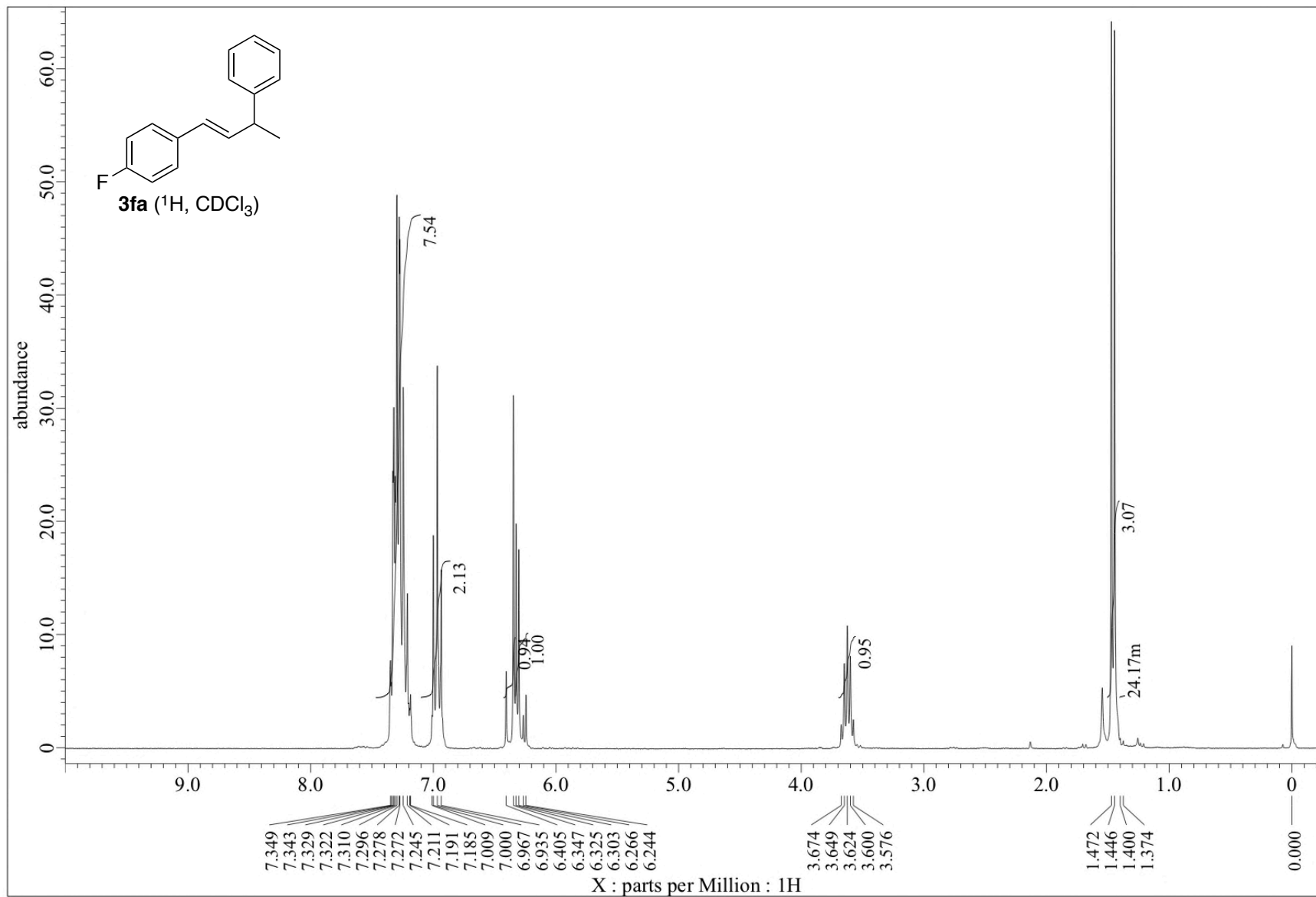


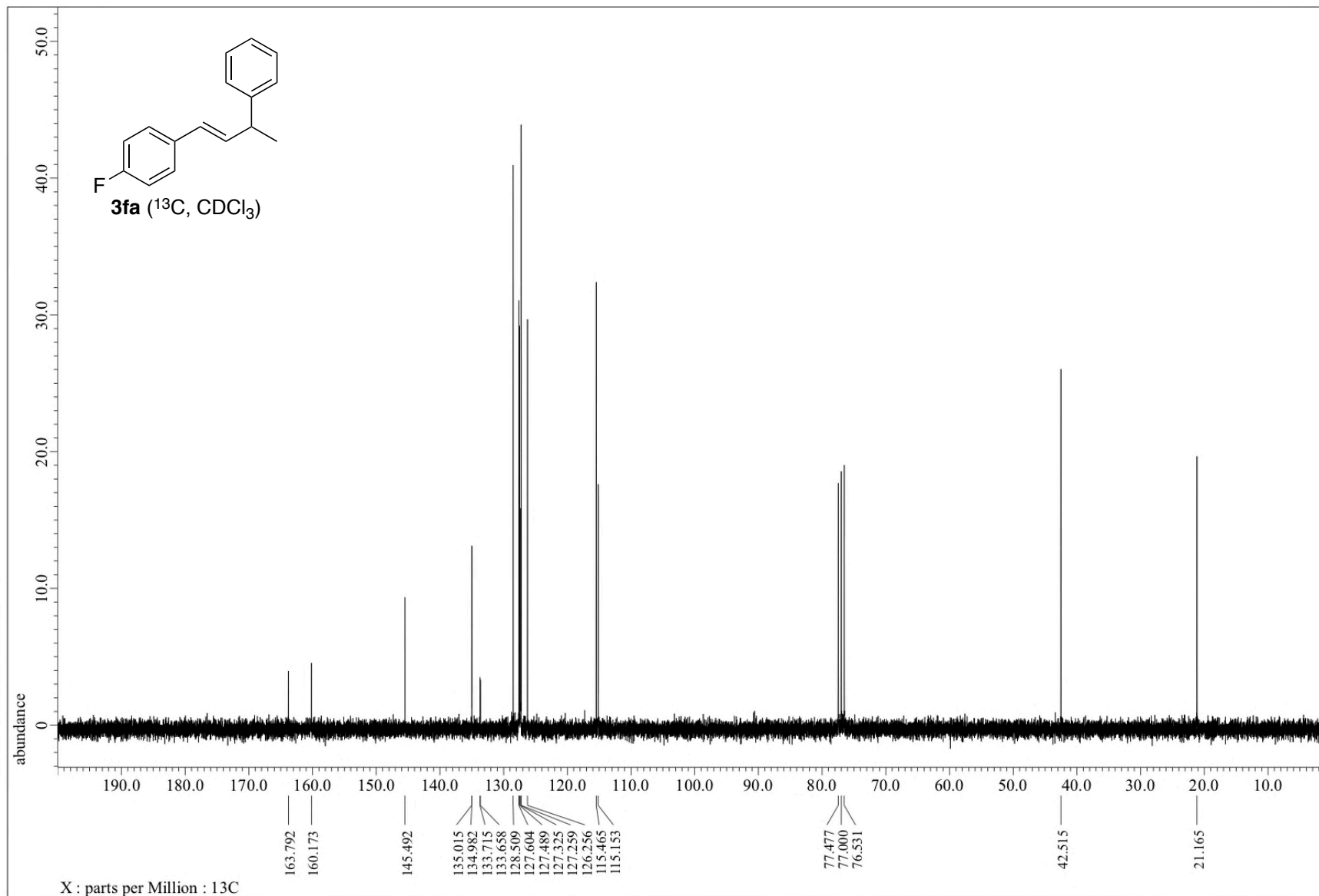


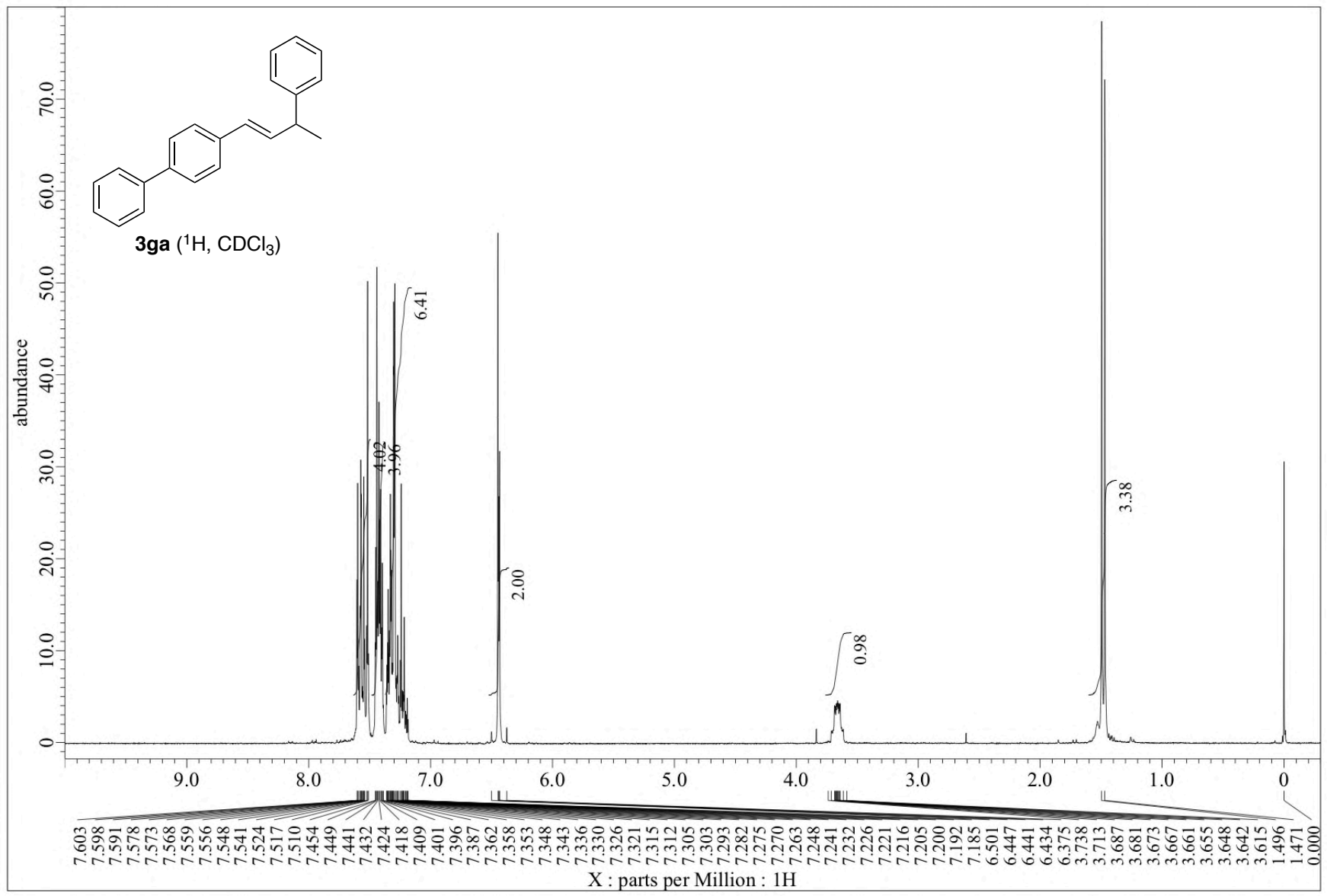


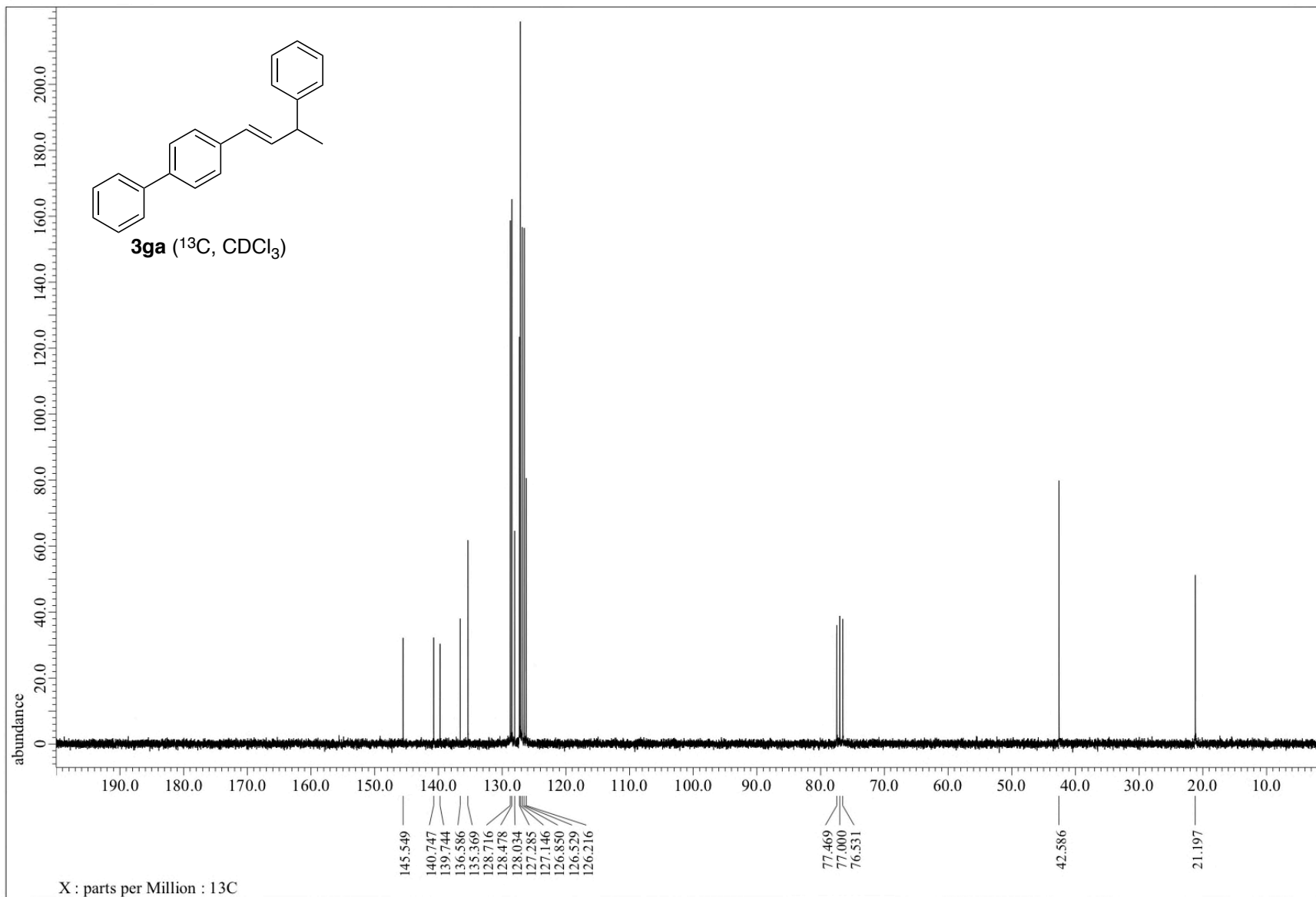


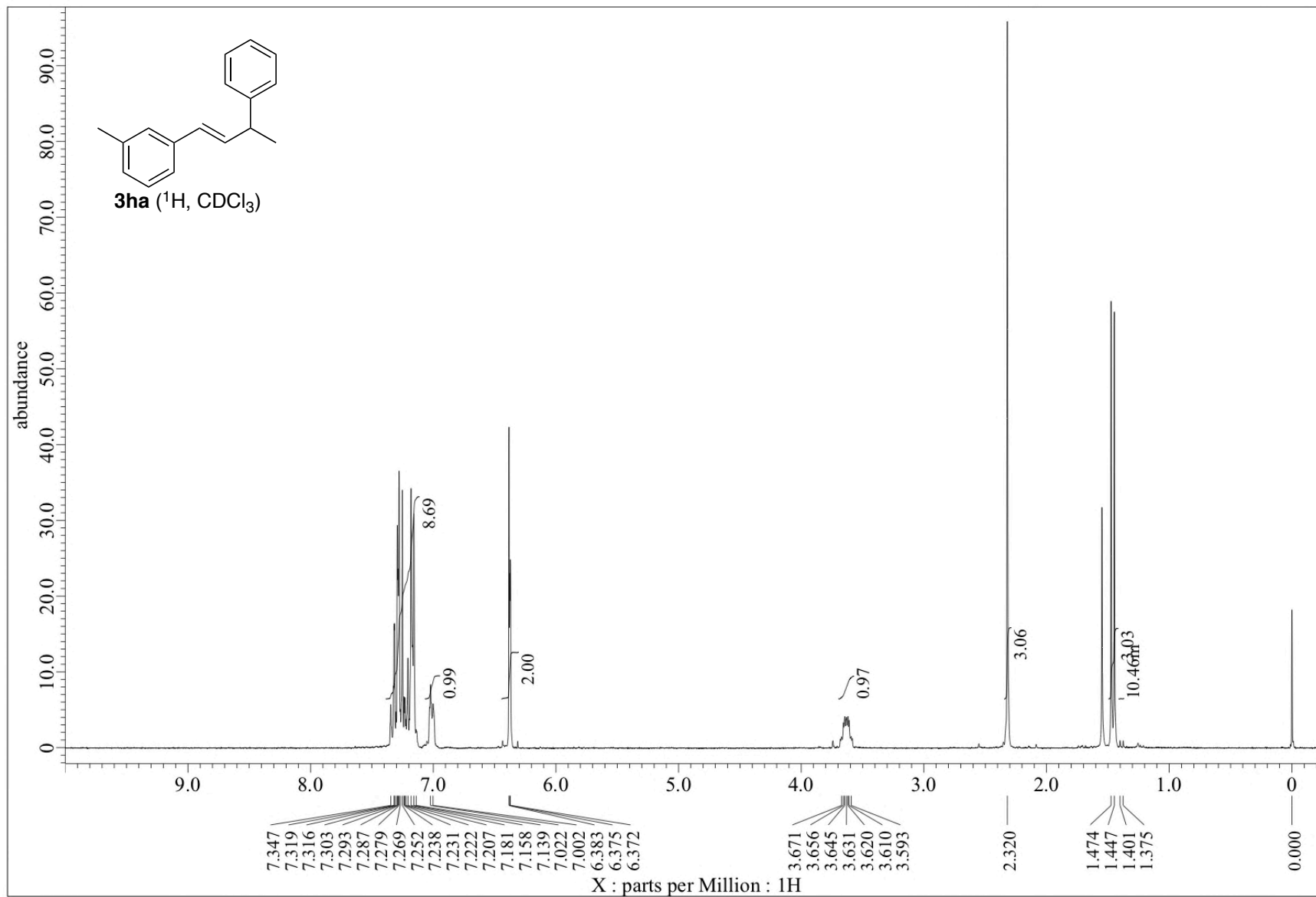


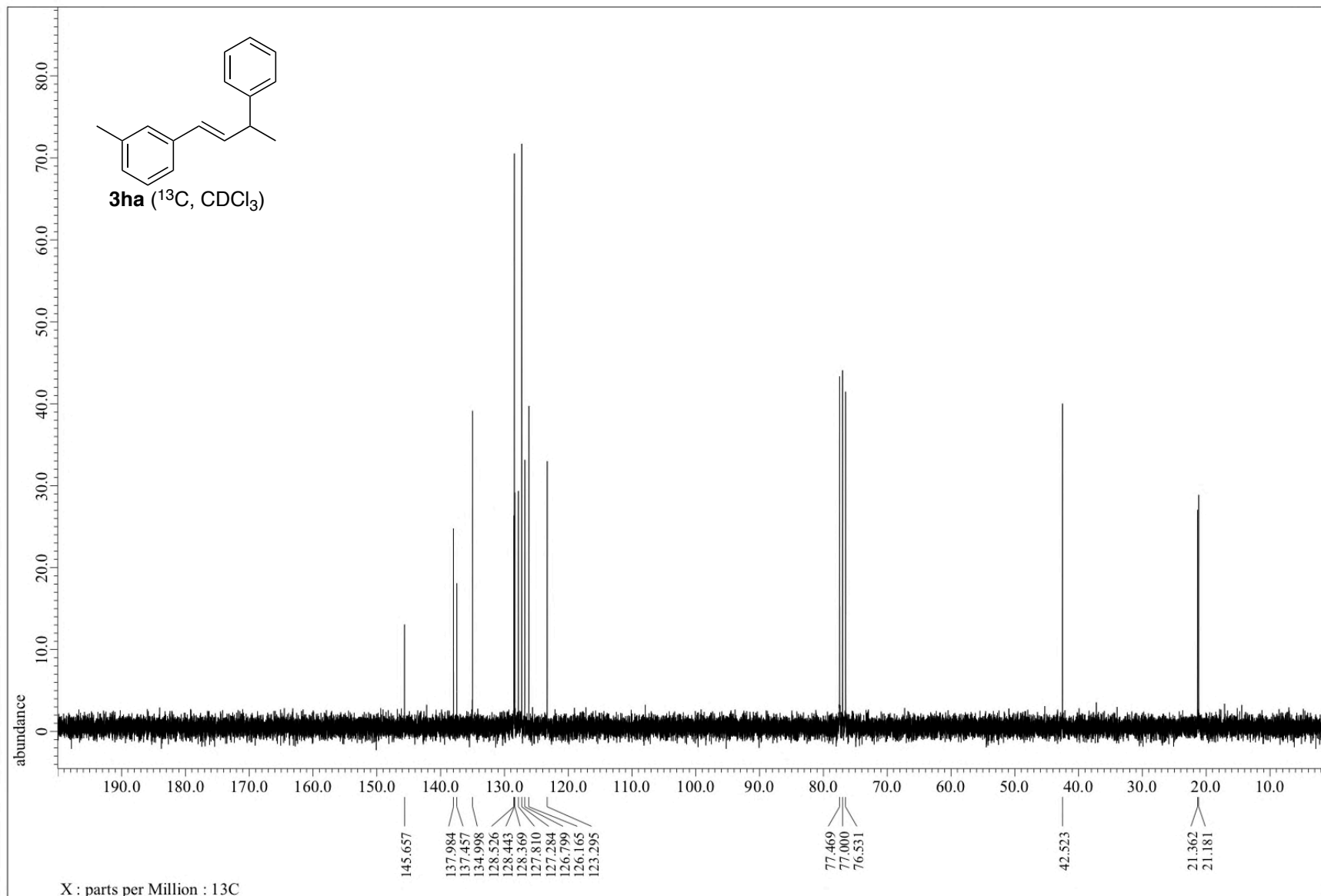


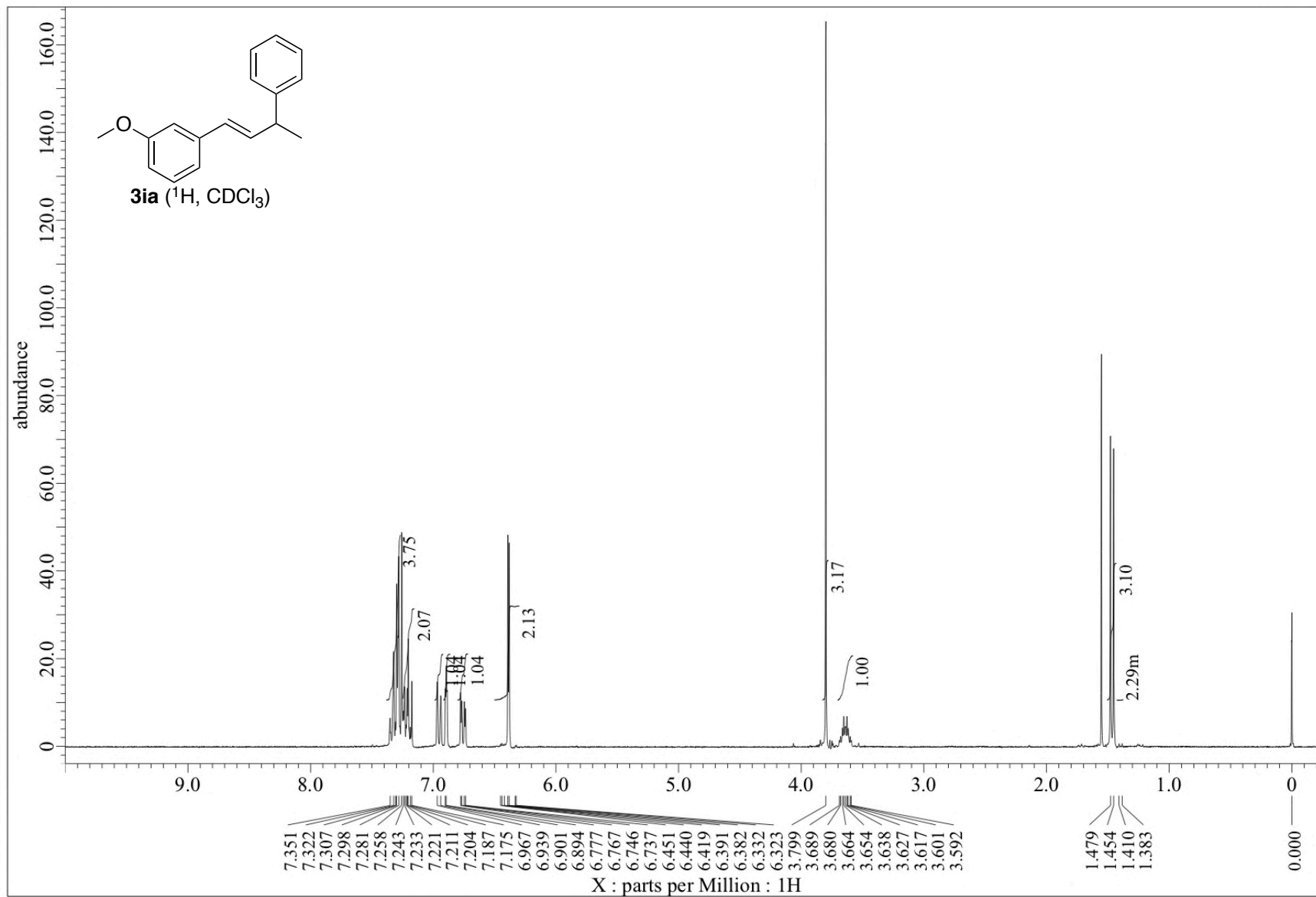


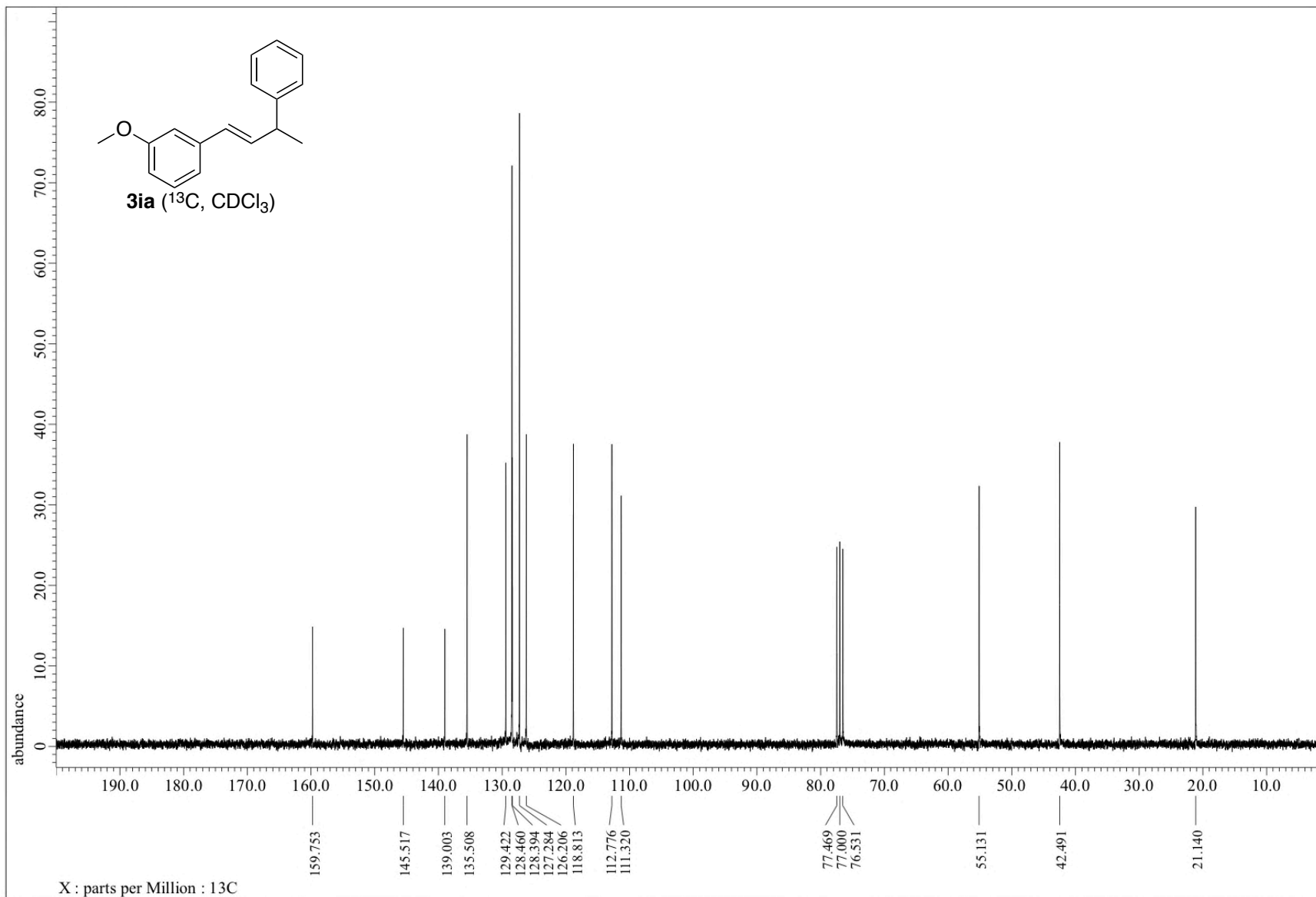


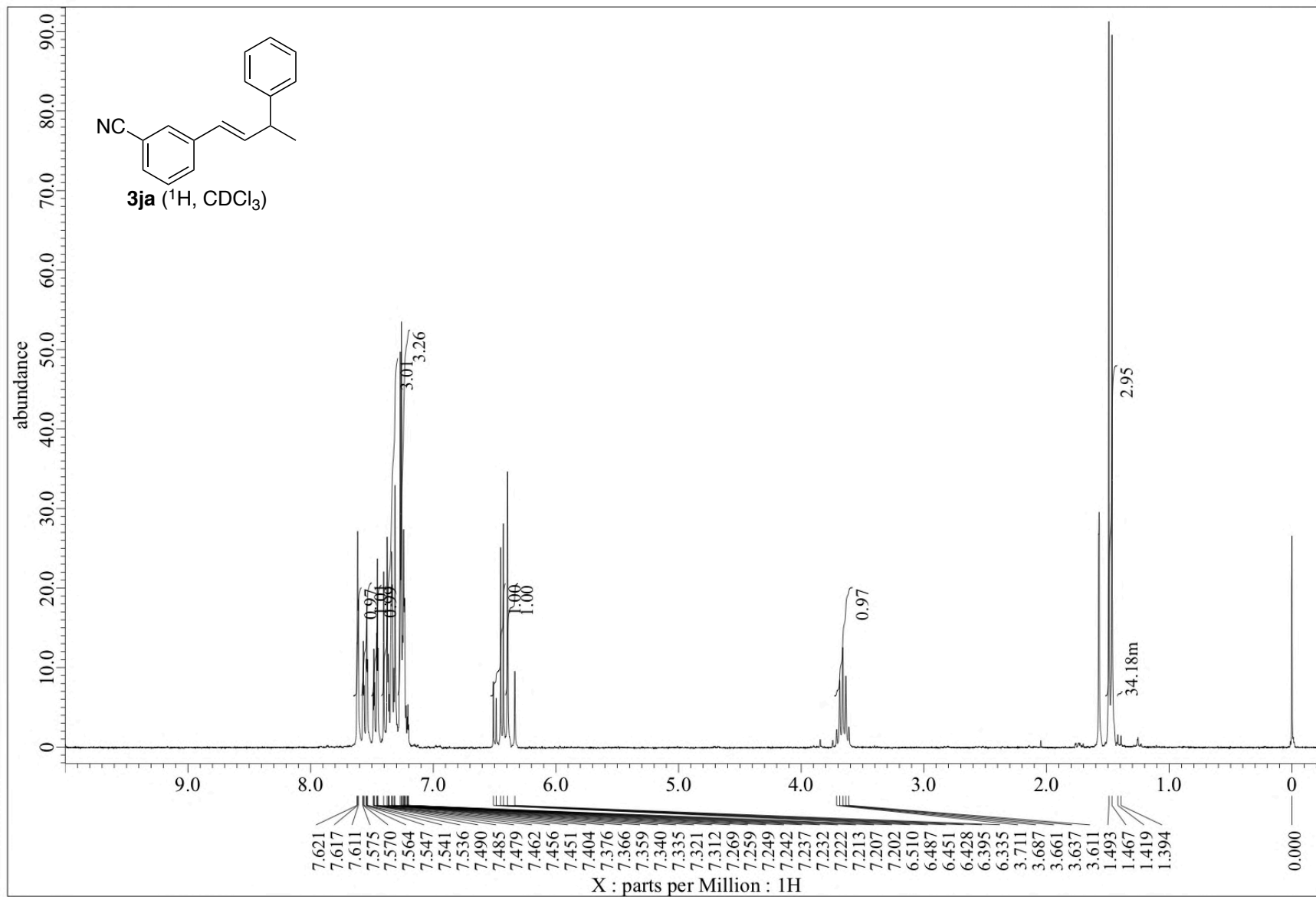


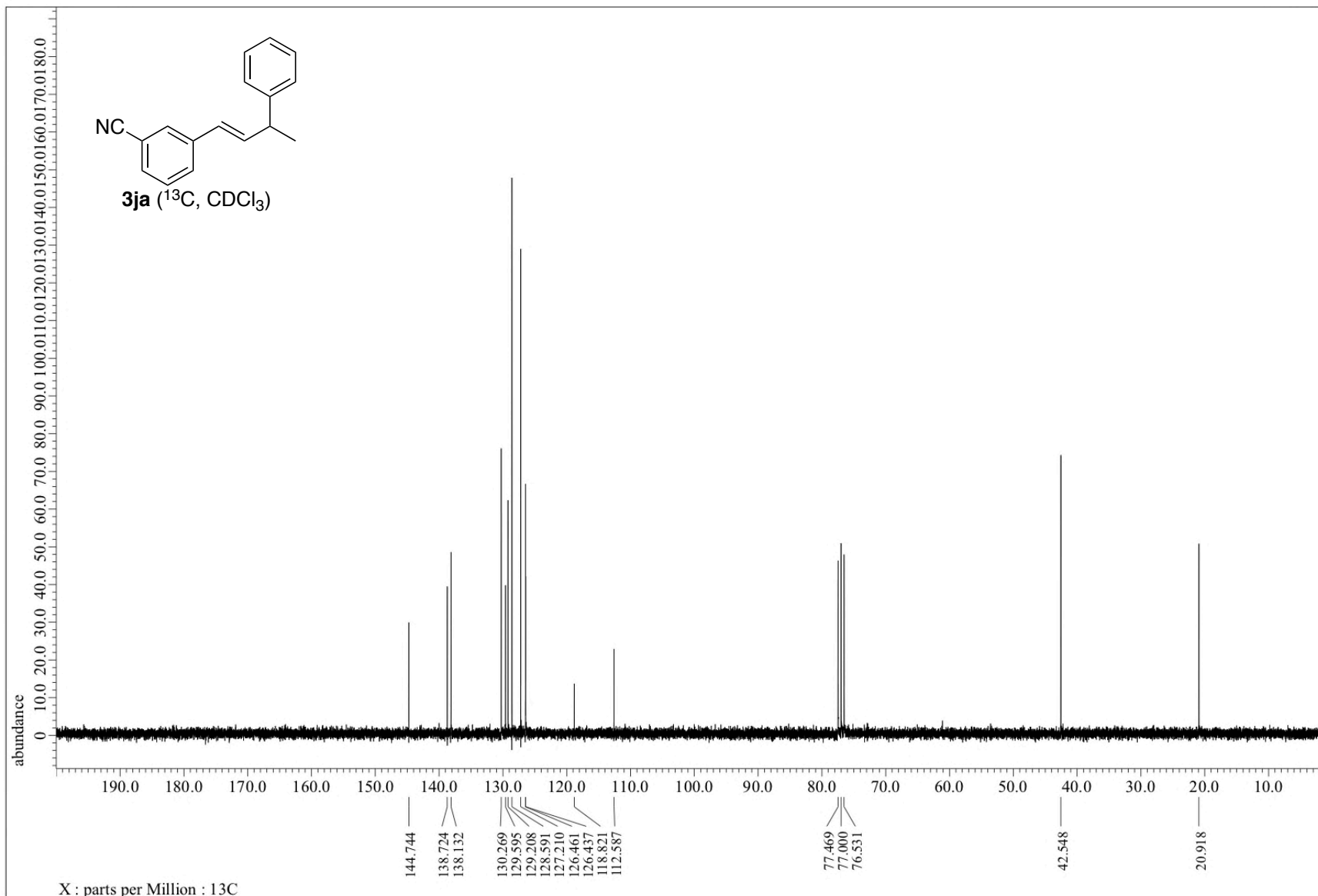


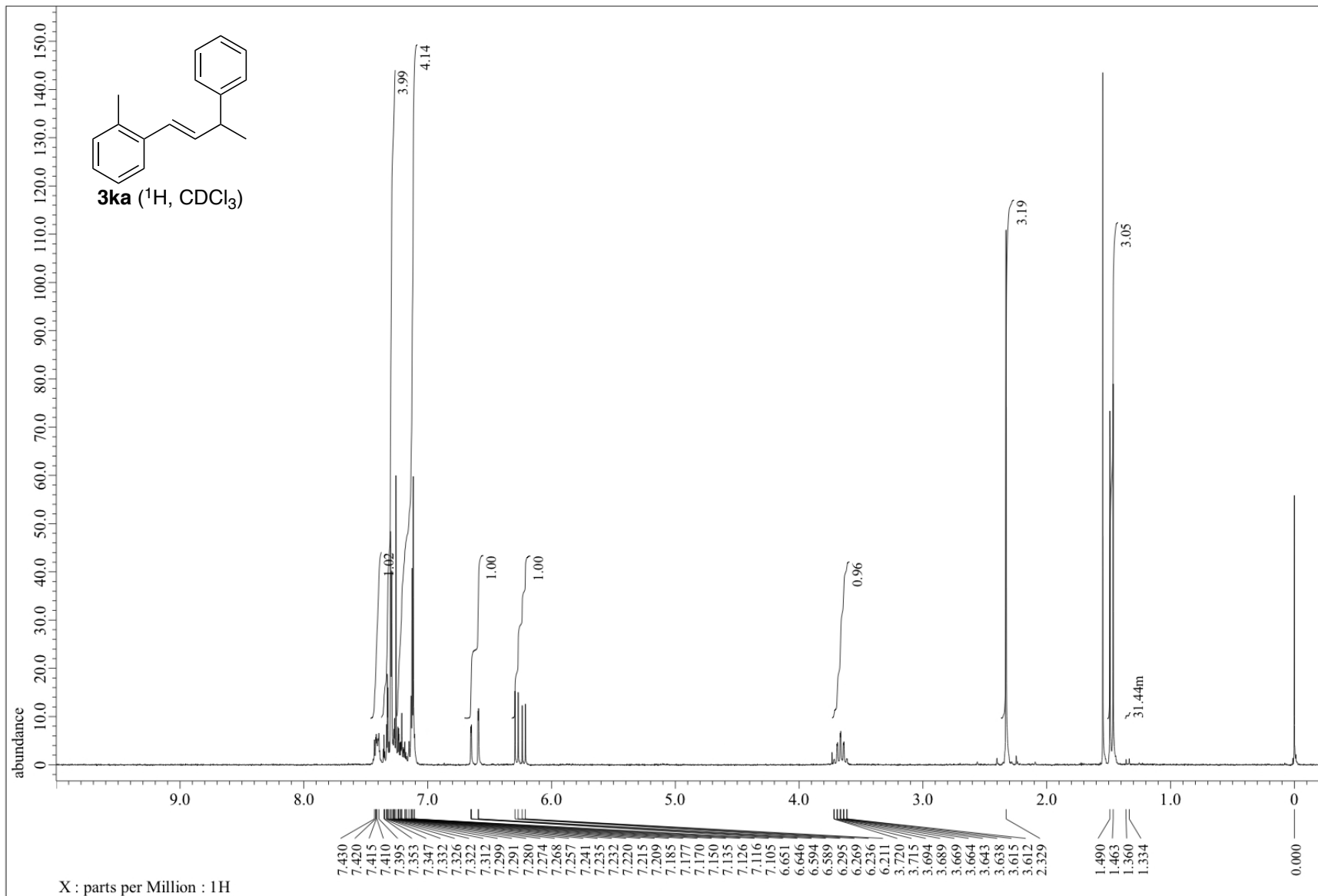


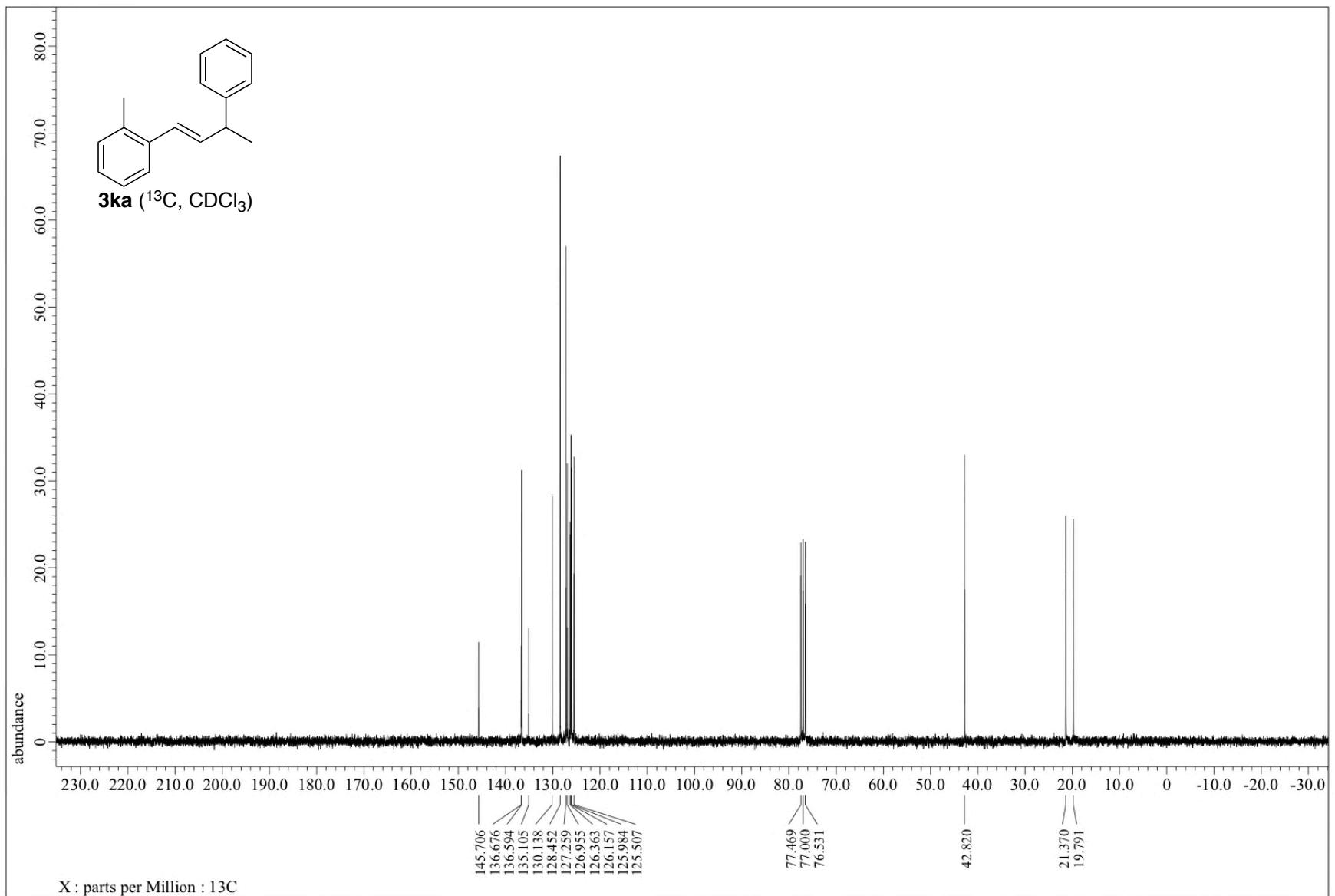


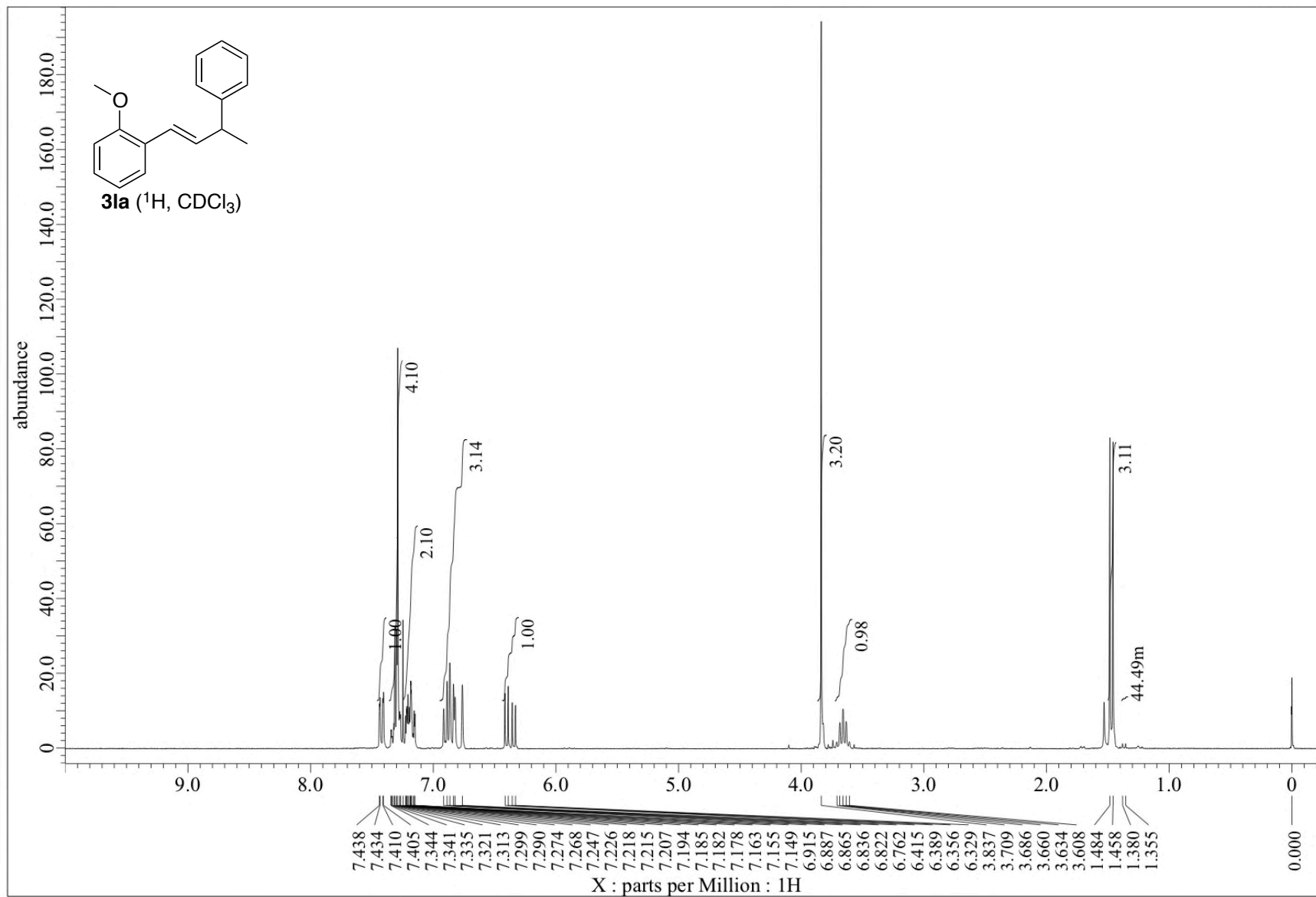


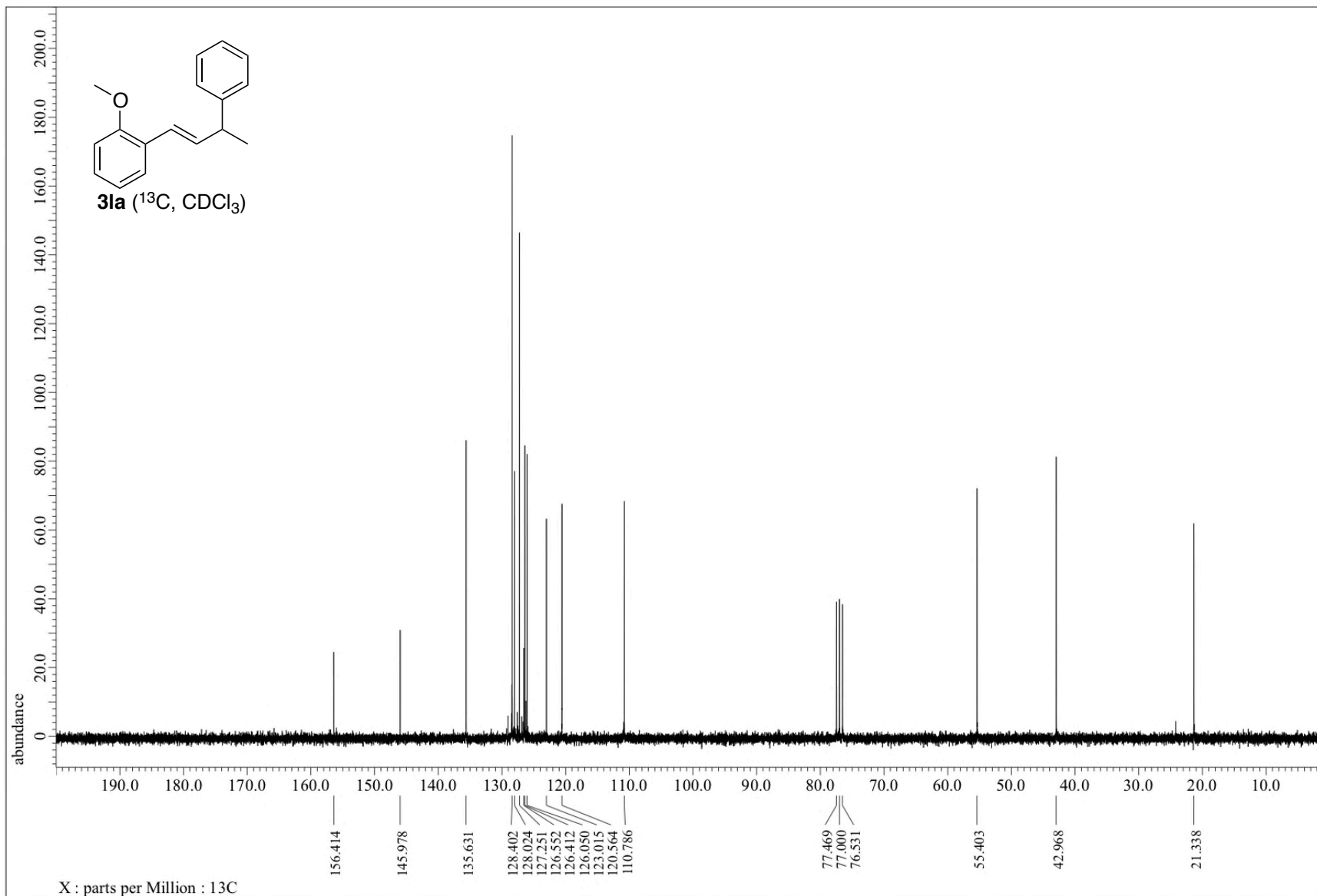


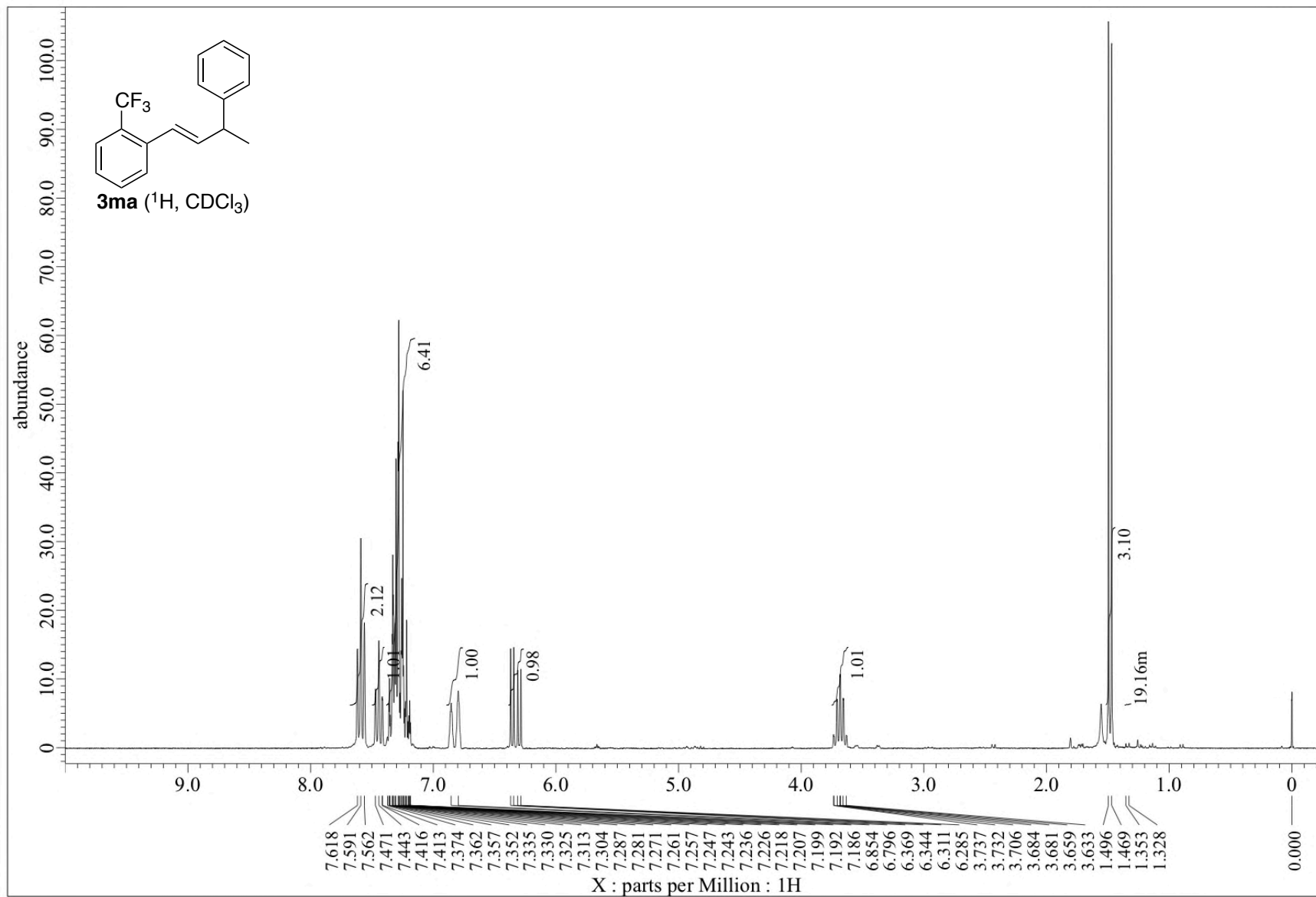


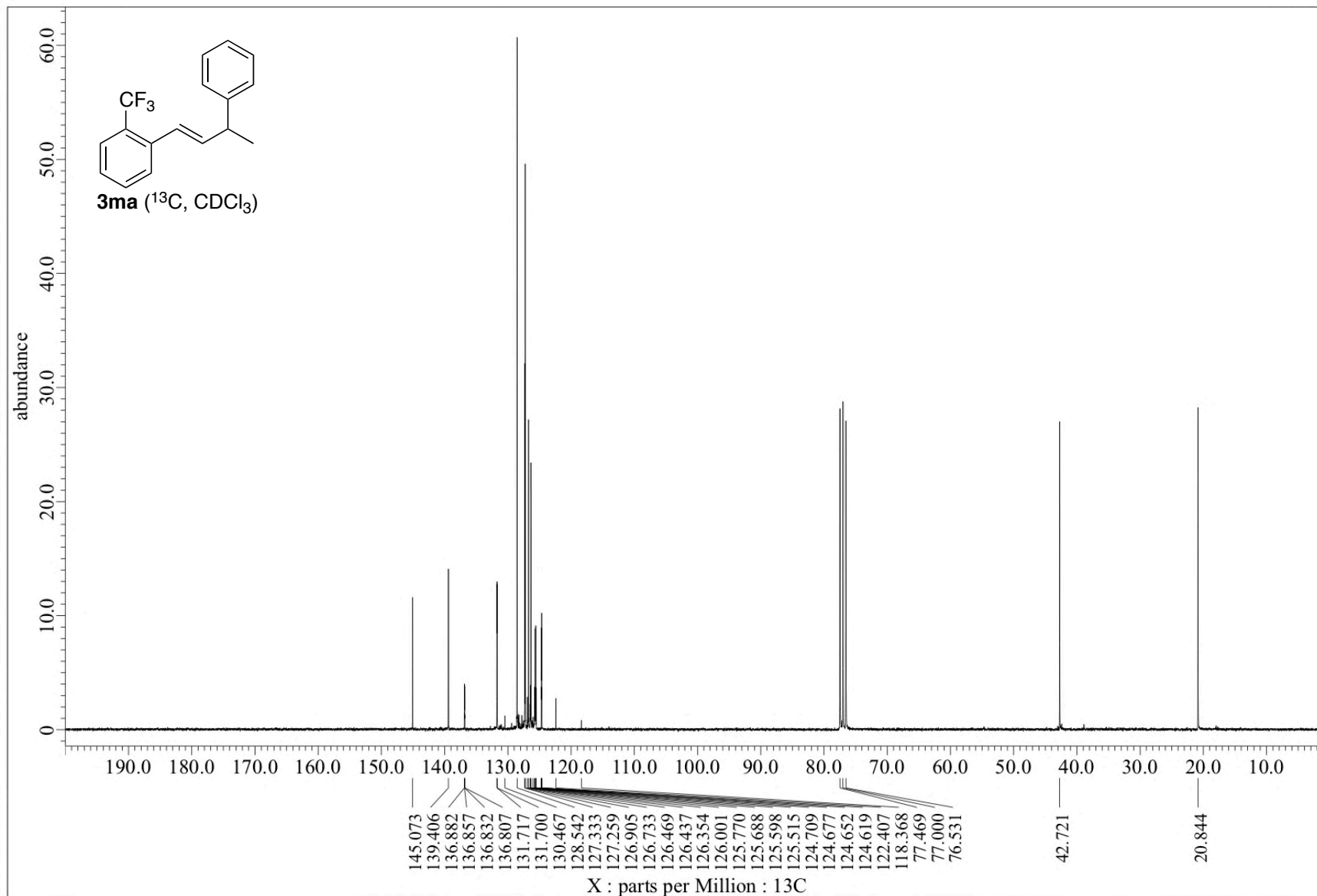


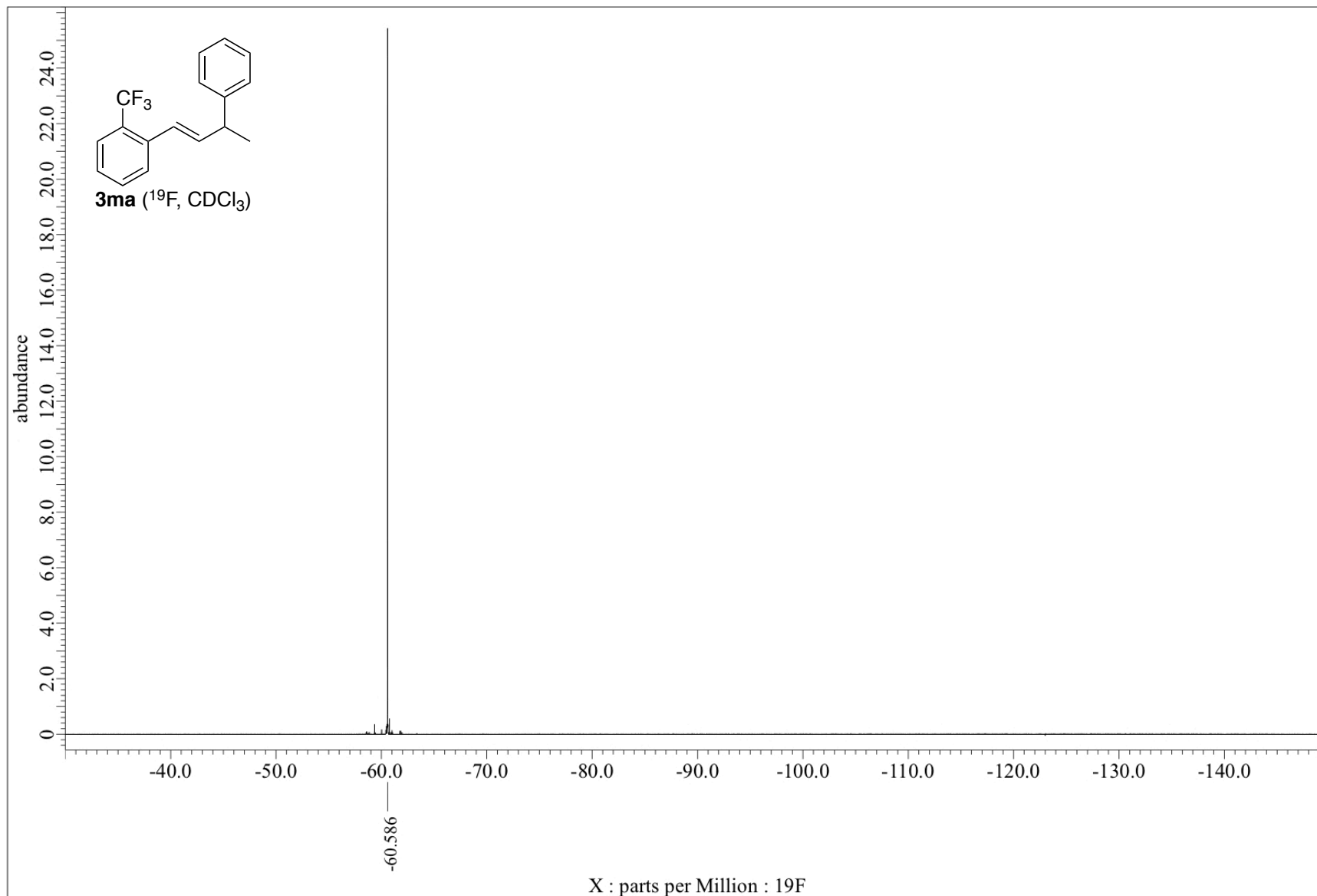












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