

Supporting Information

Rhodium-Catalyzed Arylzincation of Terminal Allenes Providing Allylzinc

Reagents

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Instrumentation and Chemicals

¹H NMR (500 MHz) and ¹³C NMR (125.7 MHz) spectra were taken on a UNITY INOVA 500 spectrometer and were recorded in CDCl₃. Chemical shifts (δ) are in parts per million relative to SiMe₄ at 0.00 ppm for ¹H and relative to CDCl₃ at 77.2 ppm for ¹³C unless otherwise noted. IR spectra were determined on a SHIMADZU FTIR-8200PC spectrometer. Mass spectra were determined on a JEOL Mstation 700 spectrometer. TLC analyses were performed on commercial glass plates bearing a 0.25-mm layer of Merck Silica gel 60F₂₅₄. Silica gel (Wakogel 200 mesh) was used for column chromatography. Elemental analyses were carried out at the Elemental Analysis Center of Kyoto University.

Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. [RhCl(cod)]₂ and P^tBu₃ were purchased from Wako Pure Chemical Industries, Ltd. THF was purchased from Kanto Chemical Co., stored under nitrogen, and used as it is. Arylzinc reagents were prepared from the corresponding aryl iodide and zinc powder (Wako Pure Chemical Industries, Ltd.) in the presence of LiCl (Wako Pure Chemical Industries, Ltd.)¹. Allenes were prepared by the reaction reported by Crabbé² and Ma³. All reactions were carried out under argon atmosphere.

¹ Krasovskiy, A.; Malakhov, V.; Gavryushin, A.; Knochel, P. *Angew. Chem., Int. Ed.* **2006**, *45*, 6040.

² Crabbé, P.; Nassim, B.; Robert-Lopes, M.-T. *Org. Synth.* **1984**, *63*, 203.

³ Kuang, J.; Ma, S. *J. Org. Chem.* **2009**, *74*, 1763.

Experimental Section

Typical procedure for rhodium-catalyzed reactions: The reaction of 1,2-tridecadiene with phenylzinc iodide • LiCl complex is representative. $[\text{RhCl}(\text{cod})]_2$ (3.7 mg, 0.0075 mmol) and P^tBu_3 (0.03 mL, 0.03 mmol, 1 M in hexane) were placed in a 20-mL reaction flask under argon. Phenylzinc iodide • LiCl complex (0.45 mL, 0.45 mmol, 1 M in THF) was added. Then, 1,2-tridecadiene (54 mg, 0.30 mmol) was added. The mixture was stirred at 25 °C for 3 h. A saturated aqueous solution of NH_4Cl (2 mL) was added. The organic compounds were extracted with ethyl acetate three times. The combined organic part was dried over Na_2SO_4 and concentrated in vacuo. Chromatographic purification on silica gel by using hexane as an eluent afforded 2-phenyl-1-tridecene (62 mg, 0.24 mmol) in 80% yield (contaminated with 7% of (*E*)-2-phenyl-2-tridecene).

Procedure for arylzincation of 1-phenyl-1,2-propadiene (1e): $[\text{RhCl}(\text{cod})]_2$ (3.7 mg, 0.0075 mmol) and P^tBu_3 (0.03 mL, 0.03 mmol, 1 M in hexane) were placed in a 20-mL reaction flask under argon. Phenylzinc iodide • LiCl complex (0.45 mL, 0.45 mmol, 1 M in THF) was added. The mixture was warmed to 66 °C. Then, 1-phenyl-1,2-propadiene (35 mg, 0.30 mmol) was slowly added over 10 min. The mixture was stirred at 66 °C for 1.5 h. After the reaction mixture was cooled to room temperature, an aqueous solution of HCl (1 M, 5 mL) was added. The organic compounds were extracted with ethyl acetate three times. The combined organic part was dried over Na_2SO_4 and concentrated in vacuo. Chromatographic purification on silica gel by using hexane as an eluent afforded 2,3-diphenyl-1-propene (49 mg, 0.25 mmol) in 84% yield (contaminated with 3% of (*E*)-1,2-diphenyl-1-propene).

Typical procedure for rhodium-catalyzed reactions followed by subsequent reaction with electrophiles: The reaction of 1,2-tridecadiene with phenylzinc iodide • LiCl complex followed by subsequent reaction with acetonitrile is representative. $[\text{RhCl}(\text{cod})]_2$ (3.7 mg, 0.0075 mmol) and P^tBu_3 (0.03 mL, 0.03 mmol, 1 M in hexane) were placed in a 20-mL reaction flask under argon. Phenylzinc iodide • LiCl complex (0.50 mL, 0.50 mmol, 1 M in THF) was added. Then, acetonitrile (12 mg, 0.3 mmol) and 1,2-tridecadiene (90 mg, 0.50 mmol) was added. The mixture was stirred at 25 °C for 1.5 h. A saturated aqueous solution of NH_4Cl (2 mL) was added. The organic compounds were extracted with ethyl acetate three times. The combined organic part was dried over Na_2SO_4 and concentrated in vacuo. Chromatographic purification on silica gel by using hexane/ethyl acetate (20/1) as an eluent afforded 3-decyl-4-phenyl-4-penten-2-one (**4a**) (72 mg, 0.24 mmol) in 81% yield.

Typical procedure for rhodium-catalyzed reactions followed by subsequent reaction with allyl bromide: The reaction of 1,2-tridecadiene with phenylzinc iodide • LiCl complex followed by subsequent

reaction with allyl bromide is representative. $[\text{RhCl}(\text{cod})]_2$ (3.7 mg, 0.0075 mmol) and P^tBu_3 (0.03 mL, 0.03 mmol, 1 M in hexane) were placed in a 20-mL reaction flask under argon. Phenylzinc iodide • LiCl complex (0.50 mL, 0.50 mmol, 1 M in THF) was added. Then, 1,2-tridecadiene (90 mg, 0.50 mmol) was added. The mixture was stirred at 25 °C for 1 h. $\text{CuCN}\cdot 2\text{LiCl}$ (0.06 mL, 0.06 mmol, 1 M in THF) and allyl bromide (36 mg, 0.30 mmol) were subsequently added to the solution. The mixture was stirred for 3 h. A saturated aqueous solution of NH_4Cl (2 mL) was added. The organic compounds were extracted with ethyl acetate three times. The combined organic part was dried over Na_2SO_4 and concentrated in vacuo. Chromatographic purification on silica gel by using hexane as an eluent afforded 3-decyl-2-phenyl-1,5-hexadiene (**5a**) (57 mg, 0.19 mmol) in 64% yield.

Synthesis of skipped polyene:

Synthesis of **7**: $[\text{RhCl}(\text{cod})]_2$ (3.7 mg, 0.0075 mmol) and P^tBu_3 (0.03 mL, 0.03 mmol, 1 M in hexane) were placed in a 20-mL reaction flask under argon. Phenylzinc iodide • LiCl complex (0.50 mL, 0.50 mmol, 1 M in THF) was added. Then, 1,2-tridecadiene (90 mg, 0.50 mmol) was added. The mixture was stirred at 25 °C for 1 h. $\text{CuCN}\cdot 2\text{LiCl}$ (0.06 mL, 0.06 mmol, 1 M in THF) and propargyl bromide (36 mg, 0.30 mmol) were subsequently added to the solution. The mixture was stirred for 3 h. A saturated aqueous solution of NH_4Cl (2 mL) was added. The organic compounds were extracted with ethyl acetate three times. The combined organic part was dried over Na_2SO_4 and concentrated in vacuo. Chromatographic purification on silica gel by using hexane/ethyl acetate (30/1) as an eluent afforded (*E*)-5-phenyl-1,2,5-hexadecatriene (**7**) (68 mg, 0.23 mmol) in 76% yield.

Synthesis of **8**: $[\text{RhCl}(\text{cod})]_2$ (3.7 mg, 0.0075 mmol) and P^tBu_3 (0.03 mL, 0.03 mmol, 1 M in hexane) were placed in a 20-mL reaction flask under argon. 4-Fluorophenylzinc iodide • LiCl complex (0.60 mL, 0.60 mmol, 1 M in THF) was added. Then, (*E*)-5-phenyl-1,2,5-hexadecatriene (**7**) (178 mg, 0.60 mmol) was added. The mixture was stirred at 25 °C for 1 h. $\text{CuCN}\cdot 2\text{LiCl}$ (0.06 mL, 0.06 mmol, 1 M in THF) and propargyl bromide (36 mg, 0.30 mmol) were subsequently added to the solution. The mixture was stirred for 3 h. A saturated aqueous solution of NH_4Cl (2 mL) was added. The organic compounds were extracted with ethyl acetate three times. The combined organic part was dried over Na_2SO_4 and concentrated in vacuo. Chromatographic purification on silica gel by using hexane/ethyl acetate (30/1) as an eluent afforded (*5E,8E*)-5-(4-fluorophenyl)-8-phenyl-1,2,5,8-nonadecatetraene (**8**) (99 mg, 0.23 mmol) in 75% yield.

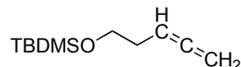
Synthesis of **9**: $[\text{RhCl}(\text{cod})]_2$ (3.7 mg, 0.0075 mmol) and P^tBu_3 (0.03 mL, 0.03 mmol, 1 M in hexane) were placed in a 20-mL reaction flask under argon. 3-Methoxyphenylzinc iodide • LiCl complex (0.45 mL, 0.45

mmol, 1 M in THF) was added. Then, (5*E*,8*E*)-5-(4-fluorophenyl)-8-phenyl-1,2,5,8-nonadecatetraene (**8**) (129 mg, 0.30 mmol) was added. The mixture was stirred at 25 °C for 1 h. CuCN•2LiCl (0.12 mL, 0.12 mmol, 1 M in THF) and propargyl bromide (71 mg, 0.60 mmol) were subsequently added to the solution. The mixture was stirred for 3 h. A saturated aqueous solution of NH₄Cl (2 mL) was added. The organic compounds were extracted with ethyl acetate three times. The combined organic part was dried over Na₂SO₄ and concentrated in vacuo. Chromatographic purification on silica gel by using hexane/ethyl acetate (30/1) as an eluent afforded (5*E*,8*E*,11*E*)-8-(4-fluorophenyl)-5-(3-methoxyphenyl)-11-phenyl-1,2,5,8,11-docosapentaene (**9**) (115 mg, 0.20 mmol) in 68% yield.

Characterization Data

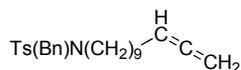
Compounds **1a**³, **1e**⁴, **3i**⁵, and **3i'**⁶ are known compounds and showed the identical spectra with those in the literature.

5-(*tert*-butyldimethylsiloxy)-1,2-pentadiene (**1b**)



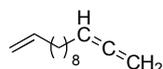
oil. IR (neat) 3735, 2930, 1954, 1717, 1507, 1257, 1102, 837, 775 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.06 (s, 6H), 0.90 (s, 9H), 2.20–2.25 (m, 2H), 3.67 (t, $J = 7.0$ Hz, 2H), 4.65 (dt, $J = 7.0$ Hz, 3.0 Hz, 2 H), 5.11 (quint., $J = 7.0$ Hz, 1H); ^{13}C NMR (CDCl_3) δ -5.08, 18.52, 26.11, 32.20, 62.98, 74.66, 86.84, 209.25; Found: C, 66.35; H, 11.45%. Calcd for $\text{C}_{11}\text{H}_{22}\text{OSi}$: C, 66.60; H, 11.18%.

N-benzyl-*N*-(10,11-dodecadienyl)-*p*-toluenesulfonamide (**1c**)



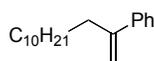
oil. IR (neat) 2924, 2854, 2500, 1952, 1597, 1157, 933, 810, 656 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.07–1.40 (m, 14H), 1.95–2.00 (m, 2H), 2.44 (s, 3H), 3.07 (t, $J = 7.5$ Hz, 2H), 4.31 (s, 2H), 4.65 (dt, $J = 3.5$ Hz, 7.0 Hz, 2H), 5.08 (quint., $J = 7.0$ Hz, 1H), 7.25–7.32 (m, 7H), 7.72–7.74 (m, 2H); ^{13}C NMR (CDCl_3) δ 21.69, 26.77, 28.07, 28.44, 29.18, 29.19, 29.27, 29.43, 29.49, 48.26, 52.04, 74.71, 90.26, 127.39, 127.87, 128.47, 128.69, 129.84, 136.83, 137.47, 143.27, 208.70; HRMS Found: 425.2380 ($\Delta = -2.1$ ppm), Calcd for $\text{C}_{26}\text{H}_{35}\text{O}_2\text{NS}$: 425.2389.

1,2,12-tridecatriene (**1d**)



oil. IR (neat) 3078, 2924, 2430, 1960, 1643, 995, 725 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.29–1.43 (m, 12H), 1.96–2.06 (m, 4H), 4.65 (dt, $J = 3.5$ Hz, 7.0 Hz, 2H), 4.92–5.01 (m, 2H), 5.09 (quint., $J = 7.0$ Hz, 1H), 5.77–5.85 (m, 1H); ^{13}C NMR (CDCl_3) δ 28.47, 29.13, 29.27, 29.32, 29.33, 29.58, 29.64, 34.01, 74.67, 90.27, 114.29, 139.39, 208.72; Found: C, 87.26; H, 12.20%. Calcd for $\text{C}_{13}\text{H}_{22}$: C, 87.56; H, 12.44%.

2-phenyl-1-tridecene (**3a**)



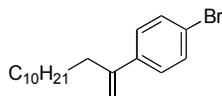
⁴ Nishina, N.; Yamamoto, Y. *Angew. Chem., Int. Ed.* **2006**, *45*, 3314.

⁵ Alacid, E.; Nájera, C. *Org. Lett.* **2008**, *10*, 5011.

⁶ Murakami, K.; Yorimitsu, H.; Oshima, K. *Org. Lett.* **2009**, *11*, 2373.

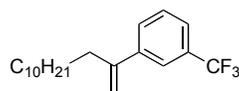
oil. IR (neat) 3065, 2925, 1738, 1628, 1466, 893, 777, 702 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.89 (t, $J = 7.0$ Hz, 3H), 1.25–1.31 (m, 16H), 1.42–1.48 (m, 2H), 2.50 (t, $J = 7.5$ Hz, 2H), 5.05 (d, $J = 1.5$ Hz, 1H), 5.26 (d, $J = 1.5$ Hz, 1H), 7.20–7.42 (m, 5H); ^{13}C NMR (CDCl_3) δ 14.30, 22.88, 28.47, 29.54 (Two signals merge.), 29.64, 29.80, 29.81, 29.84, 32.11, 35.56, 112.16, 126.31, 127.40, 128.40, 141.71, 149.02; Found: C, 88.04; H, 11.58%. Calcd for $\text{C}_{19}\text{H}_{30}$: C, 88.30; H, 11.70%.

2-(4-bromophenyl)-1-tridecene (3b)



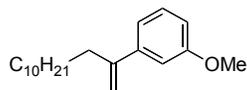
oil. IR (neat) 2924, 2853, 1628, 1075, 732 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.24–1.31 (m, 16H), 1.41 (quint., $J = 7.5$ Hz, 2H), 2.45 (t, $J = 7.5$ Hz, 2H), 5.06 (d, $J = 1.5$ Hz, 1H), 5.24 (d, $J = 1.5$ Hz, 1H), 7.23–7.28 (m, 2H), 7.40–7.45 (m, 2H); ^{13}C NMR (CDCl_3) δ 14.30, 22.88, 28.35, 29.46, 29.53, 29.61, 29.71, 29.77, 29.82, 32.11, 35.42, 112.80, 121.32, 127.99, 131.49, 140.58, 147.92; Found: C, 67.95; H, 8.82%. Calcd for $\text{C}_{19}\text{H}_{29}\text{Br}$: C, 67.65; H, 8.66%.

2-(3-trifluoromethylphenyl)-1-tridecene (3c)



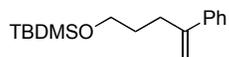
oil. IR (neat) 3085, 2925, 1631, 1468, 1074, 900, 659 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.25–1.31 (m, 16H), 1.44 (quint., $J = 7.5$ Hz, 2H), 2.50 (t, $J = 7.5$ Hz, 2H), 5.14 (d, $J = 1.0$ Hz, 1H), 5.31 (d, $J = 1.0$ Hz, 1H), 7.29–7.64 (m, 4H); ^{13}C NMR (CDCl_3) δ 14.29, 22.90, 28.29, 29.46, 29.56, 29.62, 29.79, 29.84, 29.85, 32.14, 35.38, 113.73, 123.09 (q, $J = 3.8$ Hz), 124.10 (q, $J = 3.8$ Hz), 124.45 (q, $J = 271$ Hz), 128.86, 129.58 (d, $J = 1.4$ Hz), 130.87 (q, $J = 31.5$ Hz), 142.57, 147.83; Found: C, 73.75; H, 9.19%. Calcd for $\text{C}_{20}\text{H}_{29}\text{F}_3$: C, 73.59; H, 8.95%.

2-(3-methoxyphenyl)-1-tridecene (3d)



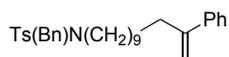
oil. IR (neat) 2924, 2854, 1852, 1049, 895, 787 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.24–1.31 (m, 16H), 1.41–1.47 (m, 2H), 2.47 (t, $J = 7.5$ Hz, 2H), 3.82 (s, 3H), 5.04 (d, $J = 1.5$ Hz, 1H), 5.25 (d, $J = 1.5$ Hz, 1H), 6.81 (m, 1H), 6.94 (m, 1H), 7.00 (m, 1H), 7.24 (m, 1H); ^{13}C NMR (CDCl_3) δ 14.31, 22.88, 28.48, 29.55 (Two signals merge.), 29.64, 29.81, 29.82, 29.84, 32.11, 35.63, 55.40, 112.32, 112.36, 112.59, 118.90, 129.33, 143.30, 148.92, 159.69; HRMS Found: 288.2456 ($\Delta = 1.1$ ppm), Calcd for $\text{C}_{20}\text{H}_{32}\text{O}$: 288.2453.

5-(*tert*-butyldimethylsiloxy)-2-phenyl-1-pentene (3f)



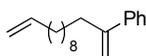
oil. IR (neat) 3905, 2929, 1772, 1447, 1100, 896, 703 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.04 (s, 6H), 0.90 (s, 9H), 1.65–1.70 (m, 2H), 2.57 (t, $J = 7.5$ Hz, 2H), 3.63 (t, $J = 6.5$ Hz, 2H), 5.07 (s, 1H), 5.29 (s, 1H), 7.26–7.34 (m, 3H), 7.40–7.43 (m, 2H); ^{13}C NMR (CDCl_3) δ -5.11, 18.53, 26.16, 31.63, 31.74, 62.75, 112.43, 125.81, 126.31, 127.49, 128.44, 148.38; Found: C, 74.02; H, 10.38%. Calcd for $\text{C}_{17}\text{H}_{28}\text{OSi}$: C, 73.85; H, 10.21%.

N-benzyl-*N*-(11-phenyl-11-dodeceny)-*p*-toluenesulfonamide (3g)



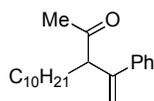
oil. IR (neat) 3032, 2924, 1805, 1597, 1157, 895, 656 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.05–1.29 (m, 14H), 1.42 (quint., $J = 7.5$ Hz, 2H), 2.43 (s, 3H), 2.48 (t, $J = 7.5$ Hz, 2H), 3.06 (t, $J = 7.5$ Hz, 2H), 4.31 (s, 2H), 5.04 (d, $J = 1.5$ Hz, 1H), 5.25 (d, $J = 1.5$ Hz, 1H), 7.24–7.34 (m, 10H), 7.37–7.41 (m, 2H), 7.71–7.73 (m, 2H); ^{13}C NMR (CDCl_3) δ 21.69, 26.77, 28.05, 28.43, 29.18, 29.48, 29.52, 29.55, 29.58, 35.54, 48.23, 52.01, 112.19, 126.29, 127.37, 127.41, 127.86, 128.40, 128.45, 128.68, 129.83, 136.81, 137.42, 141.65, 143.27, 148.95; Found: C, 76.05; H, 8.48%. Calcd for $\text{C}_{32}\text{H}_{41}\text{O}_2\text{NS}$: C, 76.30; H, 8.20%.

2-phenyl-1,12-tridecadiene (3h)



oil. IR (neat) 3078, 2924, 2854, 1636, 1458, 995, 779 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.29–1.38 (m, 12H), 1.44 (quint., $J = 7.5$ Hz, 2H), 2.01–2.05 (m, 2H), 2.45 (t, $J = 7.5$ Hz, 2H), 4.91–5.05 (m, 2H), 5.05 (d, $J = 1.5$ Hz, 1H), 5.25 (d, $J = 1.5$ Hz, 1H), 5.77–5.85 (m, 1H), 7.24–7.27 (m, 1H), 7.30–7.34 (m, 2H), 7.39–7.41 (m, 2H); ^{13}C NMR (CDCl_3) δ 28.47, 29.13, 29.32, 29.52, 29.61, 29.65, 29.72, 34.00, 35.57, 112.17, 114.28, 126.31, 127.41, 128.40, 139.45, 141.72, 149.01; Found: C, 89.00; H, 11.14%. Calcd for $\text{C}_{19}\text{H}_{28}$: C, 88.99; H, 11.01%.

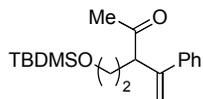
3-decyl-4-phenyl-4-penten-2-one (4a)



oil. IR (neat) 2925, 2853, 1716, 1623, 1173, 905, 704 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.23–1.30 (m, 16H), 1.60 (m, 1H), 1.90 (m, 1H), 2.12 (s, 3H), 3.56 (t, $J = 7.0$ Hz, 1H), 5.17 (s, 1H), 5.47 (s, 1H), 7.28–7.39 (m, 5H); ^{13}C NMR (CDCl_3) δ 14.30, 22.86, 27.91, 28.70, 29.50, 29.62, 29.75, 29.76, 29.80,

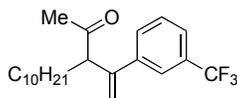
30.99, 32.08, 58.94, 115.50, 126.55, 127.99, 128.69, 141.38, 147.10, 208.76; Found: C, 83.79; H, 10.96%.
Calcd for C₂₁H₃₂O: C, 83.94; H, 10.73%.

3-[2-(*tert*-butyldimethylsiloxy)ethyl]-4-phenyl-4-penten-2-one (4b)



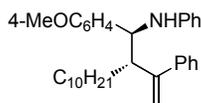
oil. IR (neat) 2955, 2862, 1713, 1620, 1466, 910, 710 cm⁻¹; ¹H NMR (CDCl₃) δ 0.01 (d, *J* = 3.5 Hz, 6H), 0.89 (s, 9H), 1.79 (m 1H), 2.13 (s, 3H), 2.19 (m, 1H), 3.56–3.63 (m, 2H), 3.90 (t, *J* = 7.0 Hz, 1H), 5.12 (s, 1H), 5.50 (s, 1H), 7.28–7.36 (m, 3H), 7.44–7.47 (m, 2H); ¹³C NMR (CDCl₃) δ -5.27, 18.43, 26.09, 29.18, 34.09, 54.46, 60.83, 115.62, 126.54, 128.07, 128.70, 141.23, 146.82, 208.23; Found: C, 71.34; H, 9.57%. Calcd for C₁₉H₃₀O₂Si: C, 71.64; H, 9.49%.

3-decyl-4-(3-trifluoromethylphenyl)-4-penten-2-one (4c)



oil. IR (neat) 2927, 2855, 1718, 1453, 1074, 806 cm⁻¹; ¹H NMR (CDCl₃) δ 0.87 (t, *J* = 7.0 Hz, 3H), 1.23–1.30 (m, 16H), 1.62 (quint., *J* = 7.0 Hz, 1H), 1.93 (quint, *J* = 7.0 Hz, 1H), 2.13 (s, 3H), 3.56 (t, *J* = 7.0 Hz, 1H), 5.27 (s, 1H), 5.52 (s, 1H), 7.45–7.62 (m, 4H); ¹³C NMR (CDCl₃) δ 14.28, 22.86, 27.86, 28.70, 29.49, 29.58, 29.73, 29.75, 31.00, 32.08, 58.83, 117.07, 123.40 (q, *J* = 3.8 Hz), 124.22 (q, *J* = 271 Hz), 124.72 (q, *J* = 3.8 Hz), 129.21, 129.83 (d, *J* = 0.9 Hz), 131.18 (q, *J* = 32 Hz), 142.20, 145.98, 208.18. (Two signals of sp³ carbon merge.); Found: C, 71.45; H, 8.66%. Calcd for C₂₂H₃₁OF₃: C, 71.71; H, 8.48%.

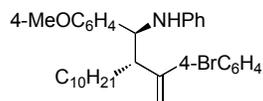
2-decyl-1-(4-methoxyphenyl)-3,*N*-diphenyl-3-butenamine (4d)⁷



oil. IR (neat) 3410, 3055, 2924, 2854, 1605, 1504, 1034, 694 cm⁻¹; ¹H NMR (CDCl₃) δ 0.87 (t, *J* = 7.0 Hz, 3H), 1.12–1.34 (m, 18H), 2.74 (m, 1H), 3.77 (s, 3H), 4.08 (d, *J* = 8.5 Hz, 1H), 4.39 (broad, 1H), 5.19 (s, 1H), 5.35 (s, 1H), 6.39–6.41 (m, 2H), 6.58 (m, 1H), 6.79–6.83 (m, 2H), 6.97–7.03 (m, 2H), 7.20–7.32 (m, 7H); ¹³C NMR (CDCl₃) δ 14.29, 22.86, 27.56, 29.49, 29.63, 29.72, 29.74, 29.76, 30.68, 32.09, 53.75, 55.38, 61.16, 113.57, 113.86, 116.53, 117.26, 127.45, 127.50, 128.45, 128.76, 129.10, 135.03, 142.47, 147.63, 150.44, 158.75; HRMS Found: 468.3257 (Δ = -2.0 ppm), Calcd for C₃₃H₄₃ON: 468.3345.

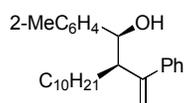
⁷ The stereochemistry of **4d** and **4e** was assigned by comparison with known compounds that have an analogous structure, see: Hopkins, C. D.; Malinakova, H. C. *Org. Lett.* **2006**, *8*, 5971.

3-(4-bromophenyl)-2-decyl-1-(4-methoxyphenyl)-*N*-phenyl-3-butenamine (4e)⁷



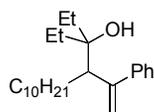
oil. IR (neat) 3410, 2924, 2854, 1605, 1504, 1034, 910, 694 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.87 (t, $J = 7.0$ Hz, 3H), 1.11–1.31 (m, 18H), 2.71 (m, 1H), 3.76 (s, 3H), 4.06 (d, $J = 8.5$ Hz, 1H), 5.22 (s, 1H), 5.34 (s, 1H), 6.40–6.42 (m, 2H), 6.60 (m, 1H), 6.76–6.82 (m, 2H), 7.00–7.04 (m, 2H), 7.06–7.12 (m, 2H), 7.15–7.20 (m, 2H), 7.37–7.39 (m, 2H) (NH was not observed.); ^{13}C NMR (CDCl_3) δ 14.30, 22.86, 27.59, 29.49, 29.62, 29.67, 29.73, 29.74, 30.61, 32.08, 53.72, 55.40, 60.70, 113.58, 113.89, 117.10, 117.46, 121.53, 128.66, 129.17, 129.18, 131.49, 134.63, 141.19, 147.32, 149.43, 158.80; Found: C, 72.07; H, 7.66%. Calcd for $\text{C}_{33}\text{H}_{42}\text{ONBr}$: C, 72.25; H, 7.72%.

2-decyl-1-(2-methylphenyl)-3-phenyl-3-buten-1-ol (4f)⁸



oil. IR (neat) 3441, 2924, 2854, 1628, 1466, 903, 702 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.87 (t, $J = 7.0$ Hz, 3H), 1.06–1.30 (m, 16H), 1.69 (q, $J = 7.0$ Hz, 2H), 1.92 (d, $J = 2.5$ Hz, 1H), 2.07 (s, 3H), 2.91 (dt, $J = 5.0$ Hz, 7.0 Hz, 1H), 4.76 (dd, $J = 2.5$ Hz, 5.0 Hz, 1H), 5.20 (s, 1H), 5.36 (s, 1H), 7.03 (m, 1H), 7.07–7.13 (m, 2H), 7.19–7.29 (m, 5H), 7.41 (m, 1H); ^{13}C NMR (CDCl_3) δ 14.31, 19.35, 22.87, 27.40, 27.68, 29.51, 29.69, 29.79, 29.81, 30.08, 32.10, 50.20, 72.00, 114.25, 125.80, 126.84, 126.99, 127.12, 127.49, 128.33, 130.45, 134.55, 140.71, 143.52, 150.69; Found: C, 85.64; H, 10.03%. Calcd for $\text{C}_{27}\text{H}_{38}\text{O}$: C, 85.66; H, 10.12%.

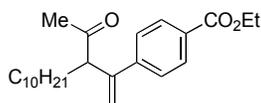
4-decyl-3-ethyl-5-phenyl-5-hexen-3-ol (4g)



oil. IR (neat) 3487, 2924, 1618, 1464, 1378, 1123, 779 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.64 (t, $J = 7.5$ Hz, 3H), 0.79 (t, $J = 7.5$ Hz, 3H), 0.88 (t, $J = 7.5$ Hz, 3H), 1.21–1.40 (m, 18H), 1.45–1.55 (m, 2H), 1.60–1.73 (m, 2H), 2.76 (dd, $J = 3.5$ Hz, 12 Hz, 1H), 5.20 (d, $J = 1.0$ Hz, 1H), 5.46 (d, $J = 1.0$ Hz, 1H), 7.25 (m, 1H), 7.30–7.34 (m, 2H), 7.36–7.39 (m, 2H) (OH was not observed.); ^{13}C NMR (CDCl_3) δ 7.90, 8.15, 14.30, 22.88, 27.59, 28.38, 29.46, 29.52, 29.78, 29.82, 29.86, 30.00, 30.38, 32.10, 50.04, 76.85, 115.38, 126.66, 127.26, 128.54, 145.84, 150.69; Found: C, 83.37; H, 11.89%. Calcd for $\text{C}_{24}\text{H}_{40}\text{O}$: C, 83.66; H, 11.70%.

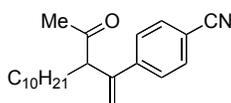
⁸ The stereochemistry of **4f** was assigned by comparison with known compounds that have an analogous structure, see: Hopkins, C. D.; Malinakova, H. C. *Org. Lett.* **2004**, *6*, 2221.

3-decyl-4-(4-ethoxycarbonylphenyl)-4-penten-2-one (4h)



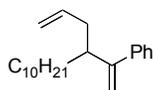
oil. IR (neat) 2924, 2854, 2361, 1720, 1612, 1180, 779 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.87 (t, $J = 7.5$ Hz, 3H), 1.23–1.30 (m, 16H), 1.40 (t, $J = 7.0$ Hz, 3H), 1.61 (m, 1H), 1.91 (m, 1H), 2.12 (s, 3H), 3.57 (t, $J = 7.0$ Hz, 1H), 4.38 (q, $J = 7.0$ Hz, 2H), 5.27 (s, 1H), 5.55 (s, 1H), 7.42–7.45 (m, 2H), 8.00–8.03 (m, 2H); ^{13}C NMR (CDCl_3) δ 14.28, 14.51, 22.85, 27.85, 28.71, 29.48, 29.58, 29.72, 29.75 (Two signals merge.), 30.96, 32.07, 58.77, 61.17, 117.17, 126.53, 129.99, 130.01, 145.73, 146.41, 166.44, 208.31; Found: C, 77.08; H, 9.75%. Calcd for $\text{C}_{24}\text{H}_{36}\text{O}_3$: C, 77.38; H, 9.74%.

3-decyl-4-(4-cyanophenyl)-4-penten-2-one (4g)



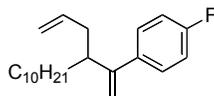
oil. IR (neat) 2924, 2854, 2361, 2230, 1713, 1605, 918 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.23–1.30 (m, 16H), 1.60 (quint., $J = 7.0$ Hz, 1H), 1.92 (quint., $J = 7.0$ Hz, 1H), 2.13 (s, 3H), 3.53 (t, $J = 7.0$ Hz, 1H), 5.34 (s, 1H), 5.56 (s, 1H), 7.45–7.48 (m, 2H), 7.62–7.65 (m, 2H); ^{13}C NMR (CDCl_3) δ 14.28, 22.85, 27.83, 28.55, 29.48, 29.56, 29.71, 29.74 (Two signals merge.), 30.90, 32.07, 58.76, 117.74, 118.33, 118.81, 127.31, 132.54, 145.73, 145.82, 207.97; Found: C, 81.26; H, 9.78%. Calcd for $\text{C}_{22}\text{H}_{31}\text{ON}$: C, 81.18; H, 9.60%.

3-decyl-2-phenyl-1,5-hexadiene (5a)



oil. IR (neat) 2924, 2854, 2361, 1636, 995, 702 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.23–1.36 (m, 16H), 1.47 (q, $J = 7.0$ Hz, 2H), 2.14–2.29 (m, 2H), 2.61 (quint., $J = 7.0$ Hz, 1H), 4.96–5.02 (m, 2H), 5.02 (d, $J = 1.5$ Hz, 1H), 5.24 (d, $J = 1.5$ Hz, 1H), 5.78 (m, 1H), 7.23–7.34 (m, 5H); ^{13}C NMR (CDCl_3) δ 14.31, 22.88, 27.19, 29.53, 29.76, 29.80, 29.82, 30.01, 32.11, 33.89, 38.97, 44.06, 112.66, 115.97, 126.97, 127.21, 128.28, 137.31, 143.54, 152.61; Found: C, 88.30; H, 11.71%. Calcd for $\text{C}_{22}\text{H}_{34}$: C, 88.52; H, 11.48%.

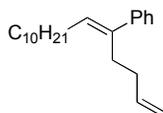
3-decyl-2-(4-fluorophenyl)-1,5-hexadiene (5b)



oil. IR (neat) 3086, 2924, 2854, 1605, 1512, 995, 841 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.23–1.34 (m, 16H), 1.41–1.48 (m, 2H), 2.13–2.27 (m, 2H), 2.56 (quint., $J = 7.0$ Hz, 1H), 4.96–5.01 (m, 2H),

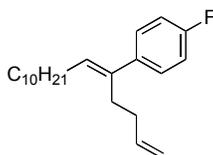
5.01 (d, $J = 1.0$ Hz, 1H), 5.20 (d, $J = 1.0$ Hz, 1H), 5.72–5.80 (m, 1H), 6.97–7.02 (m, 2H), 7.26–7.30 (m, 2H); ^{13}C NMR (CDCl_3) δ 14.30, 22.88, 27.18, 29.53, 29.75, 29.80, 29.81, 29.98, 32.11, 33.89, 38.90, 44.29, 112.81, 115.08 (d, $J = 21$ Hz), 116.09, 128.52 (d, $J = 8.0$ Hz), 137.14, 139.52, 151.65, 162.30 (d, $J = 243$ Hz); HRMS Found: 316.2568 ($\Delta = 0.5$ ppm), Calcd for $\text{C}_{22}\text{H}_{33}\text{F}$: 316.2566.

(E)-5-phenyl-1,5-hexadecadiene (6a)



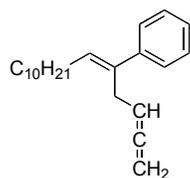
oil. IR (neat) 2924, 2854, 1643, 1597, 995, 756 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.27–1.36 (m, 14H), 1.44 (quint., $J = 7.0$ Hz, 2H), 2.05–2.11 (m, 2H), 2.18 (q, $J = 7.5$ Hz, 2H), 2.58 (t, $J = 7.5$ Hz, 2H), 4.92–5.00 (m, 2H), 5.67 (t, $J = 7.5$ Hz, 1H), 5.81 (m, 1H), 7.21 (m, 1H), 7.28–7.34 (m, 4H); ^{13}C NMR (CDCl_3) δ 14.31, 22.89, 28.79, 29.44, 29.55, 29.63, 29.79, 29.84 (Two signals merge.), 30.07, 32.12, 33.02, 114.67, 126.57, 126.65, 128.36, 129.95, 138.61, 139.34, 143.37; Found: C, 88.70; H, 11.77%. Calcd for $\text{C}_{22}\text{H}_{34}$: C, 88.52; H, 11.48%.

(E)-5-(4-fluorophenyl)-1,5-hexadecadiene (6b)



oil. IR (neat) 3077, 2925, 2854, 1641, 1603, 1158, 832 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.27–1.36 (m, 14H), 1.43 (quint., $J = 7.5$ Hz, 2H), 2.06 (q, $J = 7.5$ Hz, 2H), 2.16 (q, $J = 7.5$ Hz, 2H), 2.55 (t, $J = 7.5$ Hz, 2H), 4.92–4.99 (m, 2H), 5.60 (t, $J = 7.5$ Hz, 1H), 5.75–5.83 (m, 1H), 6.95–7.00 (m, 2H), 7.25–7.29 (m, 2H); ^{13}C NMR (CDCl_3) δ 14.29, 22.88, 28.77, 29.54, 29.58, 29.62, 29.77, 29.83 (Two signals merge.), 30.05, 32.11, 32.89, 114.81, 115.09 (d, $J = 21$ Hz), 128.05 (d, $J = 7.7$ Hz), 129.96, 138.40, 138.45, 139.43 (d, $J = 3.4$ Hz), 161.97 (d, $J = 243$ Hz); Found: C, 83.30; H, 10.47%. Calcd for $\text{C}_{22}\text{H}_{33}\text{F}$: C, 83.49; H, 10.51%.

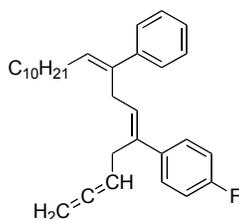
(E)-5-phenyl-1,2,5-hexadecatriene (7)



oil. IR (neat) 2924, 2854, 1952, 1597, 1458, 841, 756, 694 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H),

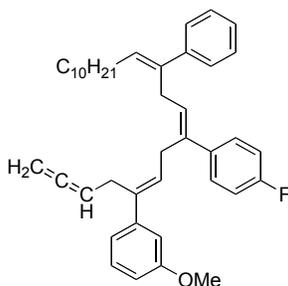
1.27–1.36 (m, 14H), 1.45 (quint., $J = 7.5$ Hz, 2H), 2.20 (q, $J = 7.5$ Hz, 2H), 3.20 (dt, $J = 3.5$ Hz, 7.0 Hz, 2H), 4.63 (dt, $J = 3.5$ Hz, 7.0 Hz, 2H), 5.10 (quint., $J = 7.0$ Hz, 1H), 5.80 (t, $J = 7.5$ Hz, 1H), 7.21 (m, 1H), 7.26–7.31 (m, 2H), 7.36–7.38 (m, 2H); ^{13}C NMR (CDCl_3) δ 14.30, 22.88, 28.92, 29.49, 29.54, 29.65, 29.79, 29.83, 29.84, 29.93, 32.11, 75.46, 88.91, 126.36, 126.70, 128.32, 130.73, 137.24, 143.09, 209.15; HRMS Found: 296.2503 ($\Delta = -0.2$ ppm), Calcd for $\text{C}_{22}\text{H}_{32}$: 296.2504.

(5E,8E)-5-(4-fluorophenyl)-8-phenyl-1,2,5,8-nonadecatetraene (8)



oil. IR (neat) 3024, 2924, 2854, 1952, 1597, 1504, 1458, 1227, 1157, 841, 756, 694 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88–0.90 (m, 3H), 1.26–1.38 (m, 14H), 1.43–1.49 (m, 2H), 2.23 (q, $J = 7.5$ Hz, 2H), 3.24 (dt, $J = 3.5$ Hz, 7.0 Hz, 2H), 3.39 (d, $J = 6.5$ Hz, 2H), 4.63 (dt, $J = 3.5$ Hz, 7.0 Hz, 2H), 5.08 (quint., $J = 6.5$ Hz, 1H), 5.62 (t, $J = 6.5$ Hz, 1H), 5.79 (t, $J = 7.5$ Hz, 1H), 6.91–6.96 (m, 2H), 7.20–7.26 (m, 3H), 7.27–7.31 (m, 2H), 7.36–7.38 (m, 2H); ^{13}C NMR (CDCl_3) δ 14.29, 22.88, 28.97, 29.54, 29.67, 29.80, 29.83, 29.86, 29.99, 32.11, 75.74, 88.23, 115.04 (d, $J = 21$ Hz), 126.42, 126.80, 127.93 (d, $J = 7.6$ Hz), 128.39, 128.63, 130.29, 136.76, 138.20, 138.71, 143.28, 162.06 (d, $J = 244$ Hz), 209.21. (Three signals merge.); HRMS Found: 430.3029 ($\Delta = -1.7$ ppm), Calcd for $\text{C}_{31}\text{H}_{39}\text{F}$: 430.3036.

(5E,8E,11E)-8-(4-fluorophenyl)-5-(3-methoxyphenyl)-11-phenyl-1,2,5,8,11-docosapentaene (9)



oil. IR (neat) 3441, 2924, 2854, 1952, 1682, 1597, 1504, 1234, 1142, 841, 702, 617 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.88 (t, $J = 7.0$ Hz, 3H), 1.25–1.32 (m, 14H), 1.46 (m, 2H), 2.23 (q, $J = 7.5$ Hz, 2H), 3.26 (dt, $J = 3.5$ Hz, 6.5 Hz, 2H), 3.43 (m, 4H), 3.78 (s, 3H), 4.60 (dt, $J = 3.5$ Hz, 6.5 Hz, 2H), 5.12 (quint., $J = 6.5$ Hz, 1H), 5.61 (t, $J = 6.5$ Hz, 1H), 5.65 (t, $J = 6.5$ Hz, 1H), 5.79 (t, $J = 7.5$ Hz, 1H), 6.77 (m, 1H), 6.83 (m, 1H), 6.88 (m, 1H), 6.90–6.95 (m, 2H), 7.18–7.29 (m, 6H), 7.35–7.38 (m, 2H); ^{13}C NMR (CDCl_3) δ 14.30, 22.88, 29.03, 29.55, 29.69, 29.82, 29.83, 29.85, 29.93, 29.97, 30.01, 30.25, 32.20, 55.83, 75.79, 88.33, 112.12, 112.51, 115.10 (d,

$J = 21$ Hz), 118.98, 126.44, 126.83, 127.94 (d, $J = 7.6$ Hz), 128.04, 128.16, 128.42, 129.28, 130.34, 137.70, 138.18, 138.21, 138.98, 143.26, 144.04, 159.69, 162.08 (d, $J = 244$ Hz), 209.17.; HRMS Found: 576.3760 ($\Delta = -1.3$ ppm), Calcd for $C_{41}H_{49}FO$: 576.3767.

Figure S1. ^1H NMR spectrum of **1a**

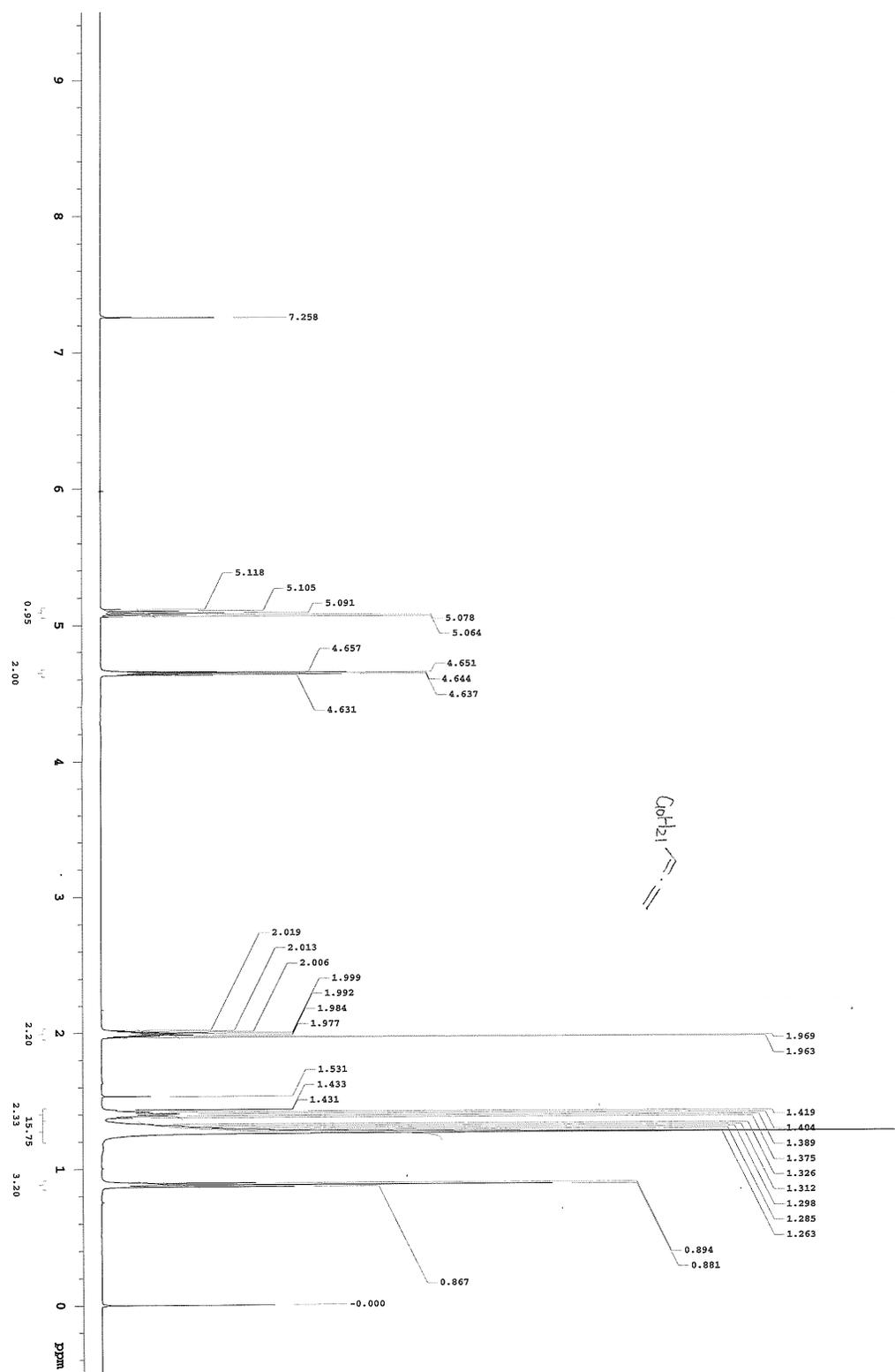


Figure S2. ^{13}C NMR spectrum of **1a**

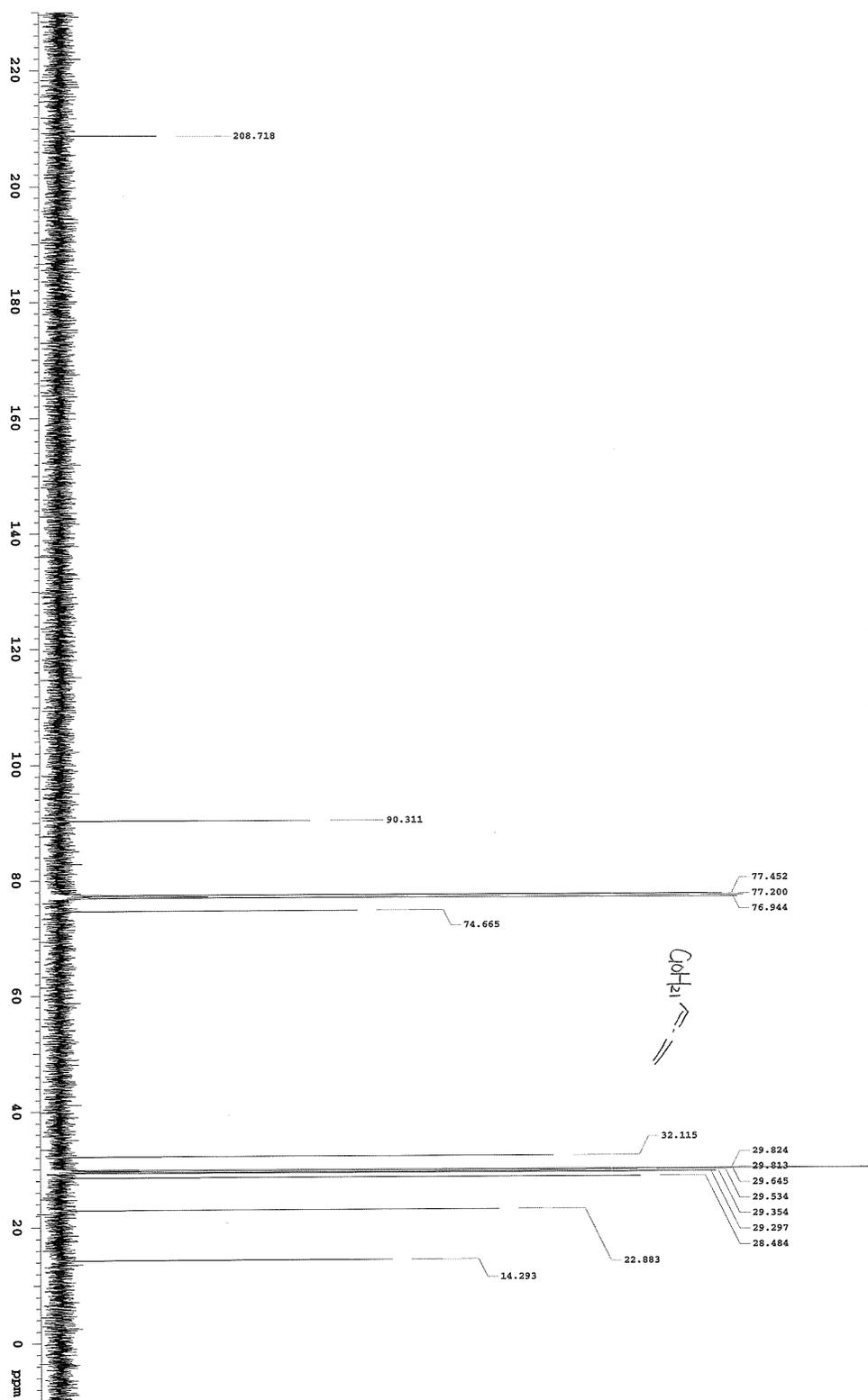


Figure S3. ^1H NMR spectrum of **1b**

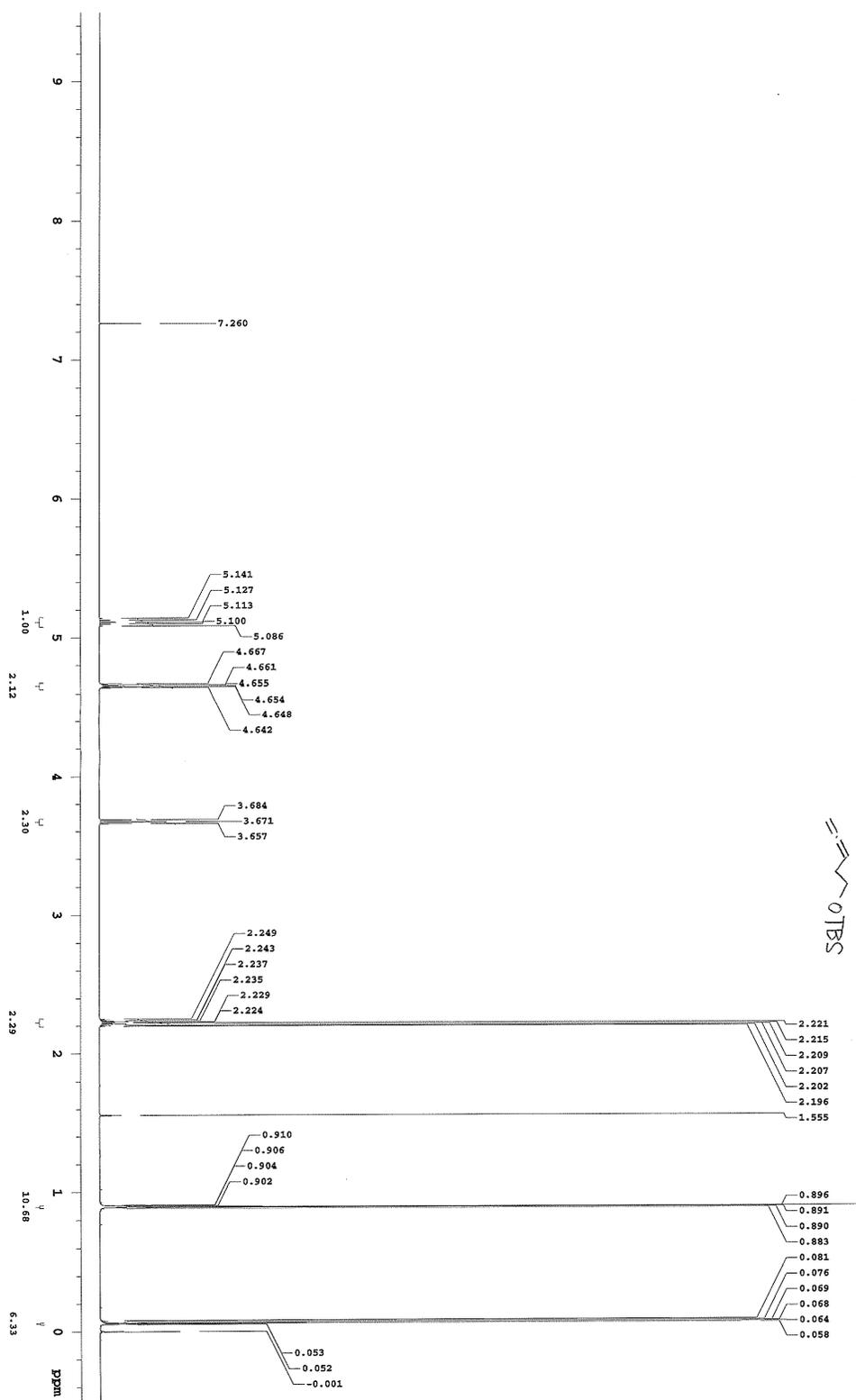


Figure S4. ^{13}C NMR spectrum of **1b**

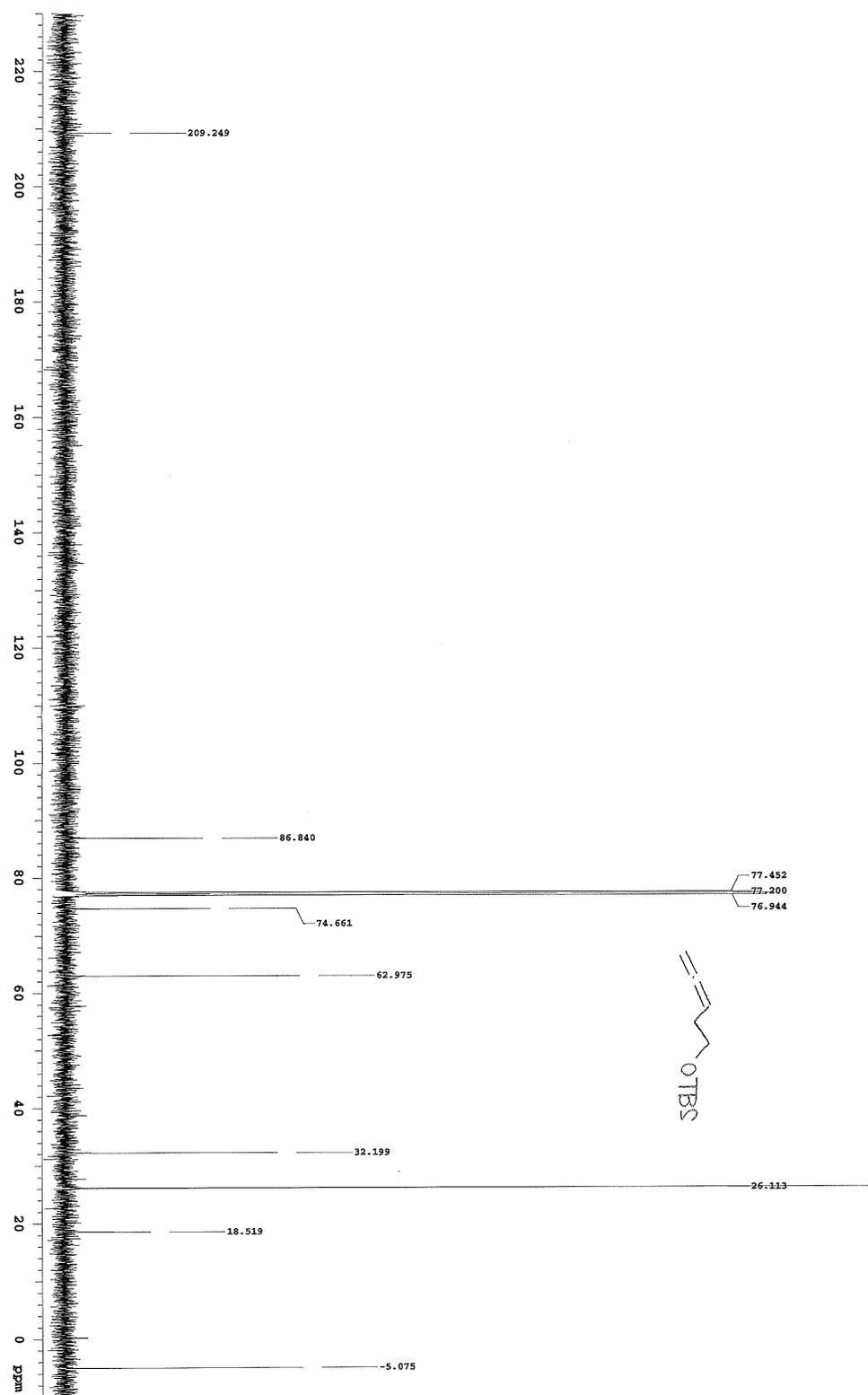


Figure S5. ¹H NMR spectrum of **1c**

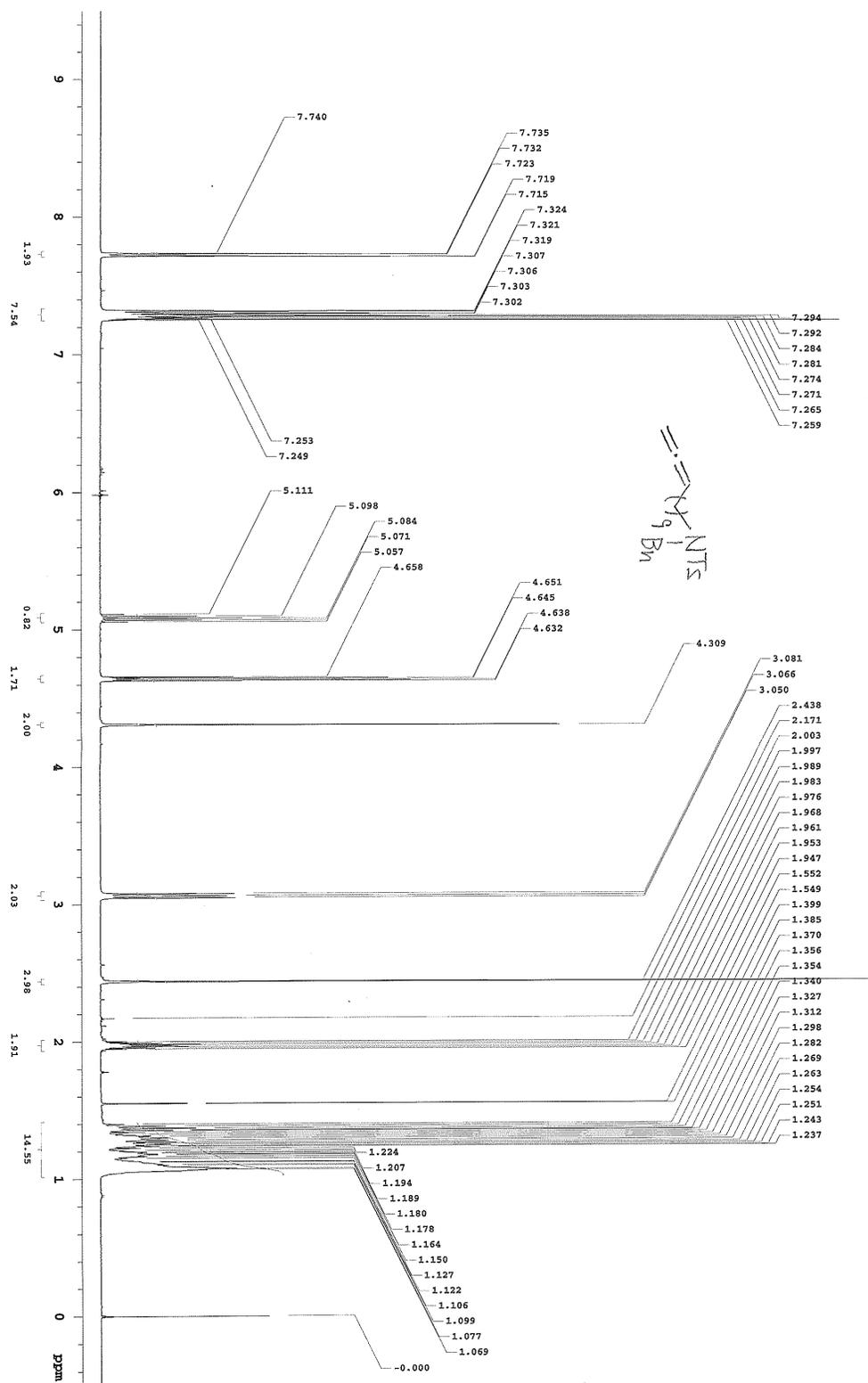


Figure S6. ^{13}C NMR spectrum of **1c**

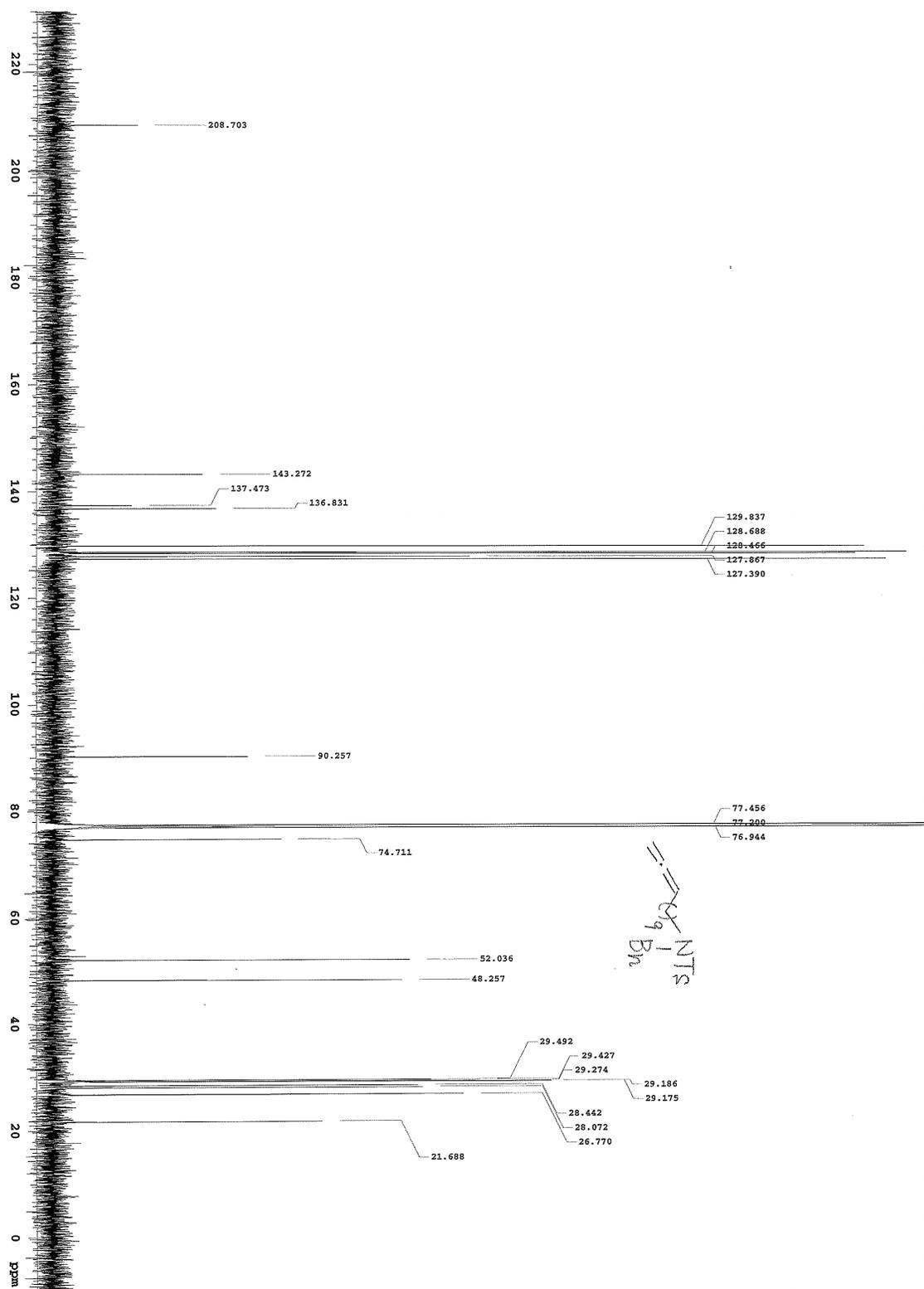


Figure S7. ^1H NMR spectrum of **1d**

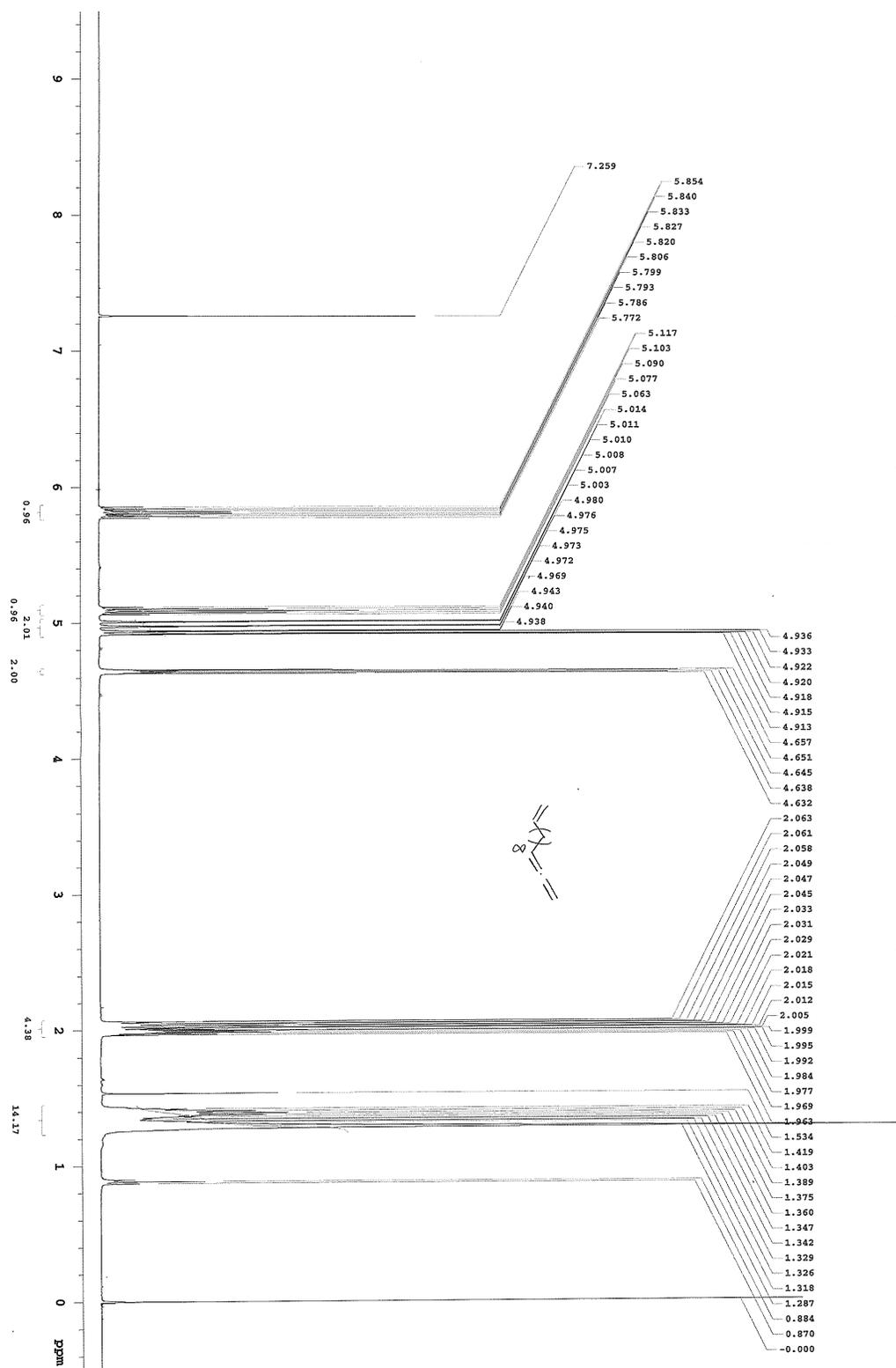


Figure S8. ^{13}C NMR spectrum of **1d**

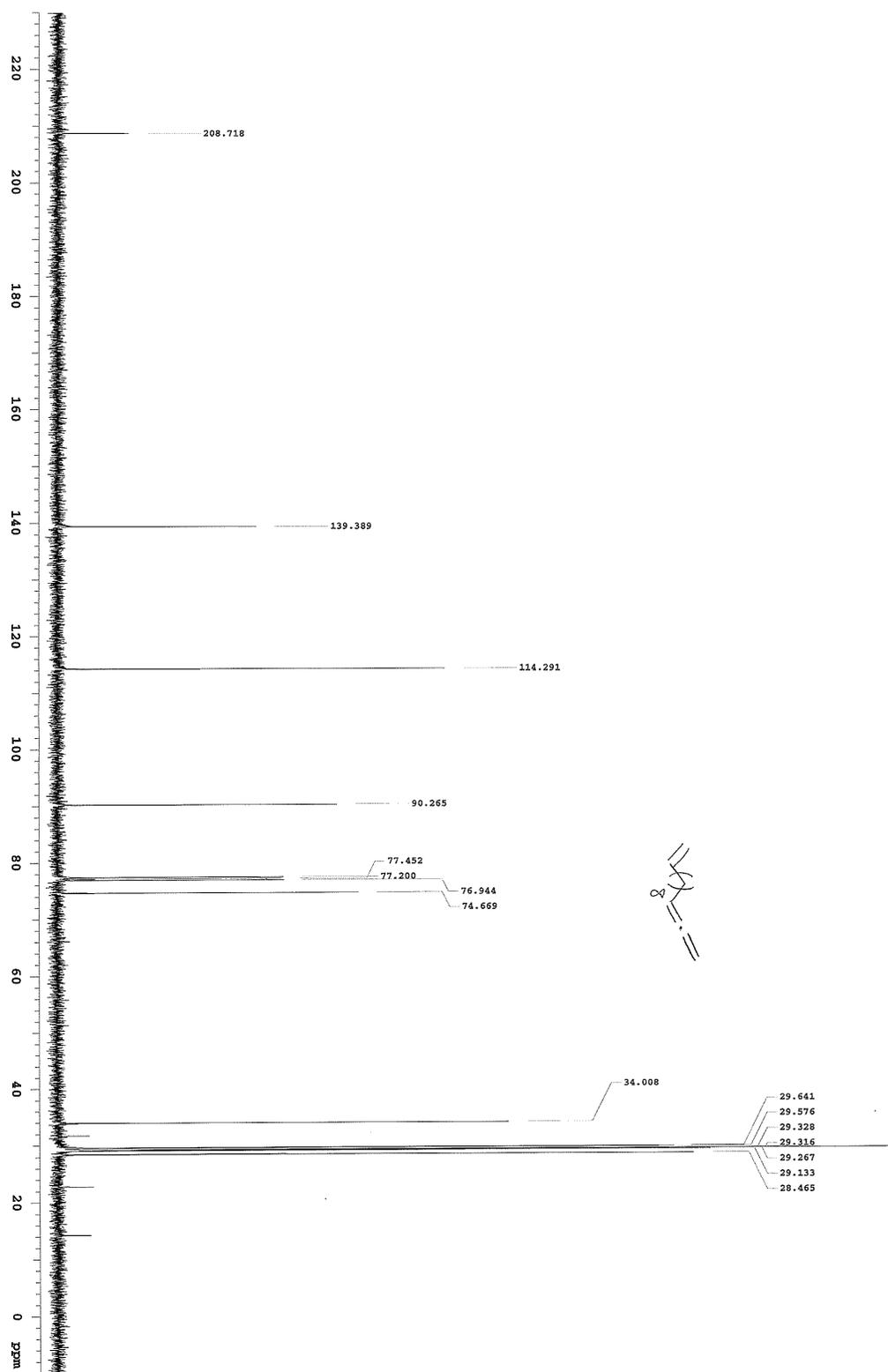


Figure S9. ^1H NMR spectrum of **1e**

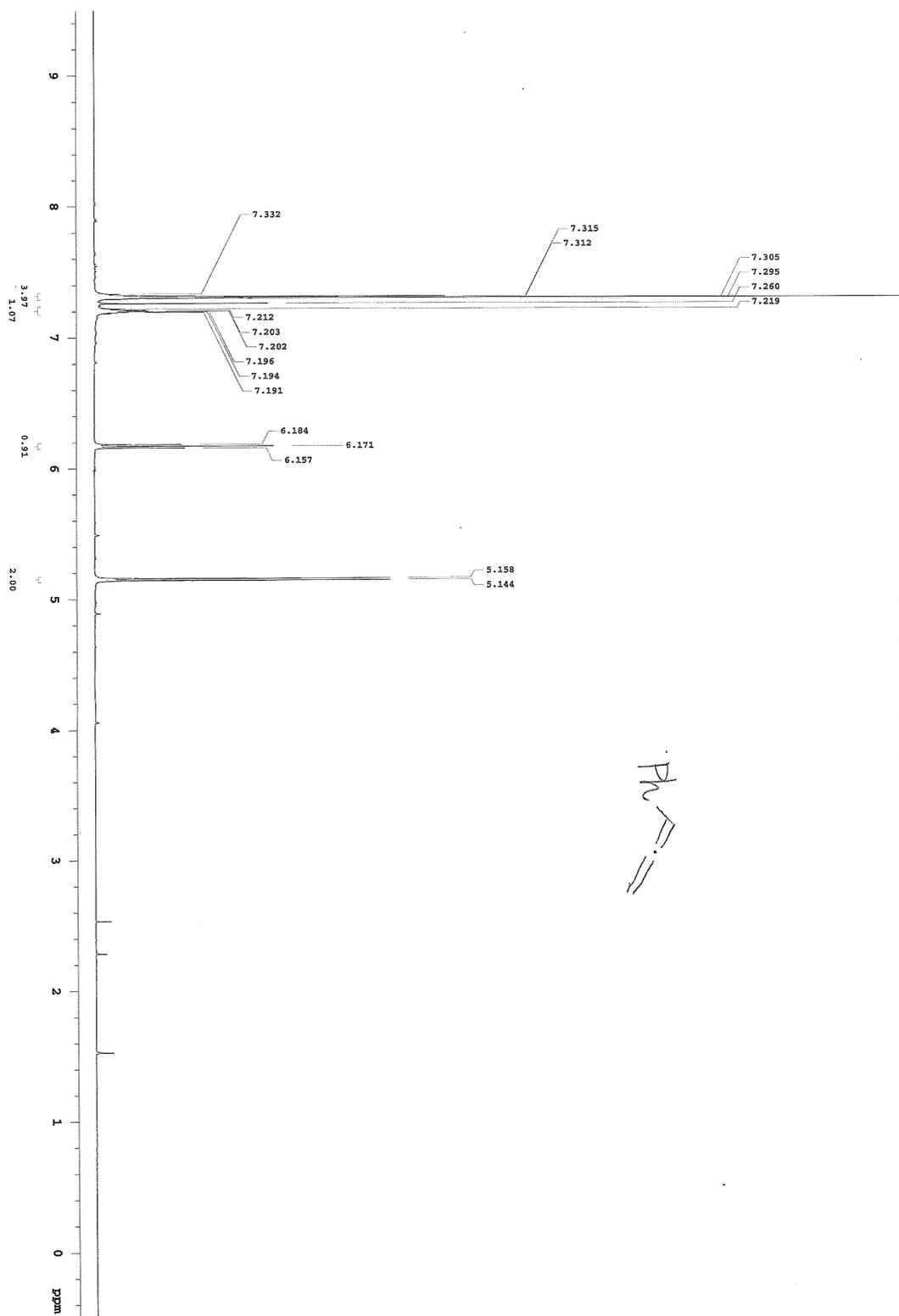


Figure S10. ^{13}C NMR spectrum of **1e**

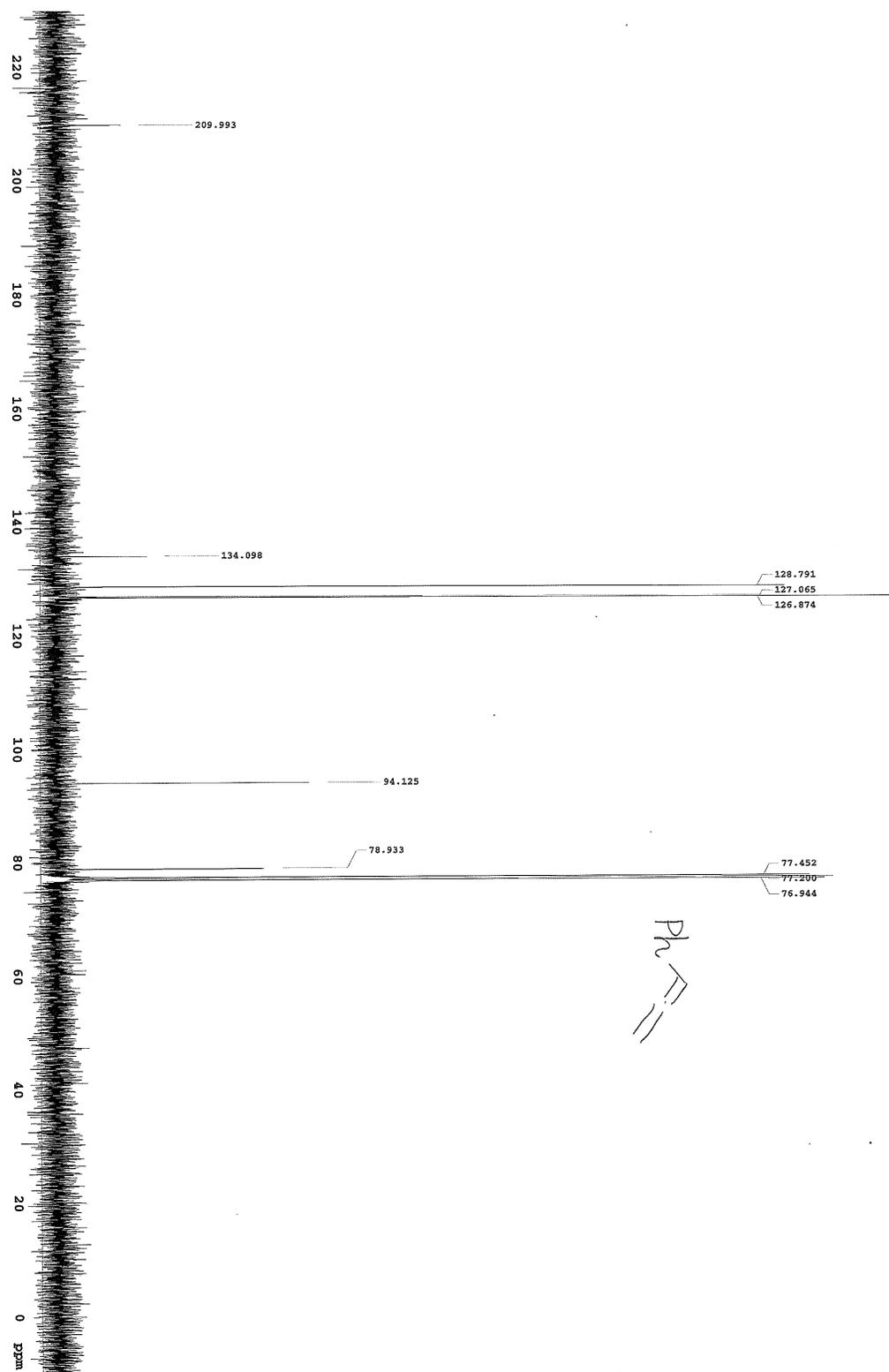


Figure S11. ^1H NMR spectrum of **3a**

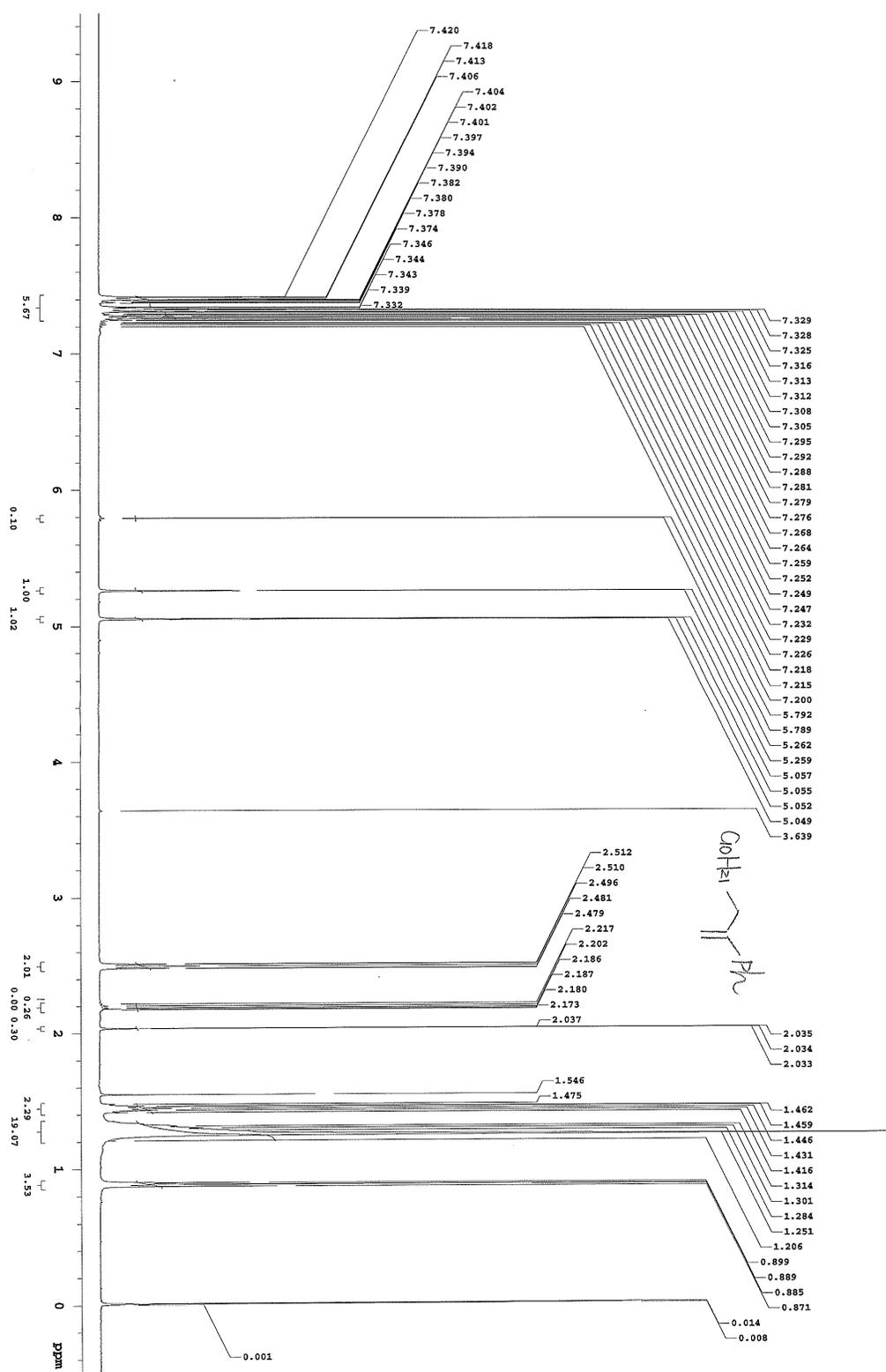


Figure S12. ^{13}C NMR spectrum of **3a**

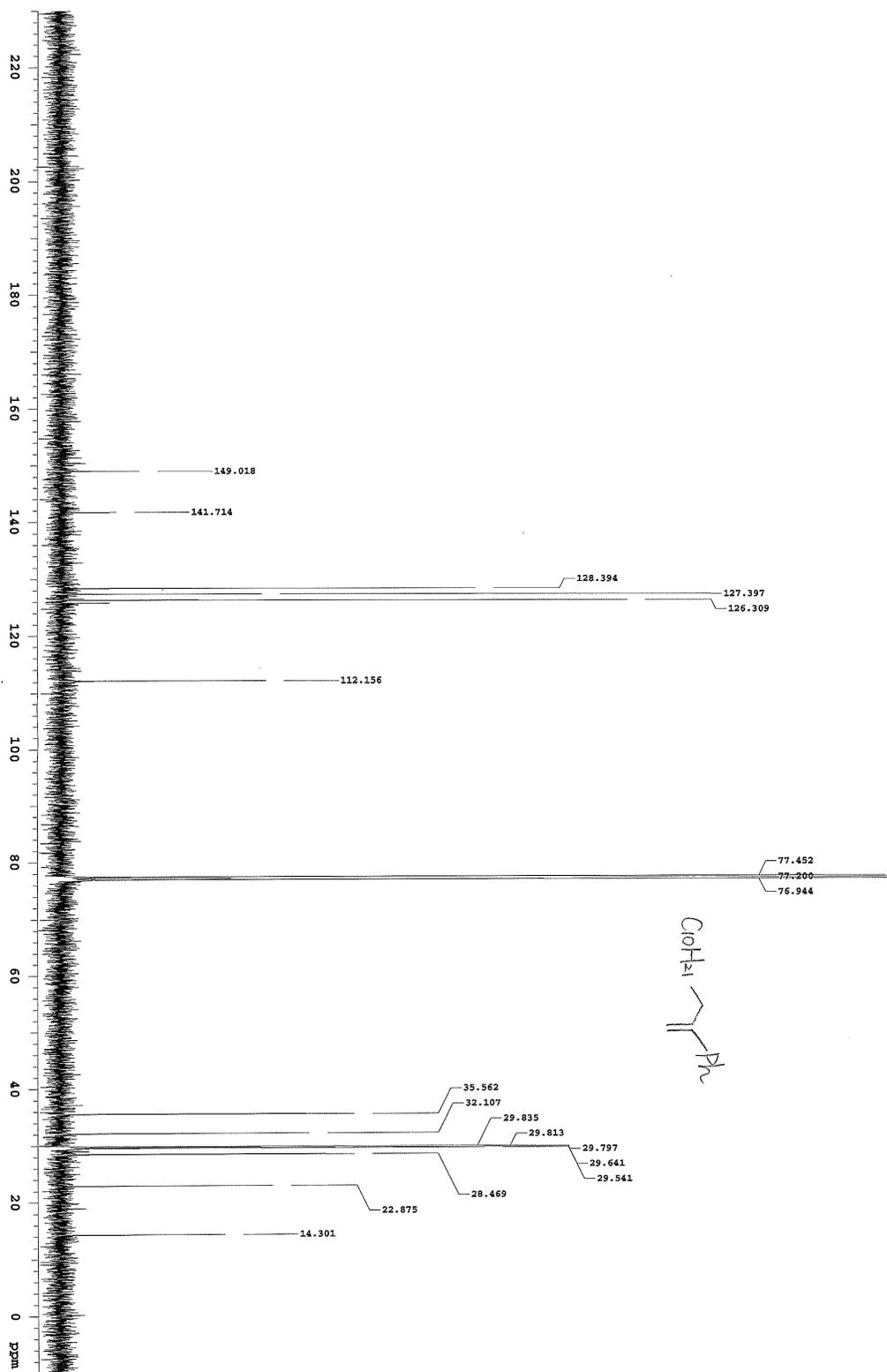


Figure S13. ¹H NMR spectrum of **3b**

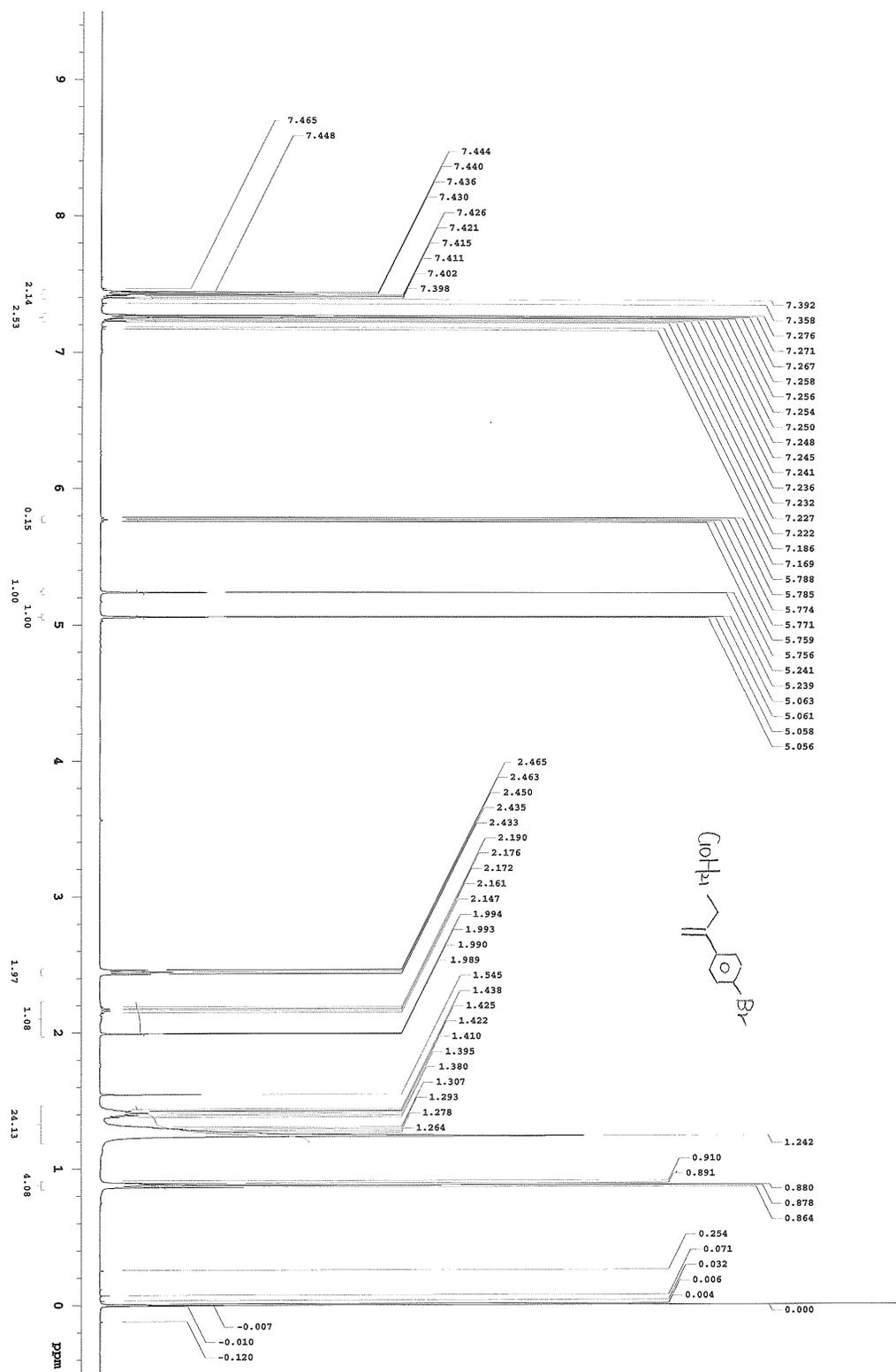


Figure S14. ^{13}C NMR spectrum of **3b**

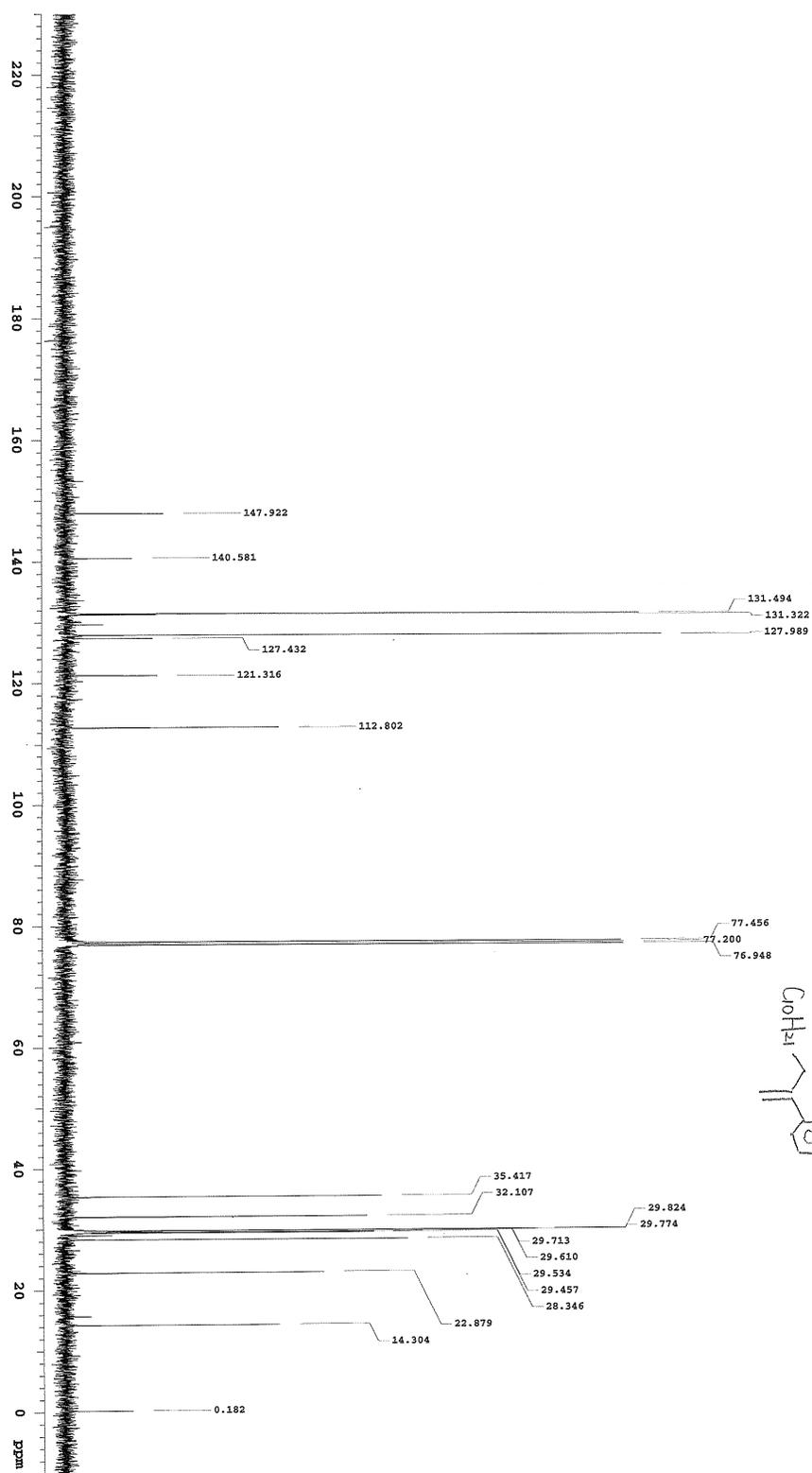


Figure S15. ^1H NMR spectrum of **3c**

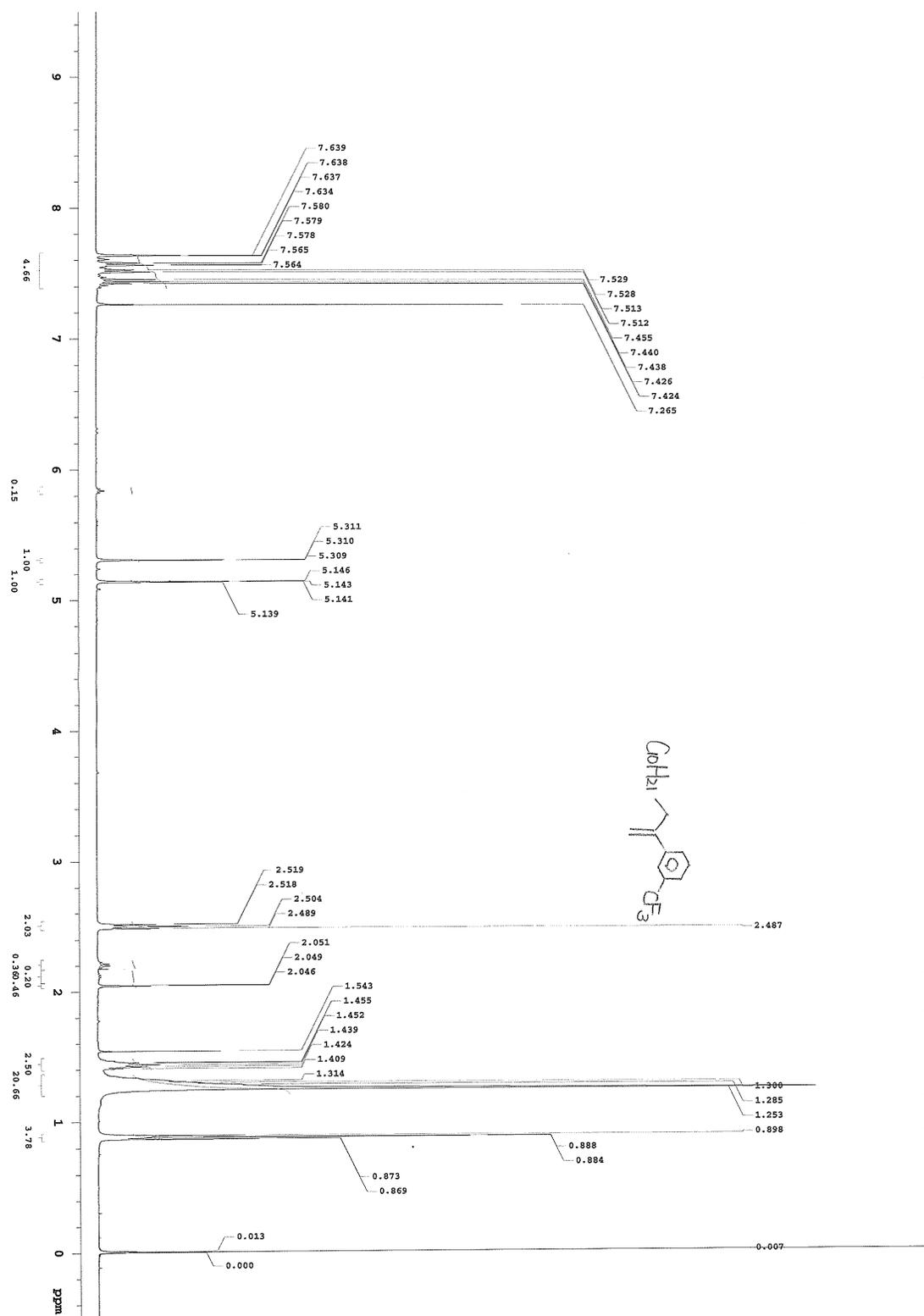


Figure S16. ^{13}C NMR spectrum of **3c**

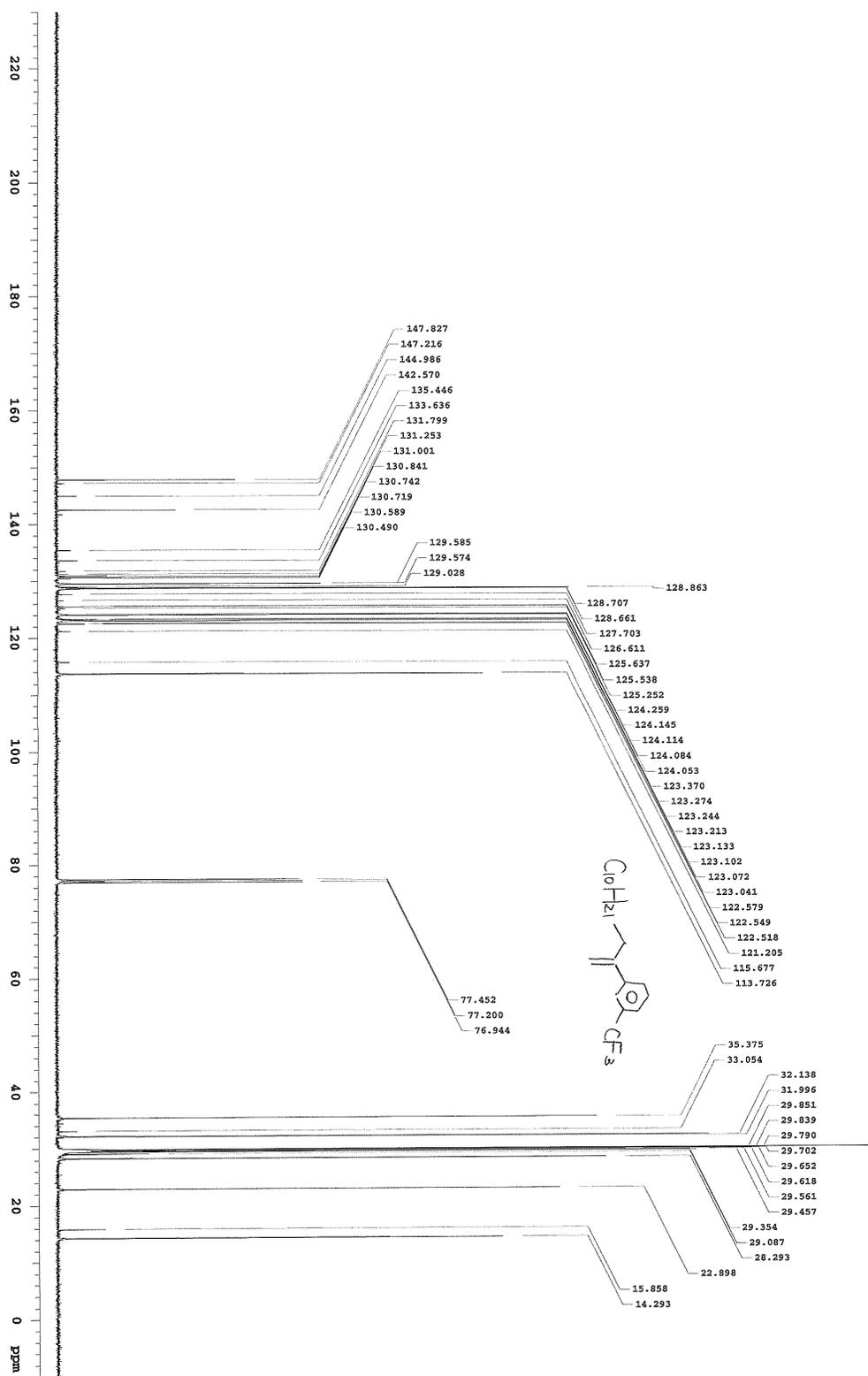


Figure S17. ^1H NMR spectrum of **3d**

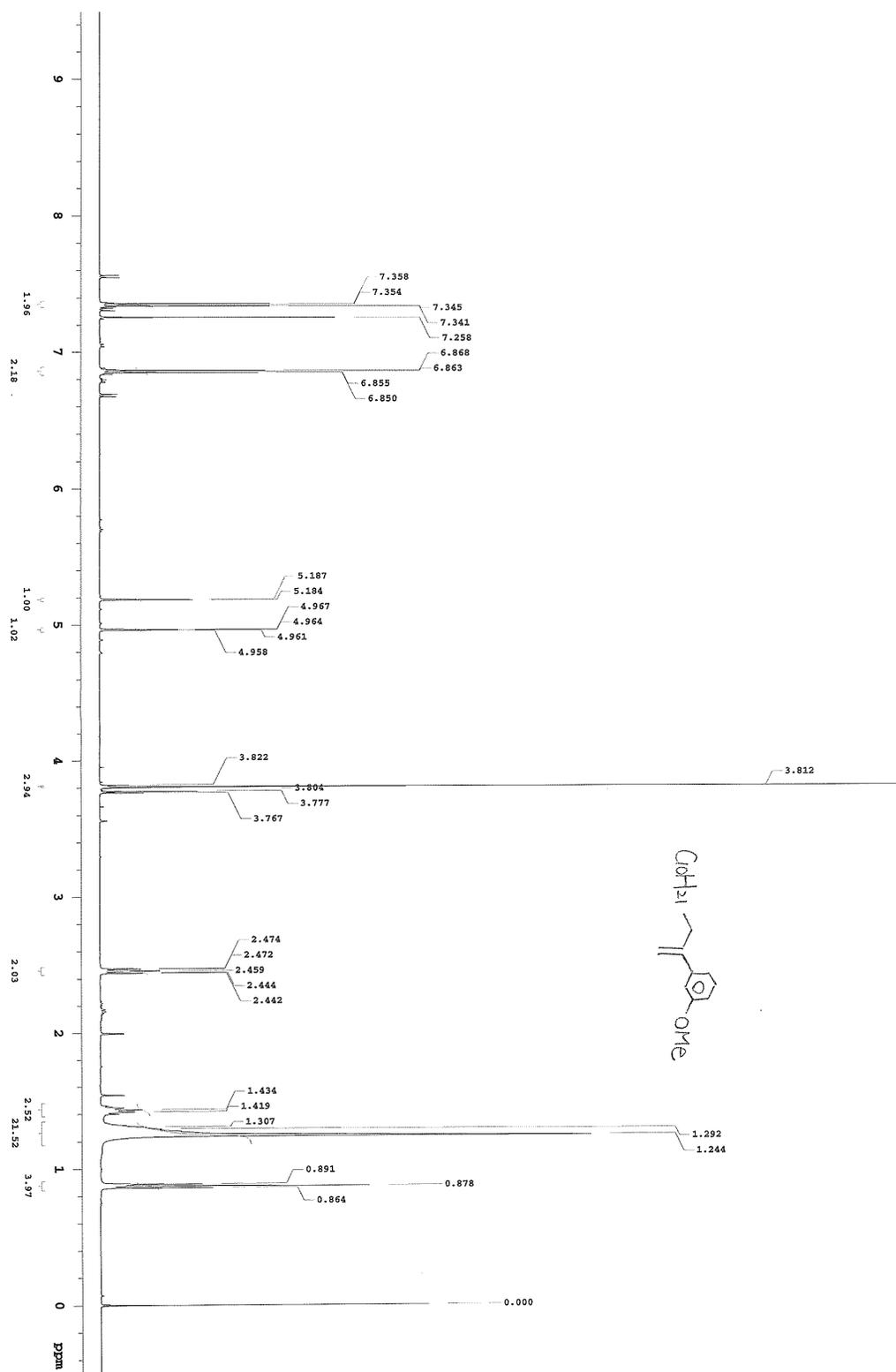


Figure S18. ^{13}C NMR spectrum of **3d**

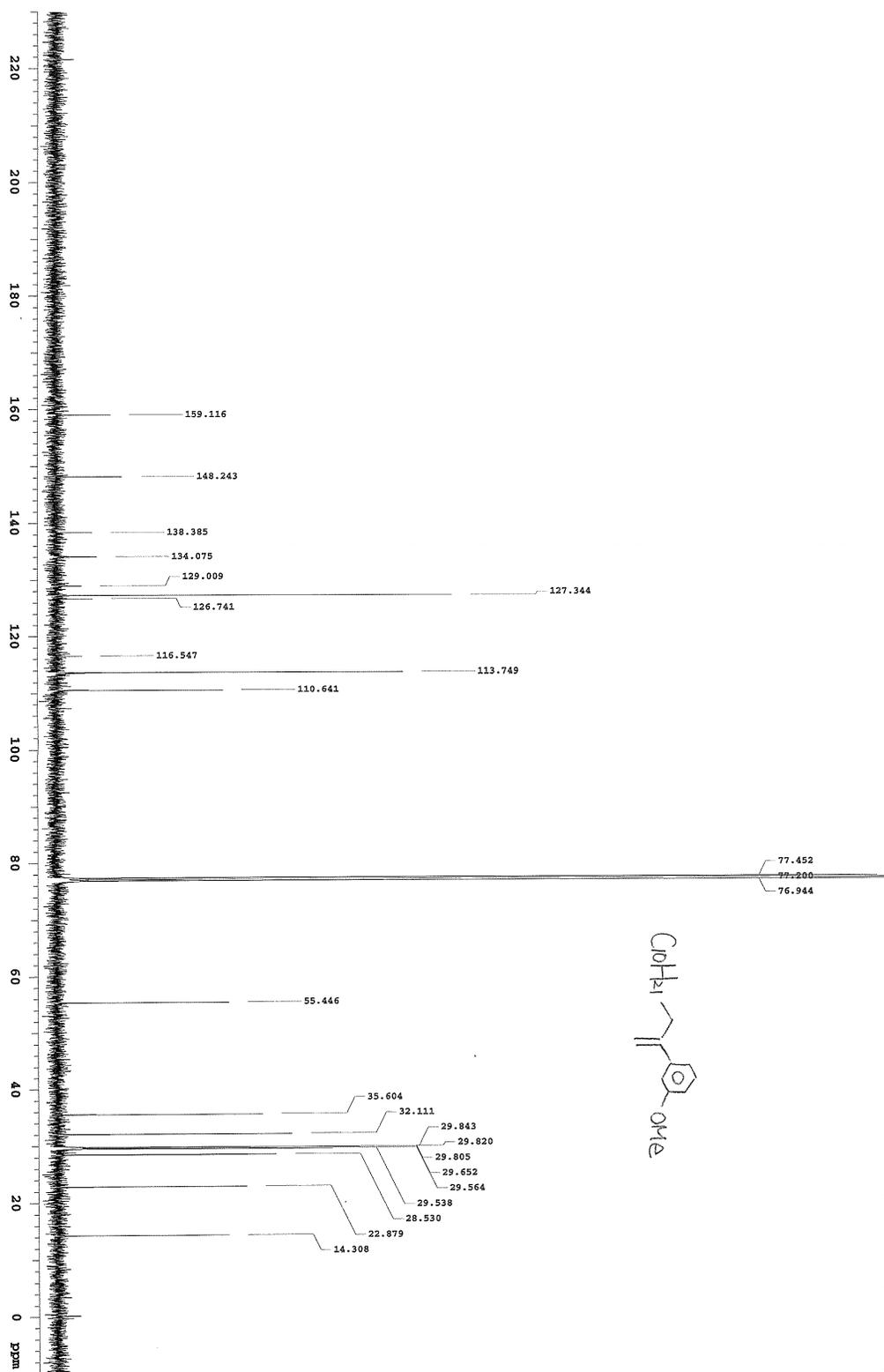


Figure S19. ^1H NMR spectrum of **3f**

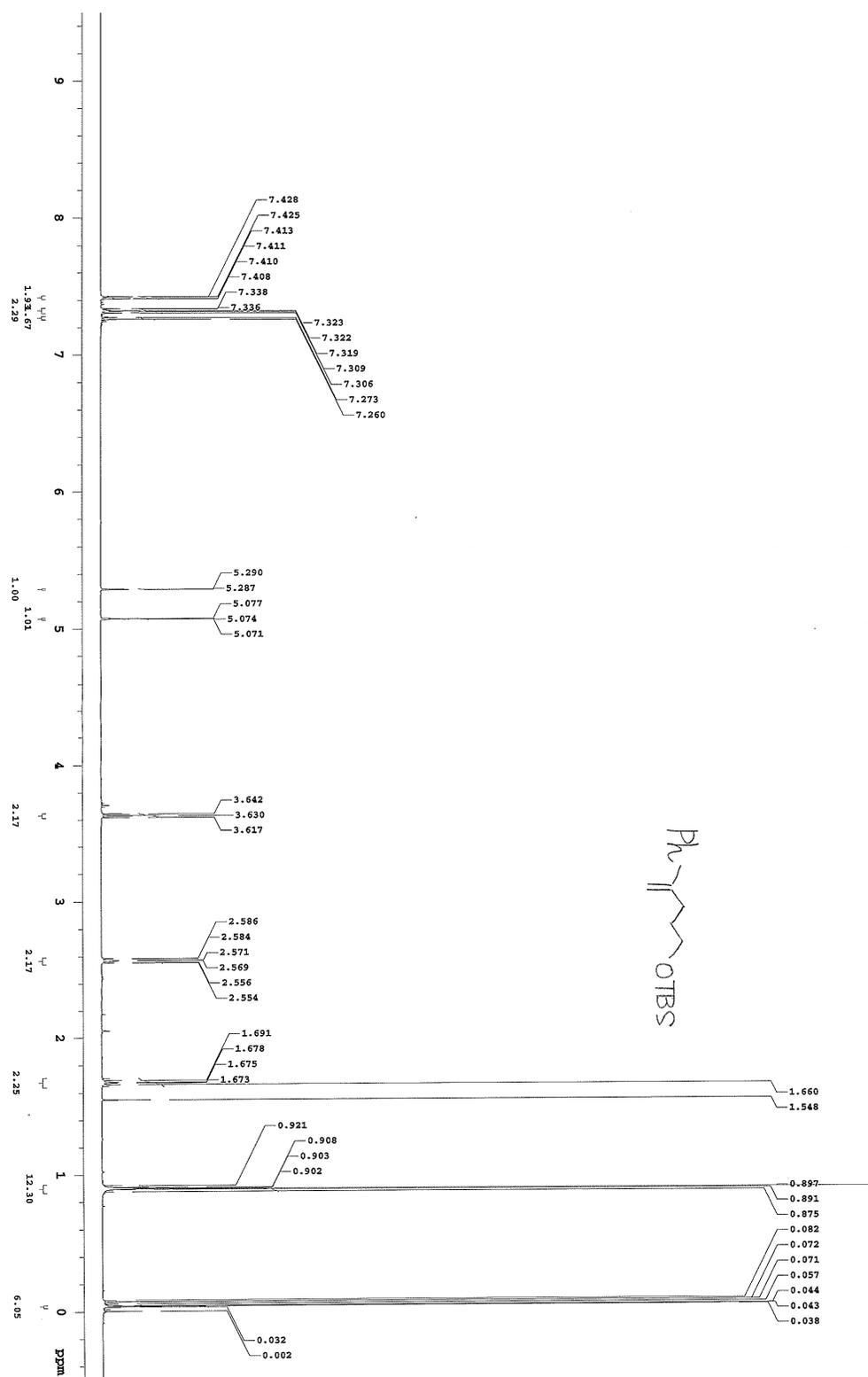


Figure S20. ^{13}C NMR spectrum of **3f**

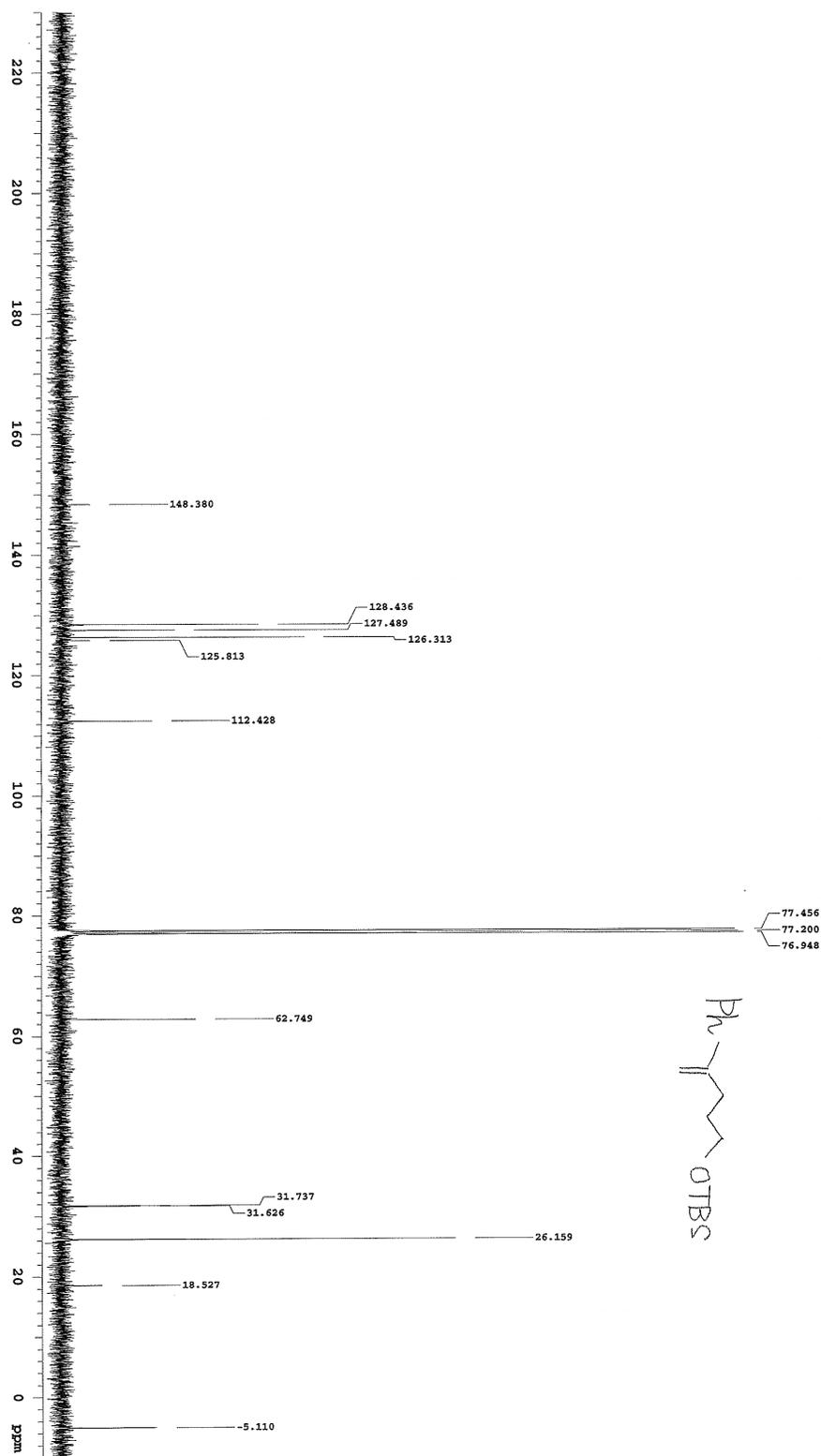


Figure S21. ¹H NMR spectrum of **3g**

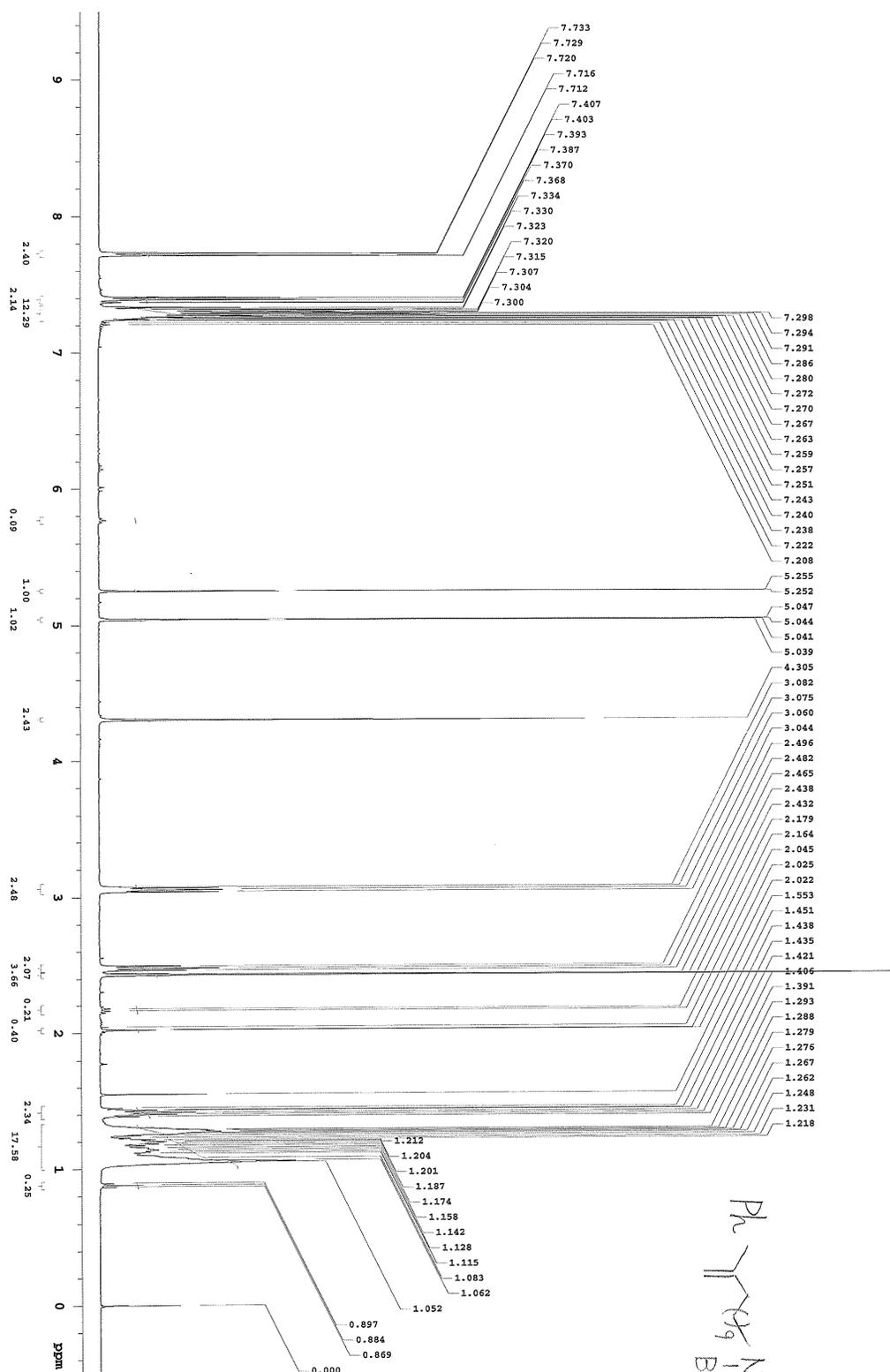


Figure S22. ^{13}C NMR spectrum of **3g**

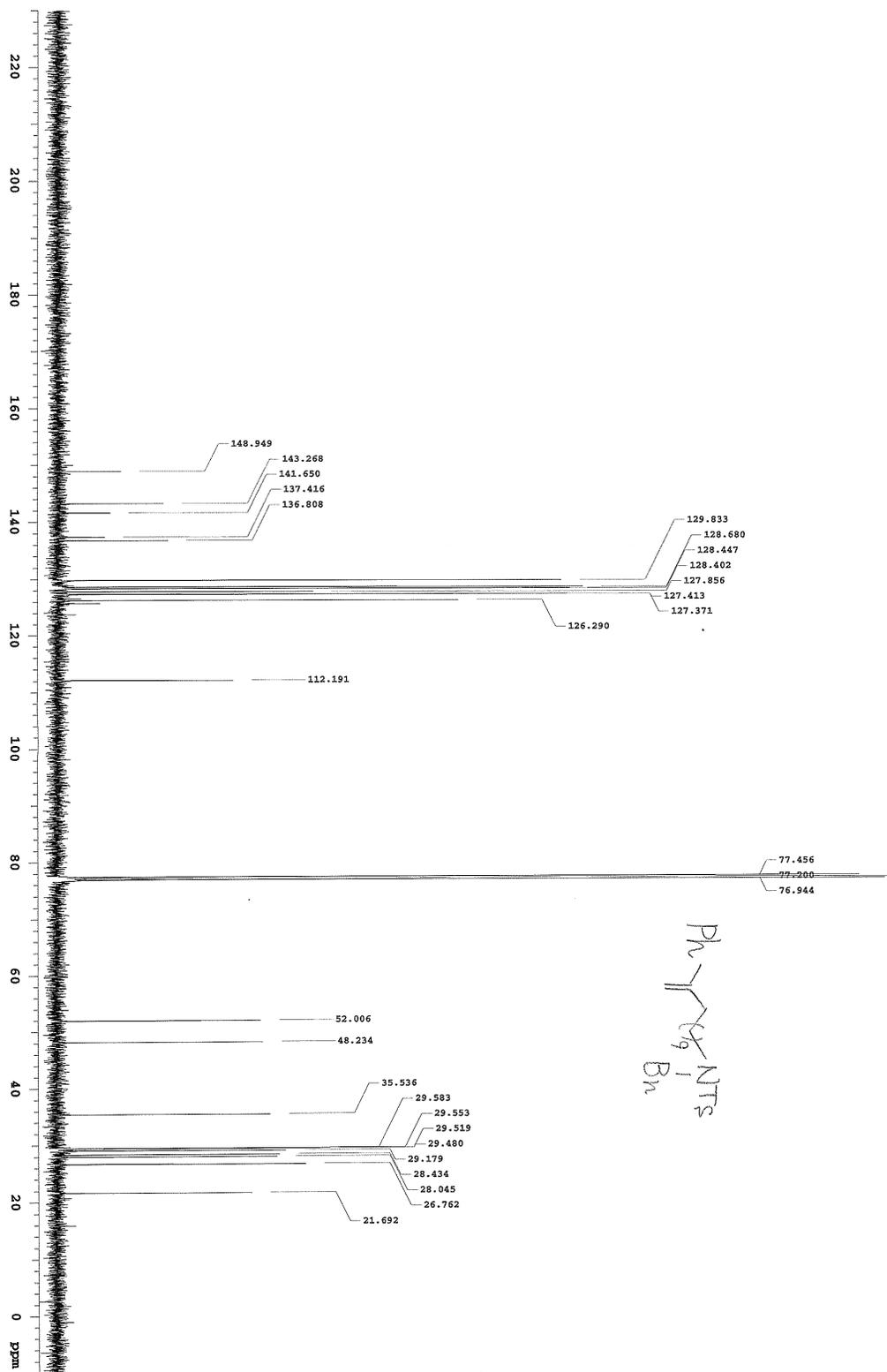


Figure S23. ¹H NMR spectrum of **3h**

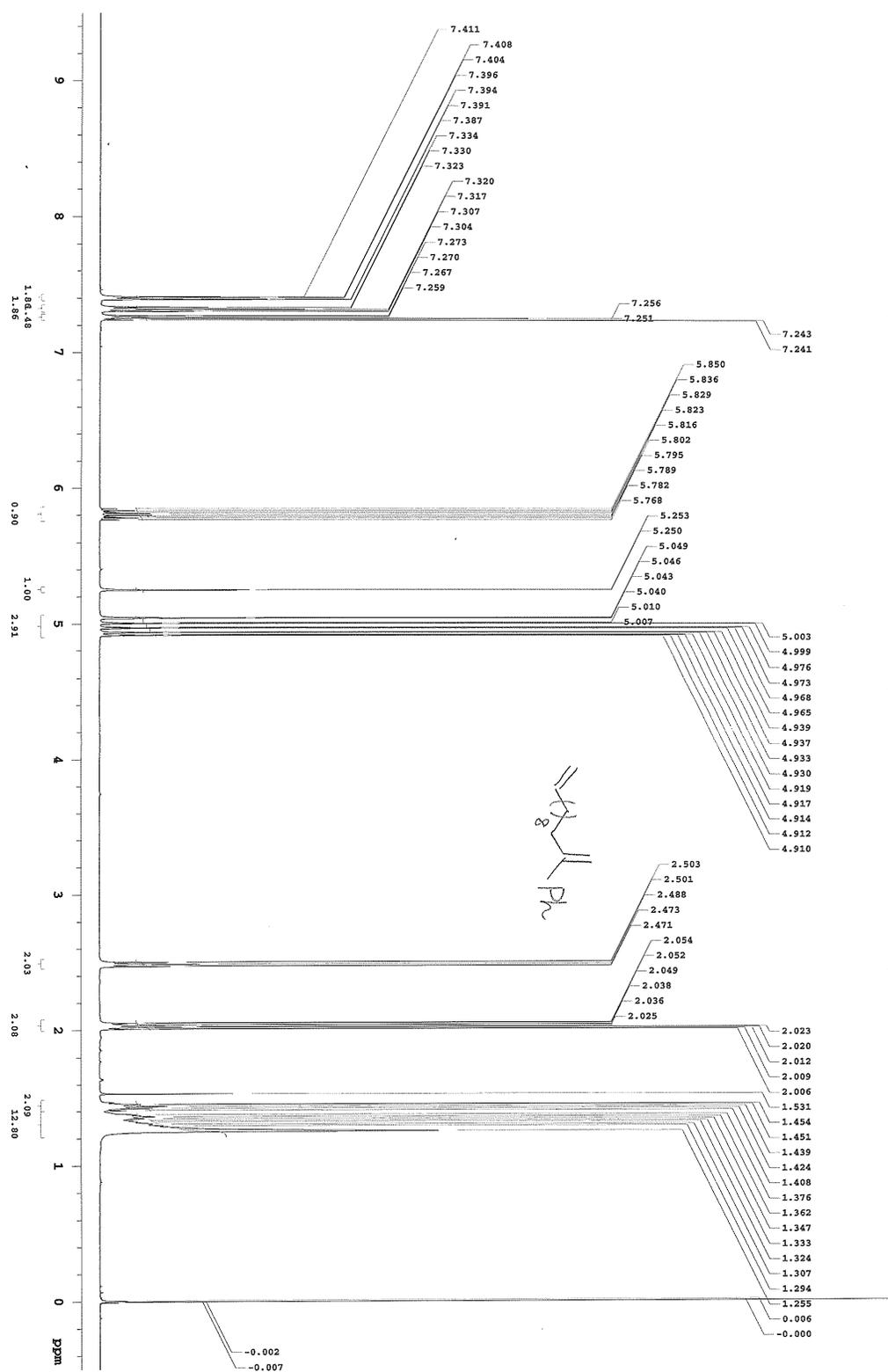


Figure S24. ^{13}C NMR spectrum of **3h**

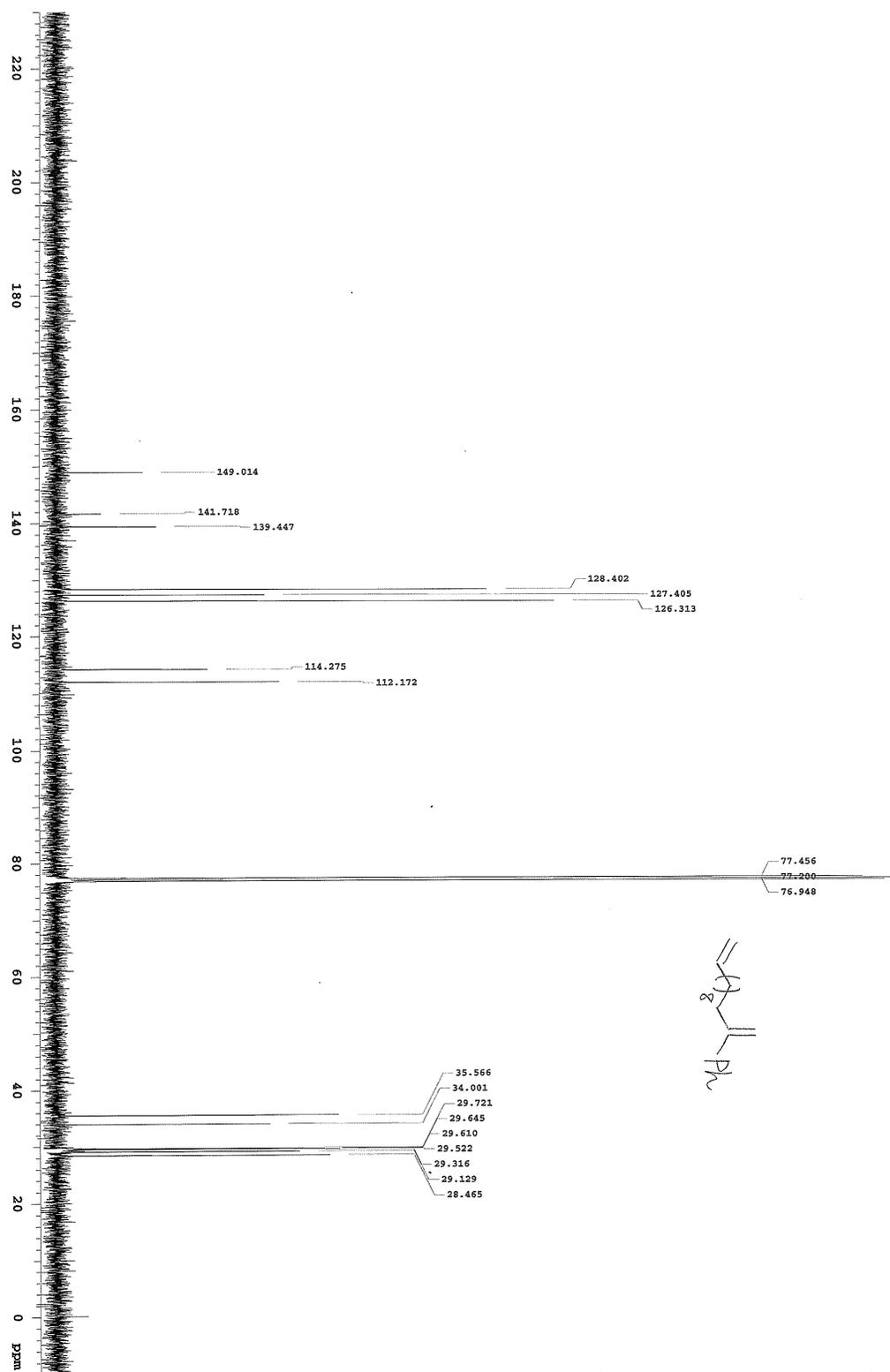


Figure S25. ¹H NMR spectrum of **3i**

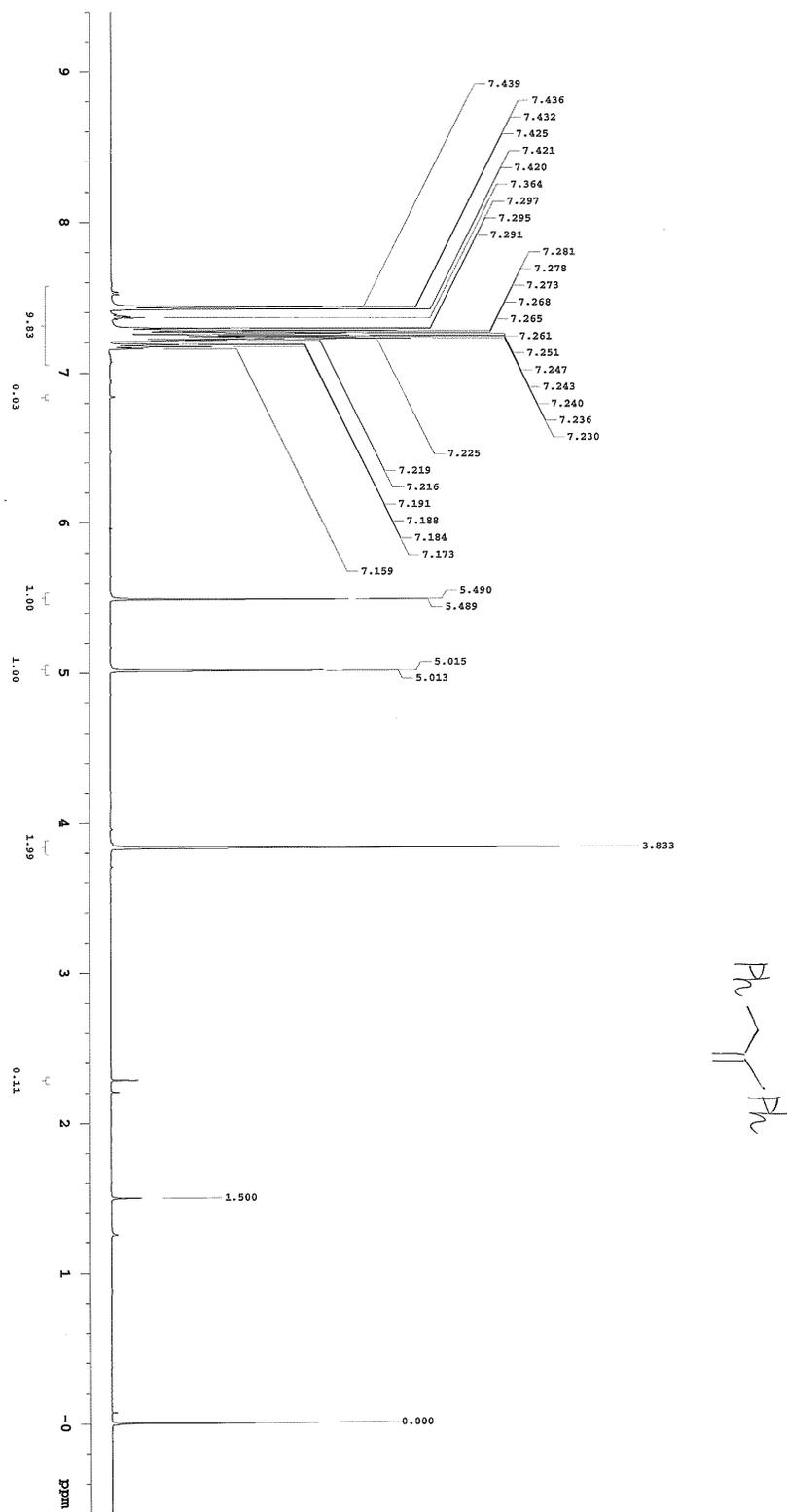


Figure S26. ^{13}C NMR spectrum of **3i**

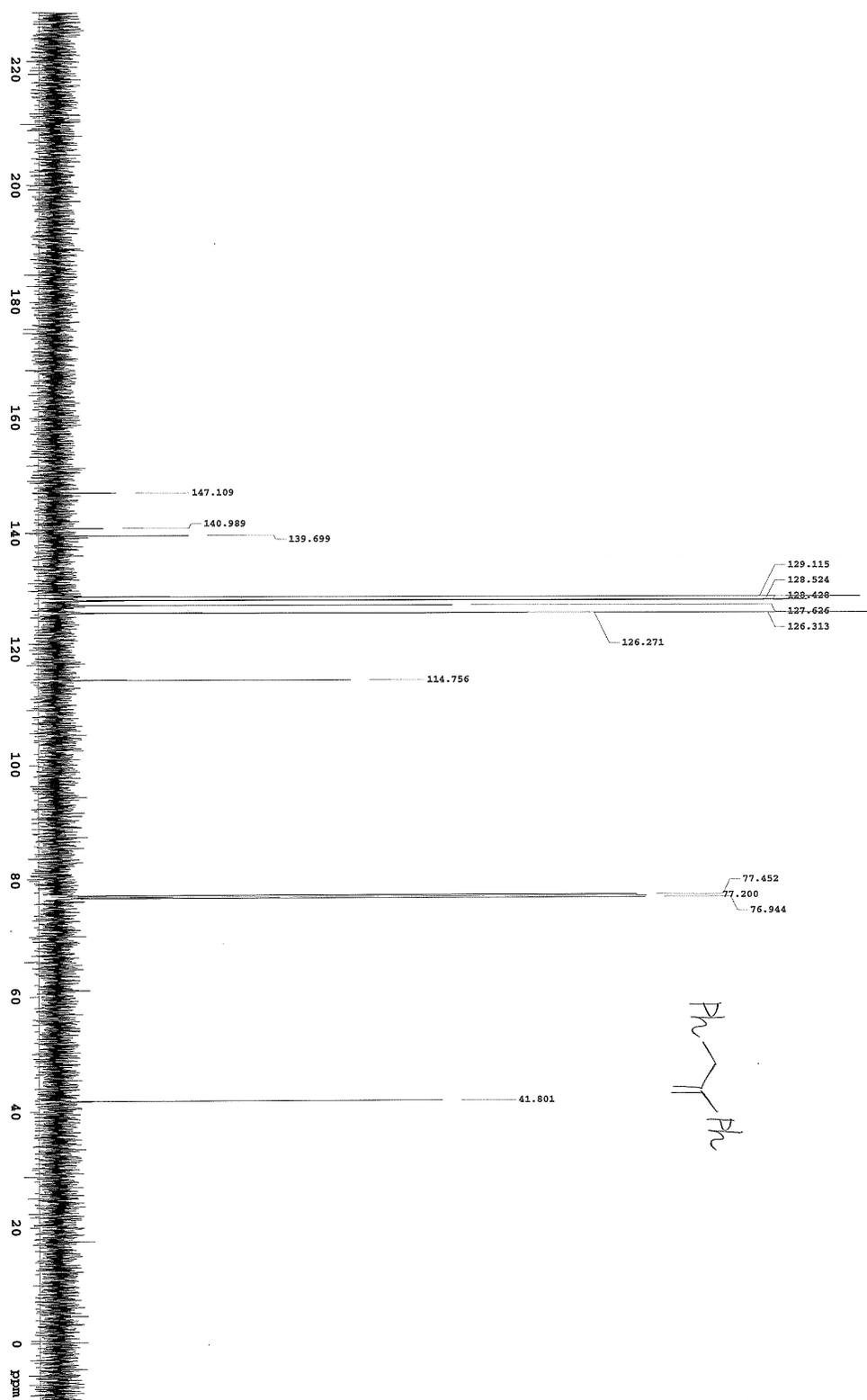


Figure S27. ^1H NMR spectrum of **4a**

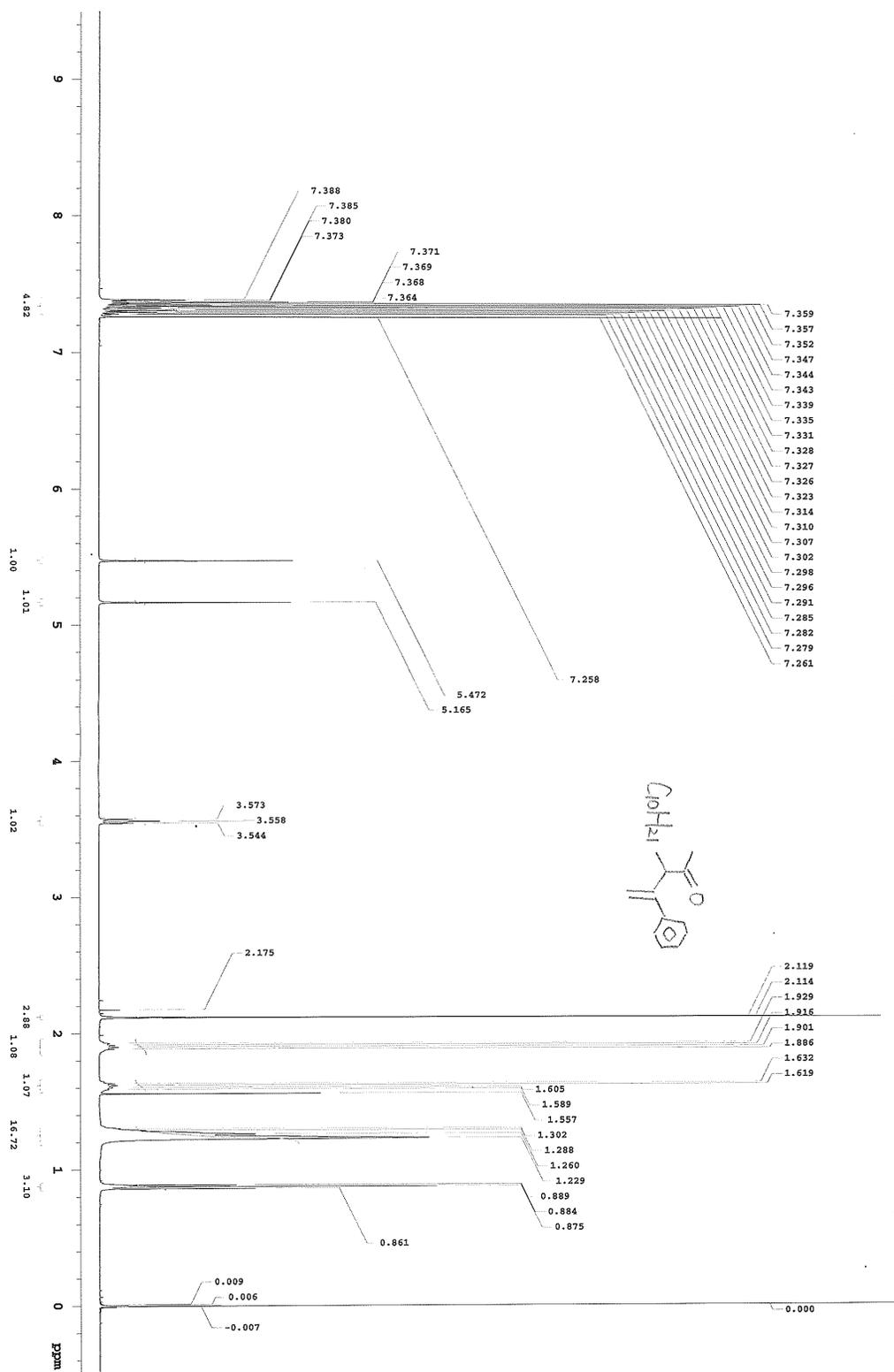


Figure S28. ^{13}C NMR spectrum of **4a**

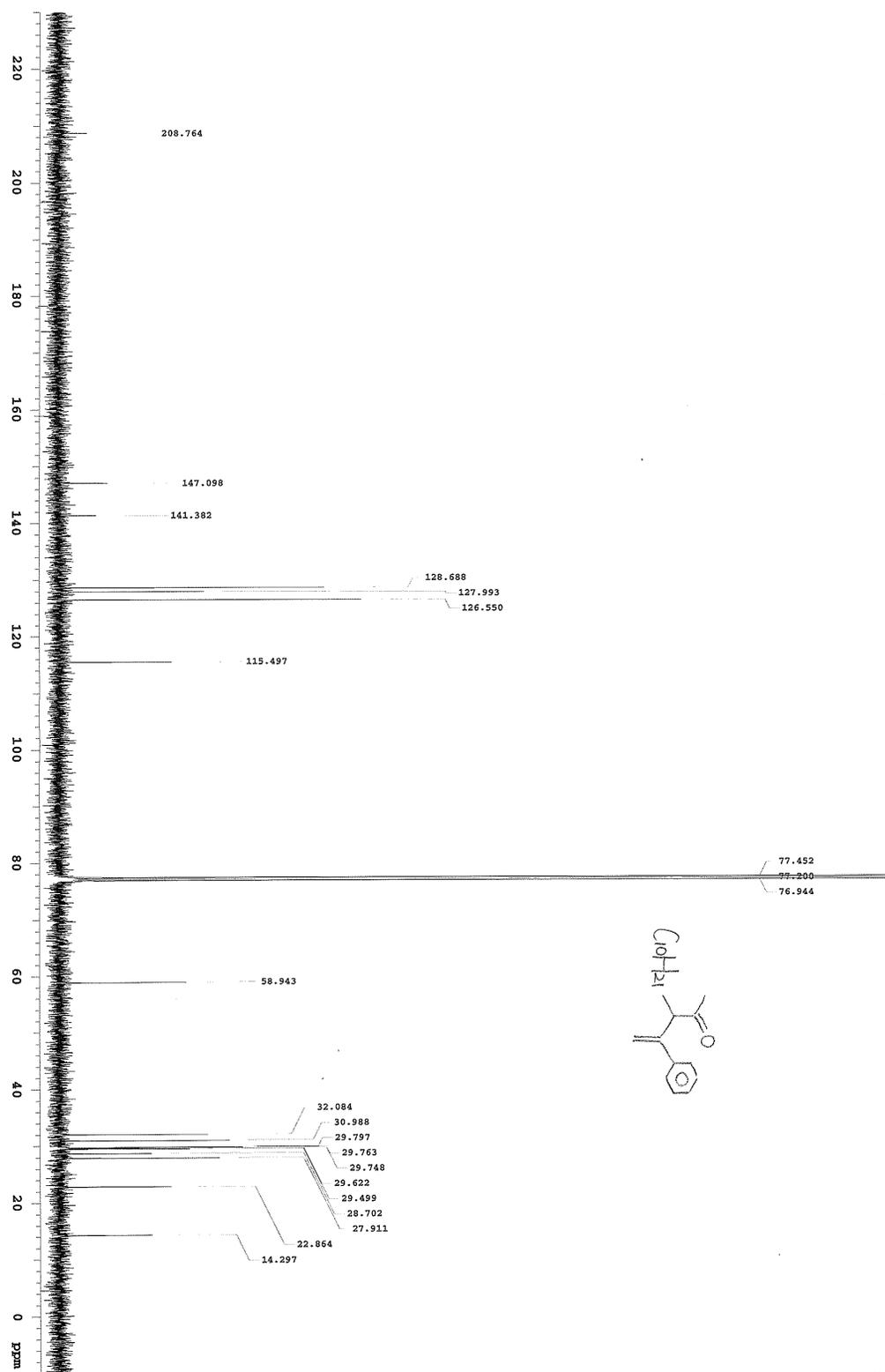


Figure S29. ¹H NMR spectrum of **4b**

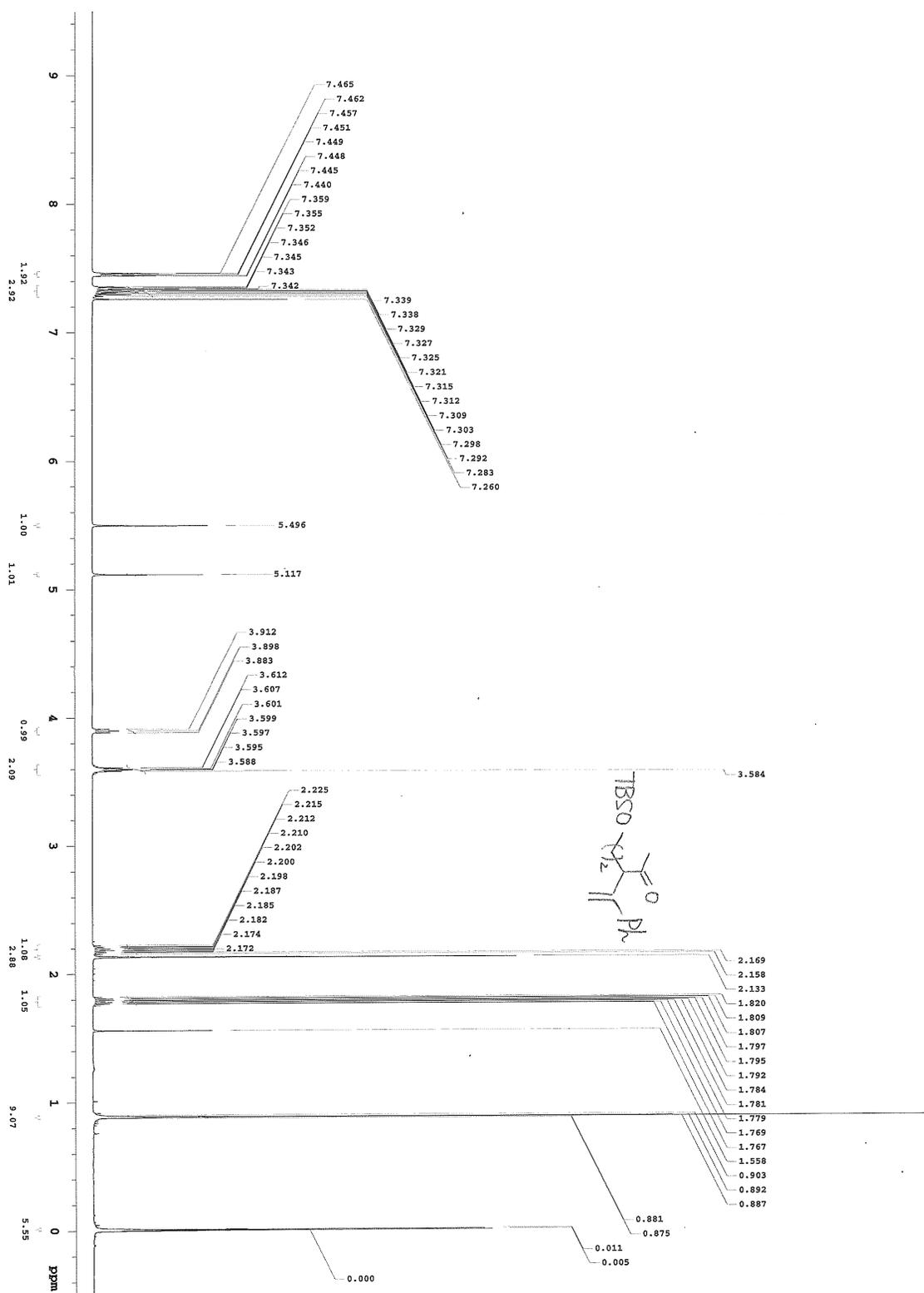


Figure S30. ^{13}C NMR spectrum of **4b**

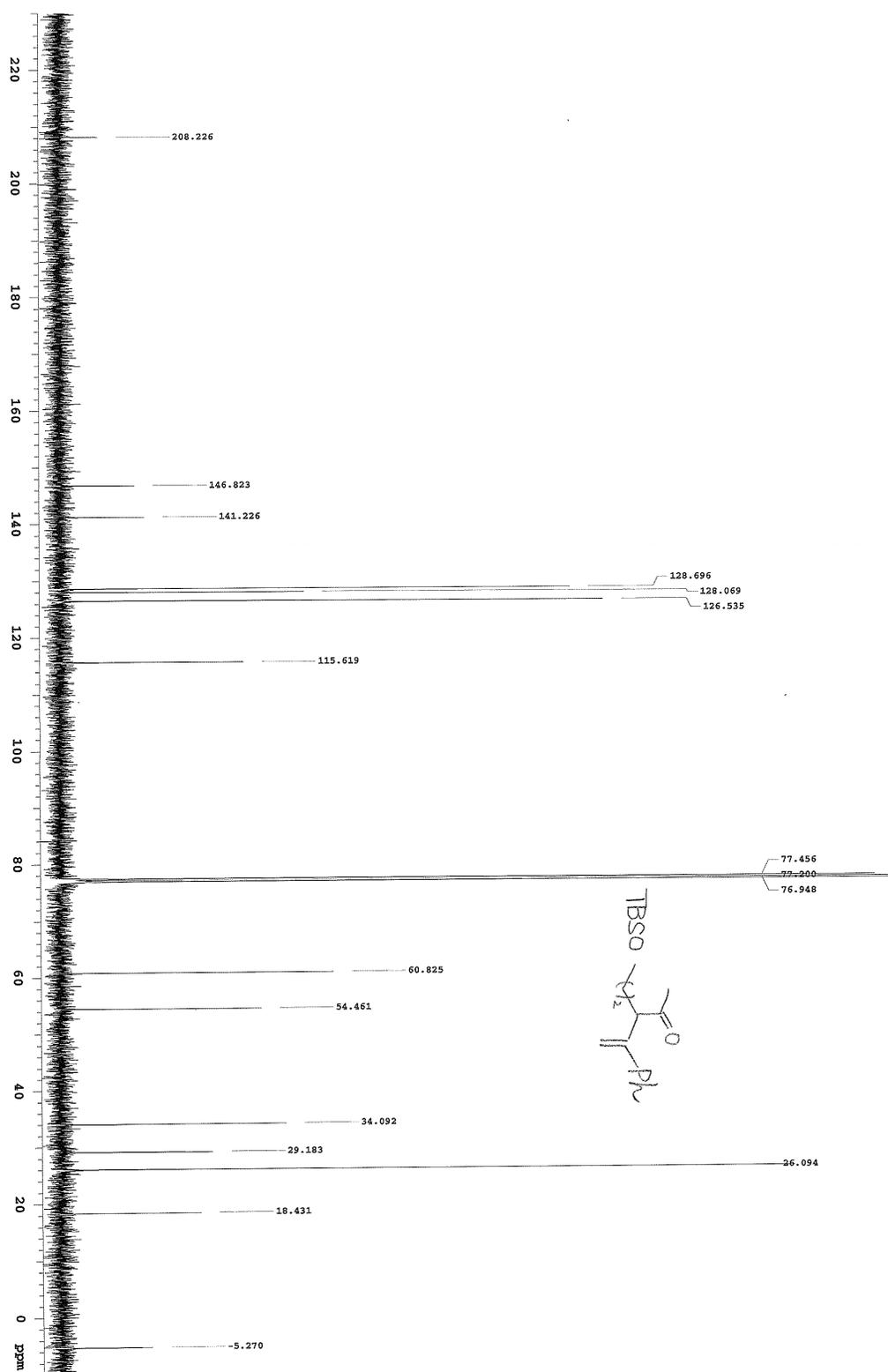


Figure S31. ^1H NMR spectrum of **4c**

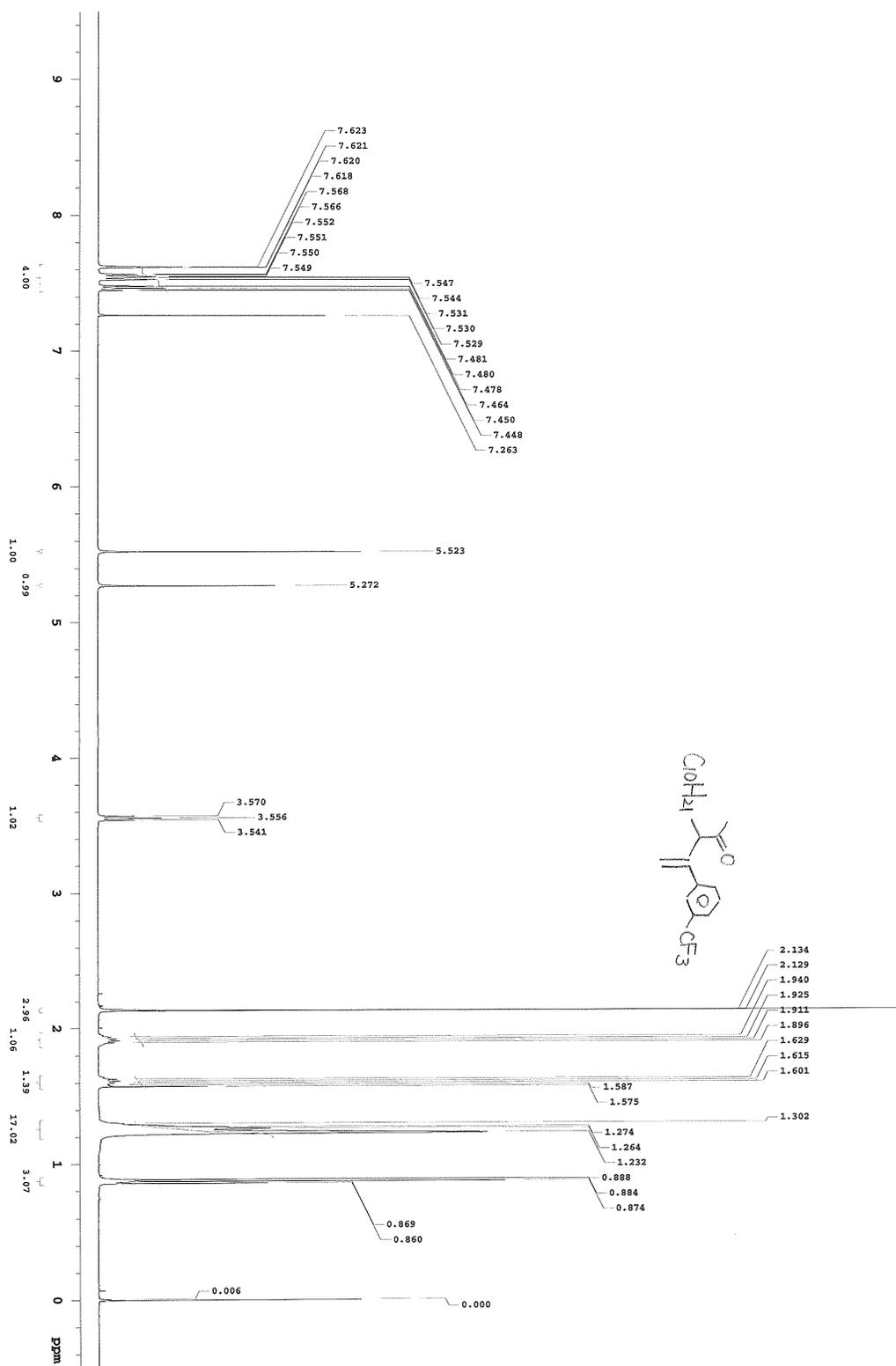


Figure S32. ^{13}C NMR spectrum of **4c**

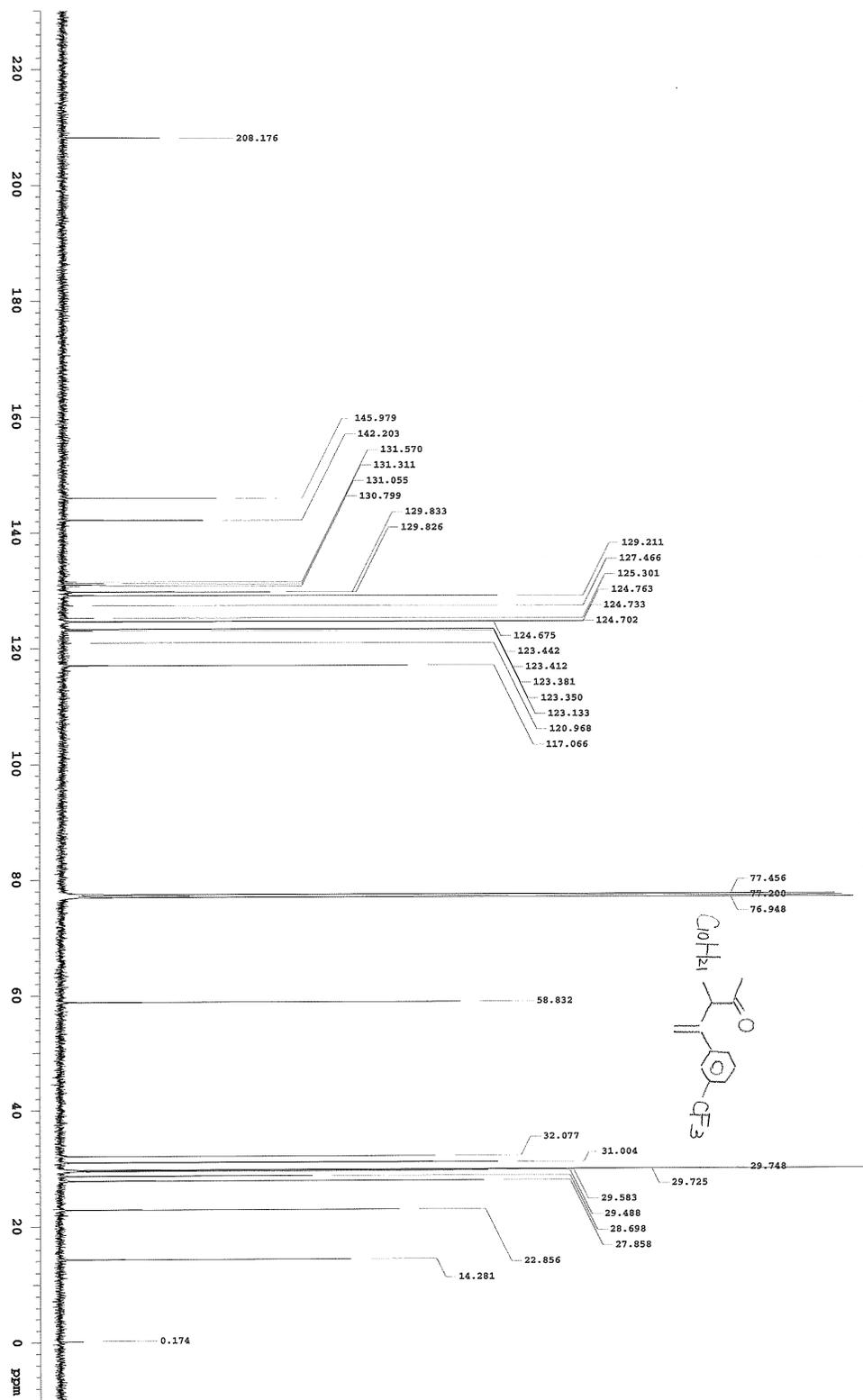


Figure S33. ¹H NMR spectrum of **4d**

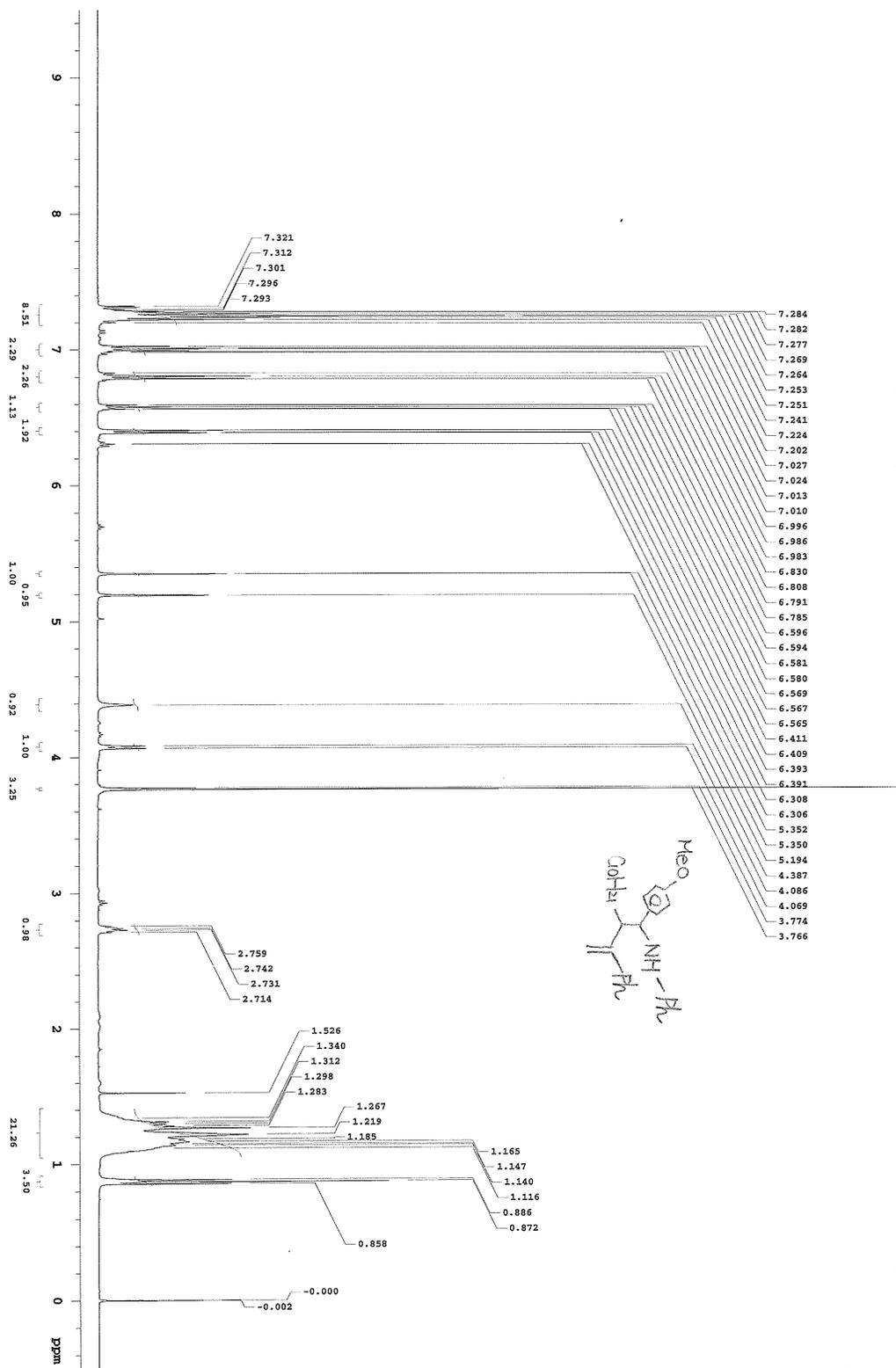


Figure S34. ^{13}C NMR spectrum of **4d**

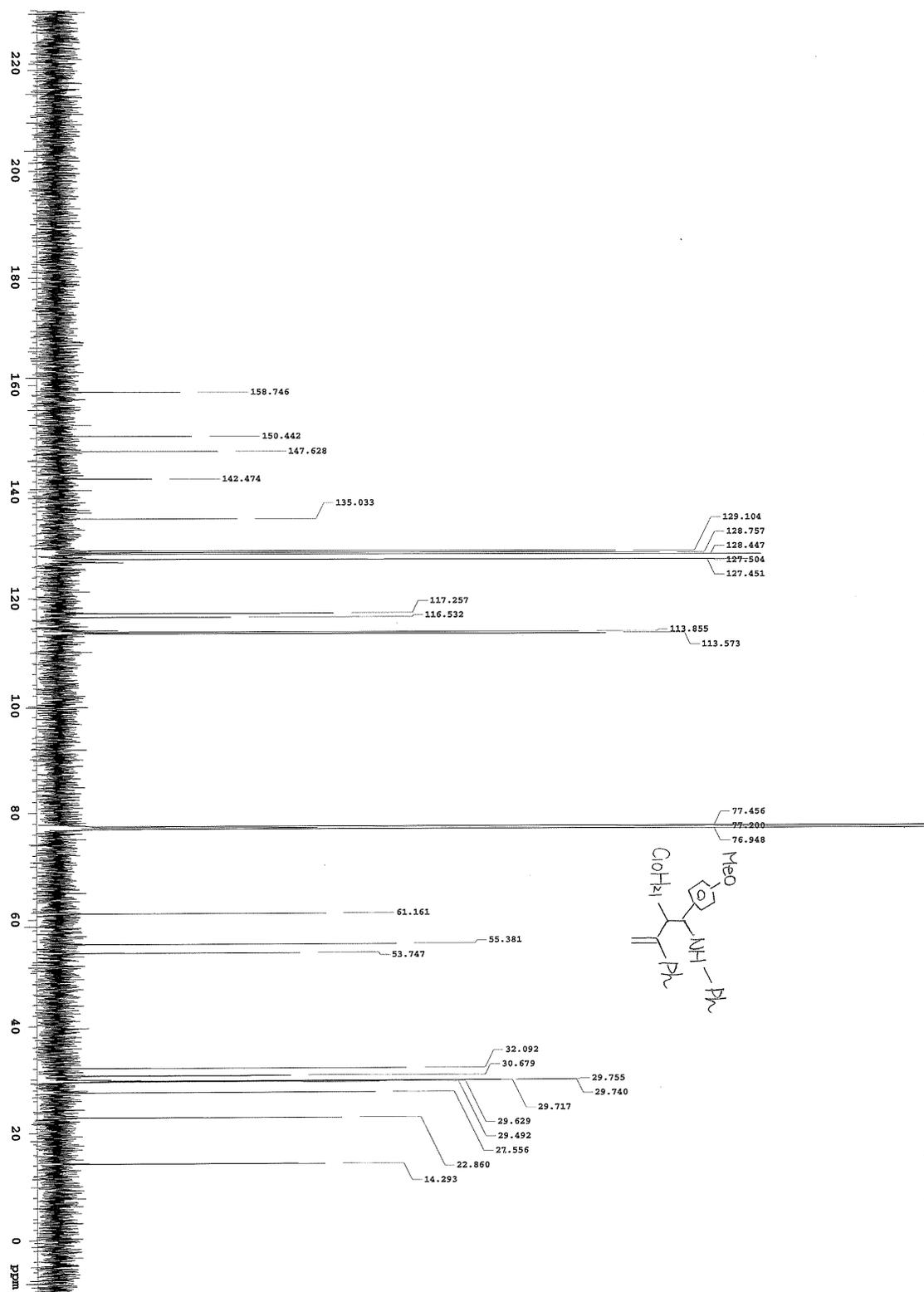


Figure S35. ¹H NMR spectrum of **4e**

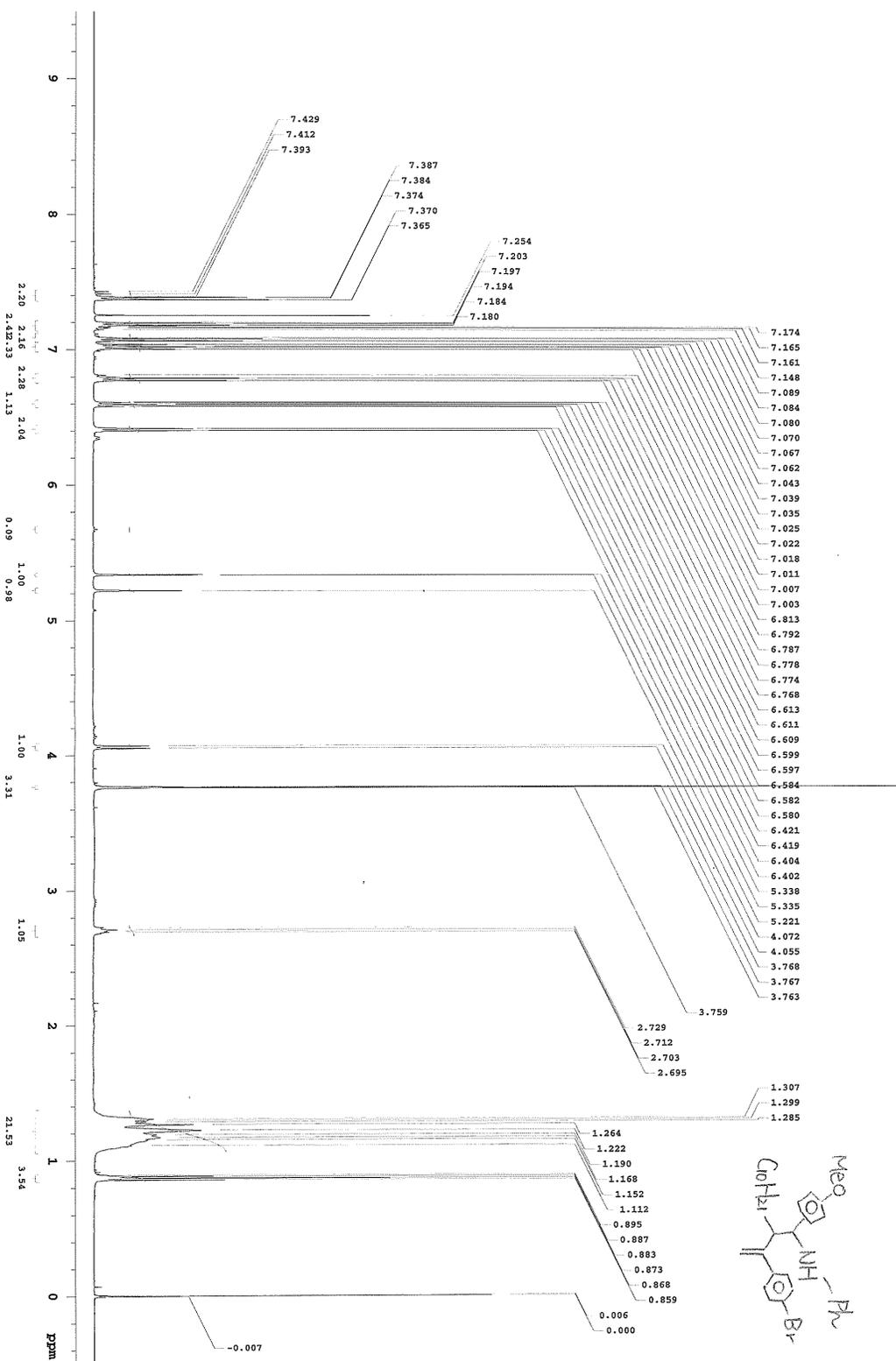


Figure S36. ^{13}C NMR spectrum of **4e**

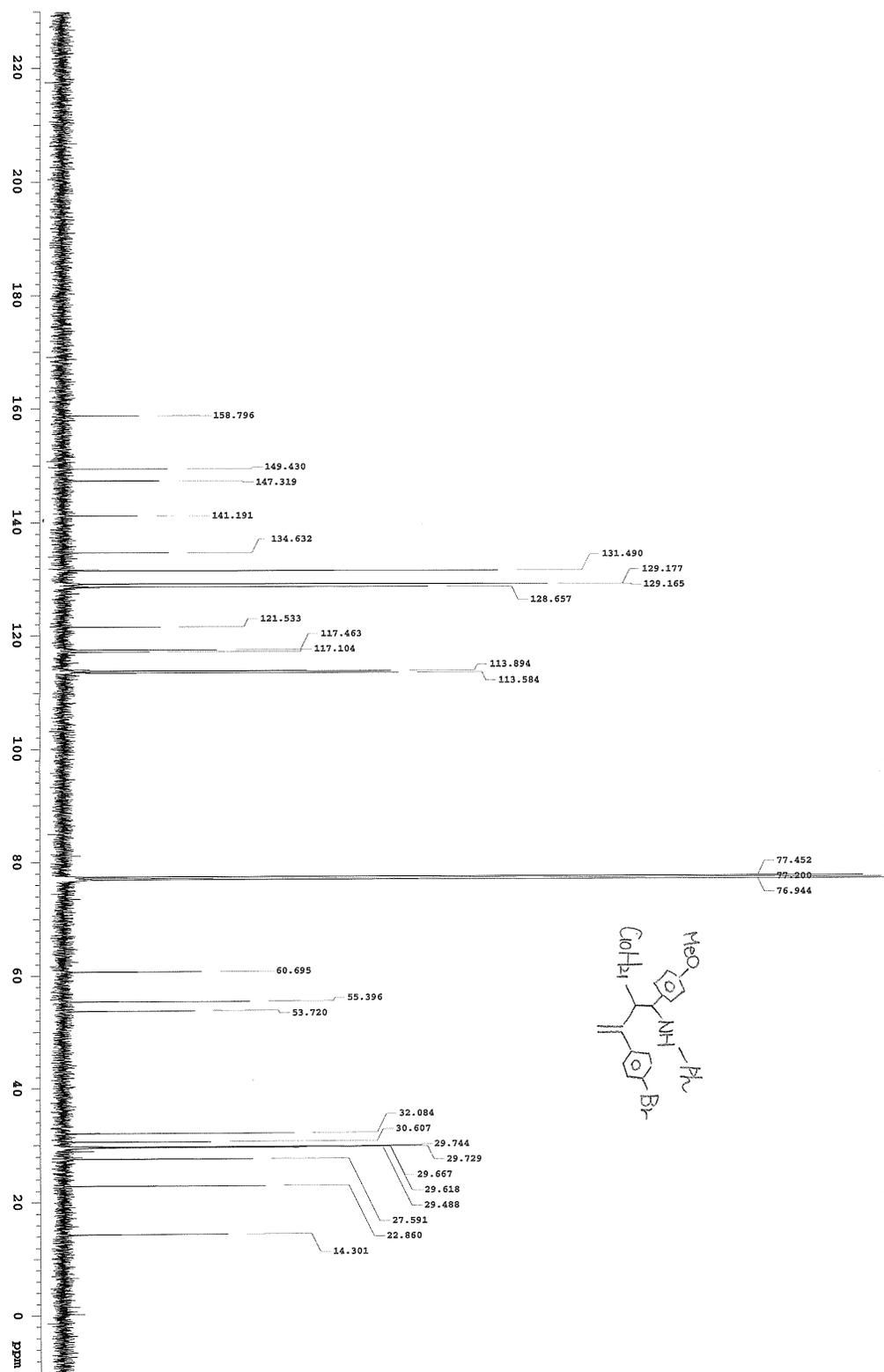


Figure S37. ¹H NMR spectrum of **4f**

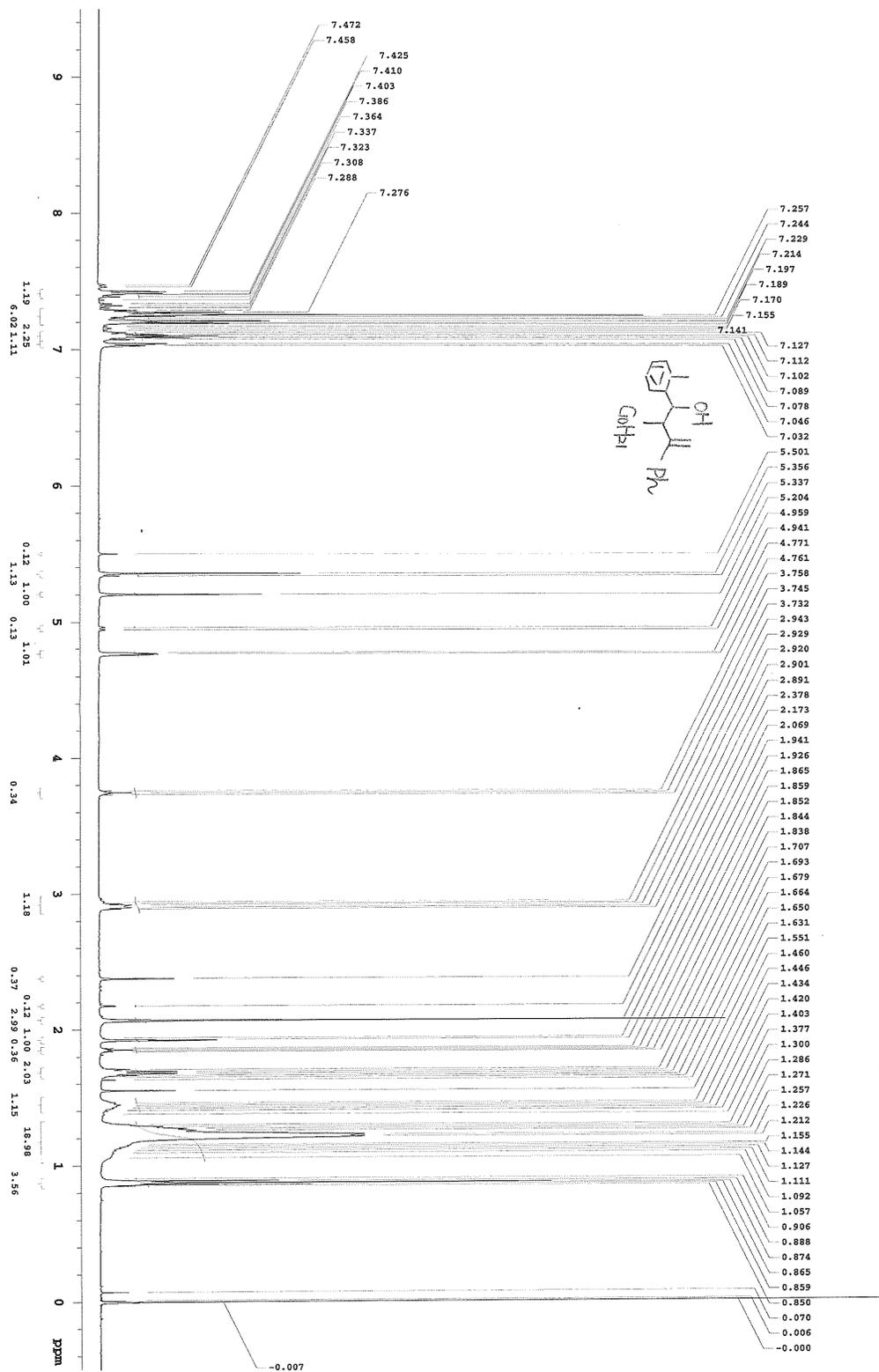


Figure S38. ^{13}C NMR spectrum of **4f**

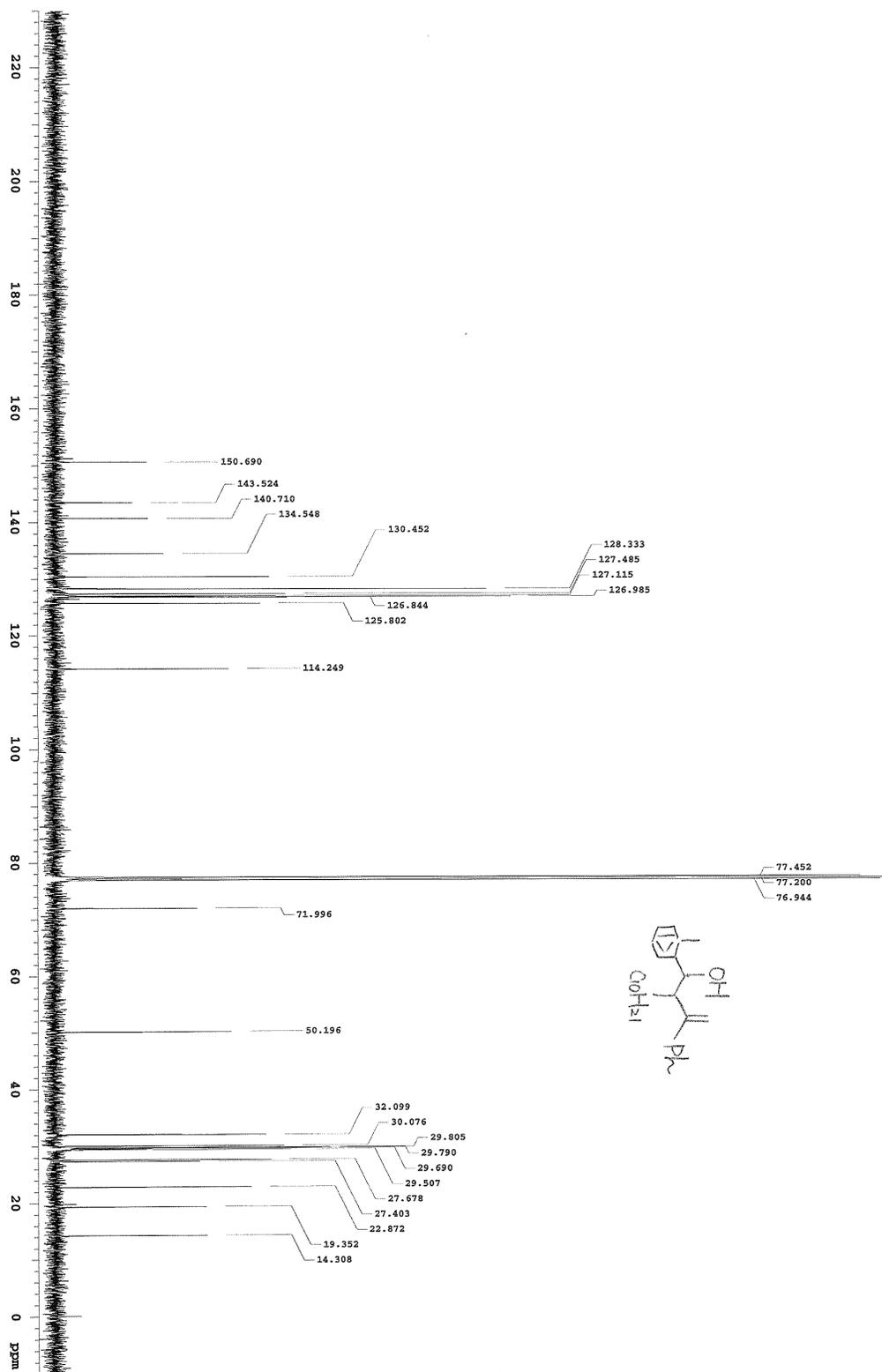


Figure S39. ^1H NMR spectrum of **4g**

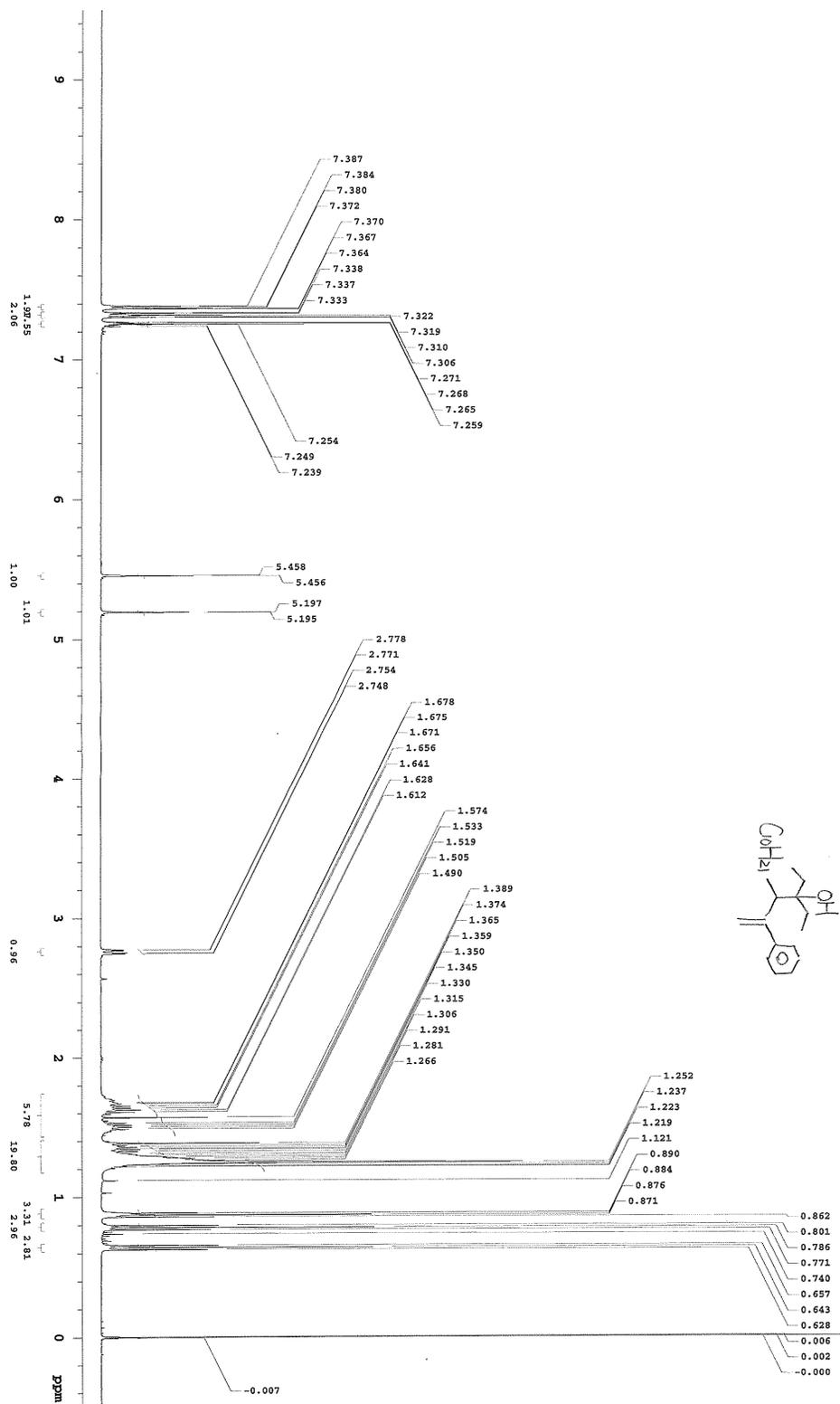


Figure S40. ^{13}C NMR spectrum of **4g**

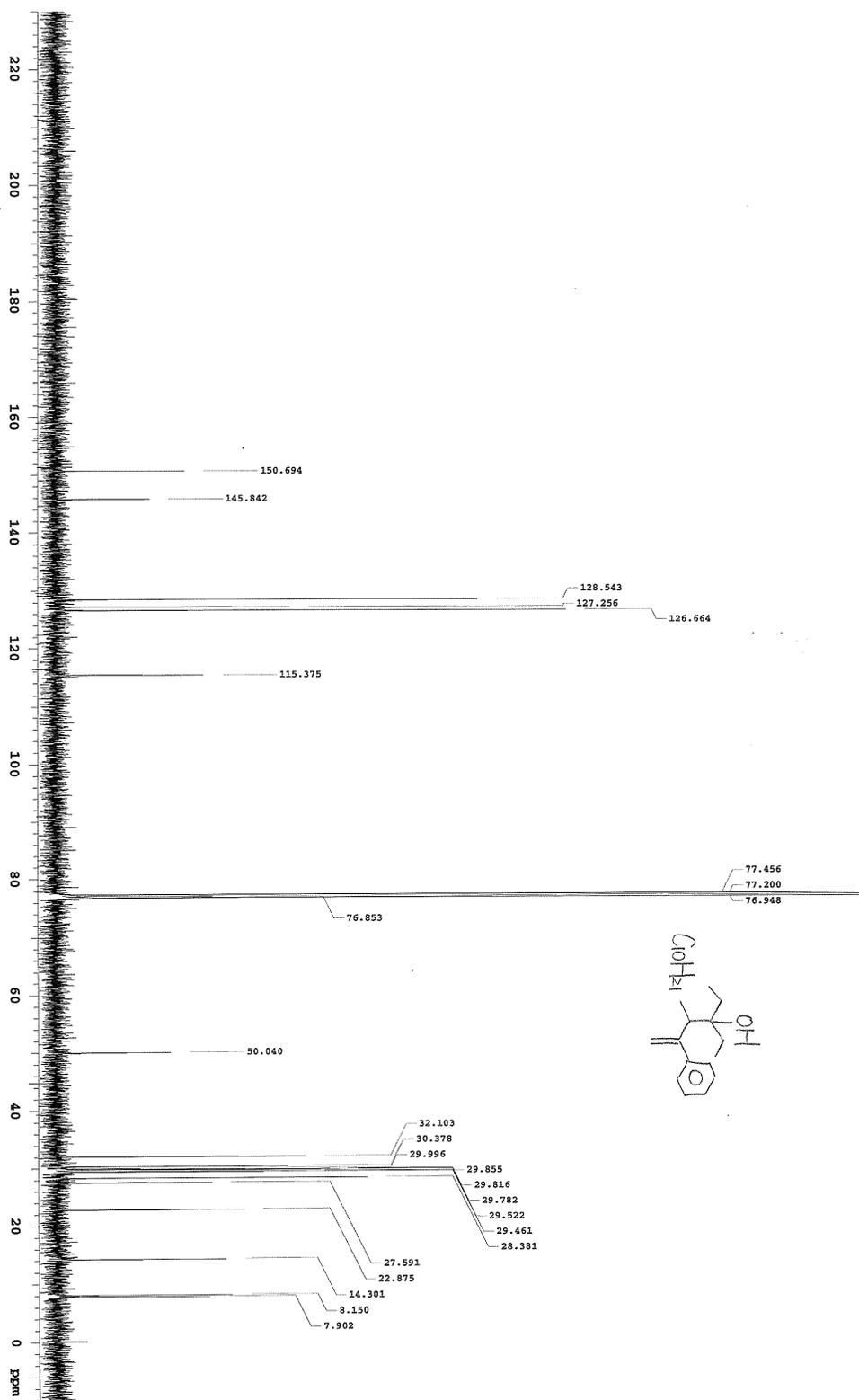


Figure S41. ^1H NMR spectrum of **4h**

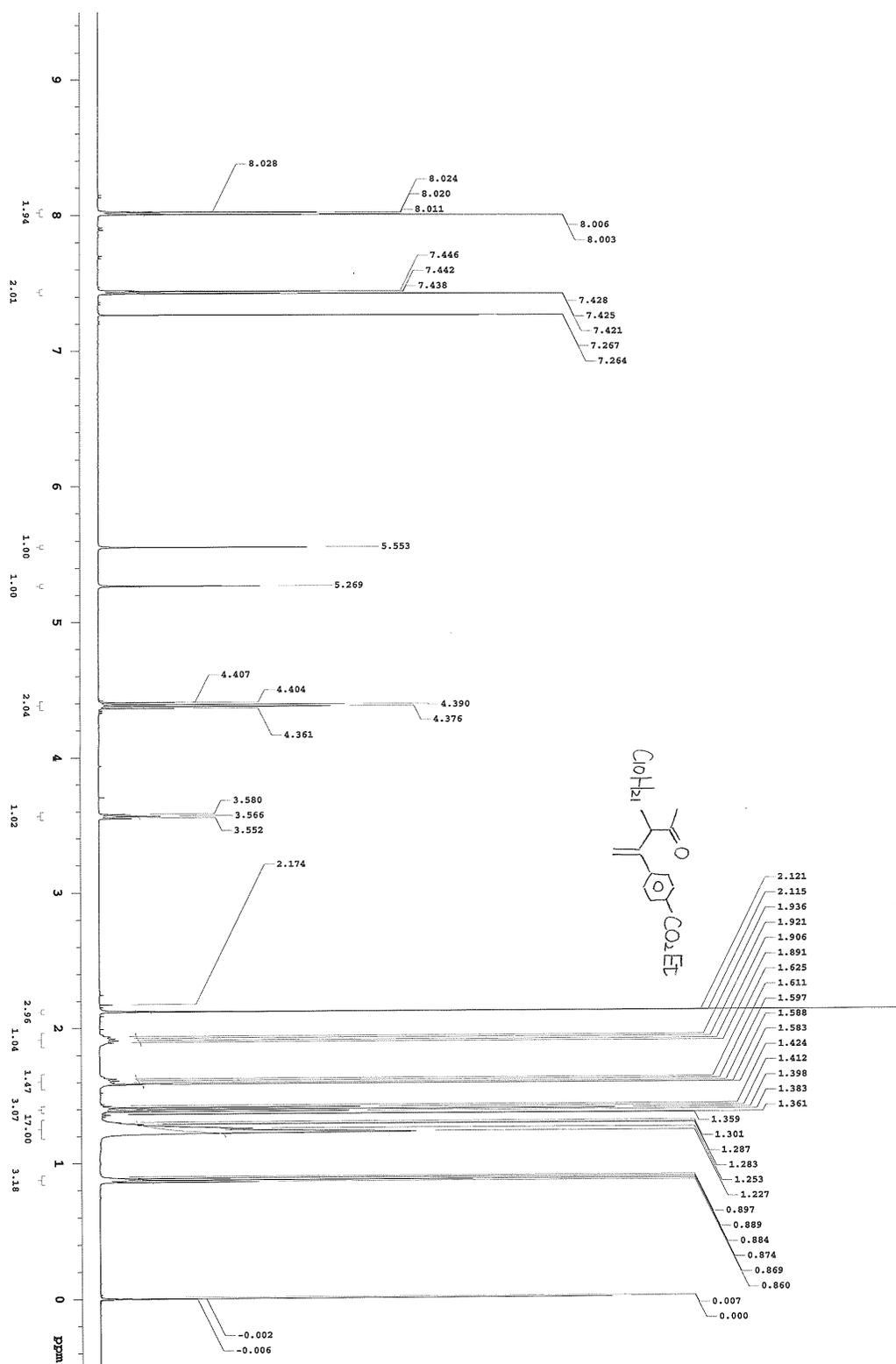


Figure S42. ^{13}C NMR spectrum of **4h**

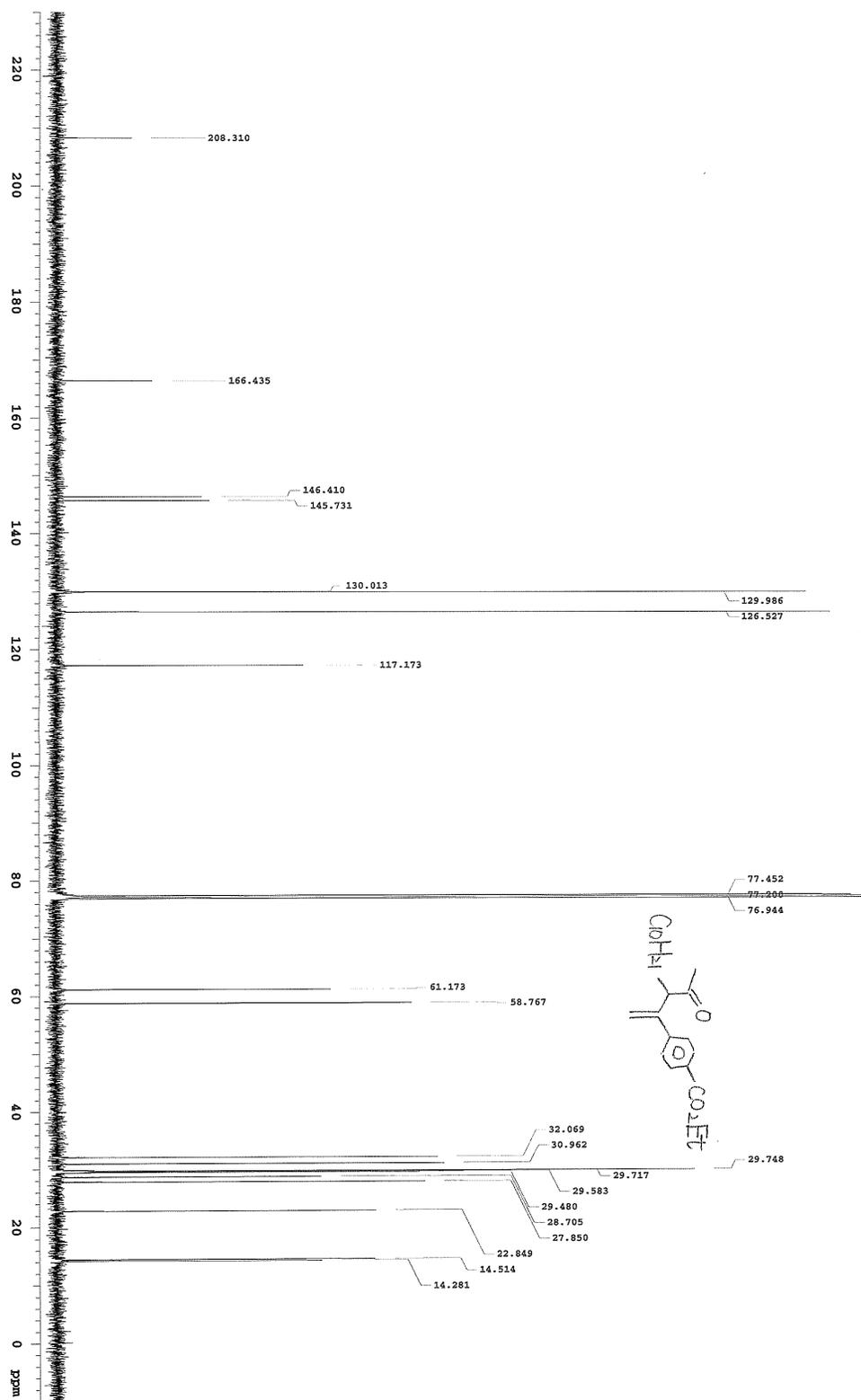


Figure S43. ¹H NMR spectrum of **4i**

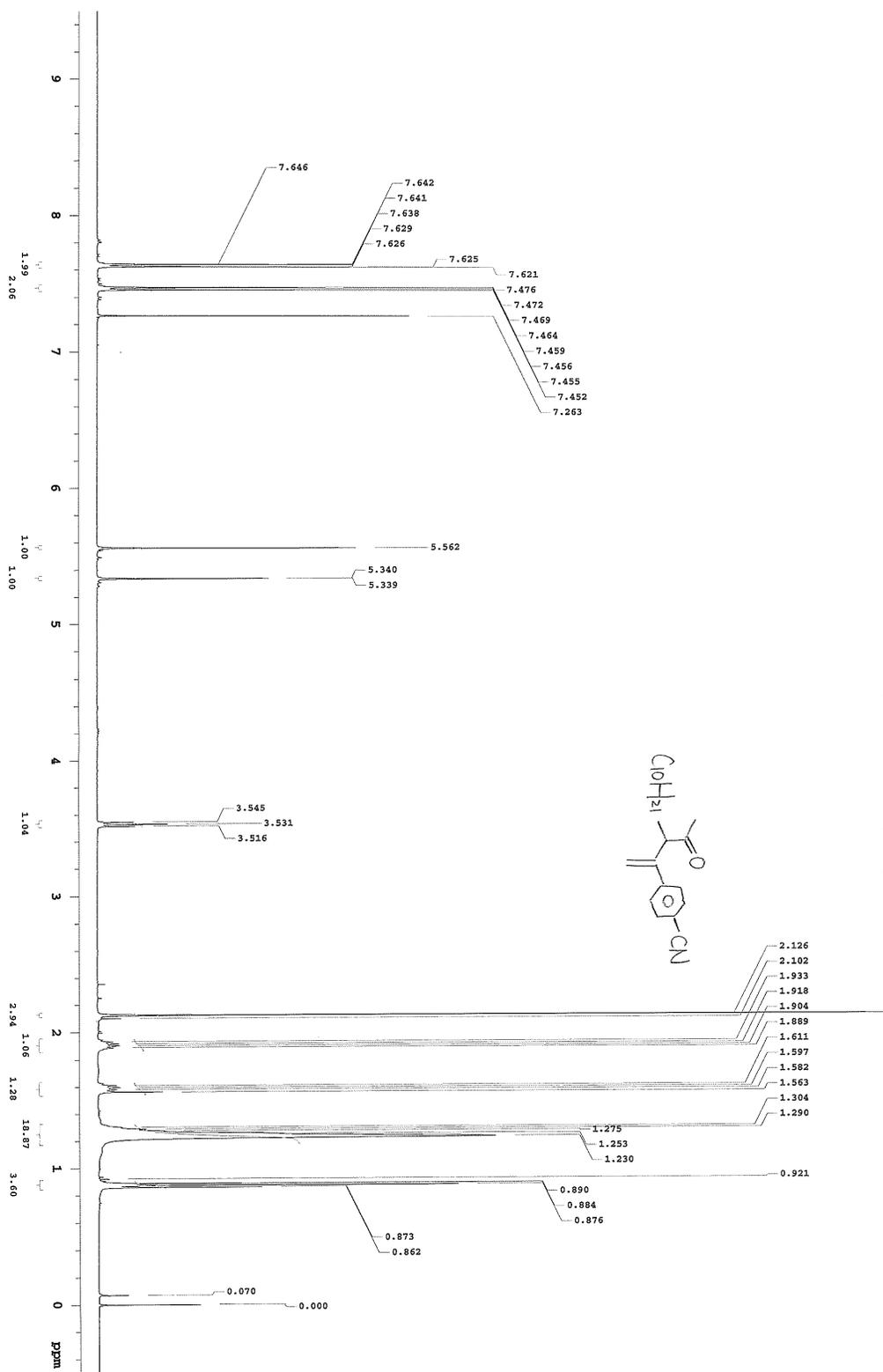


Figure S44. ^{13}C NMR spectrum of **4i**

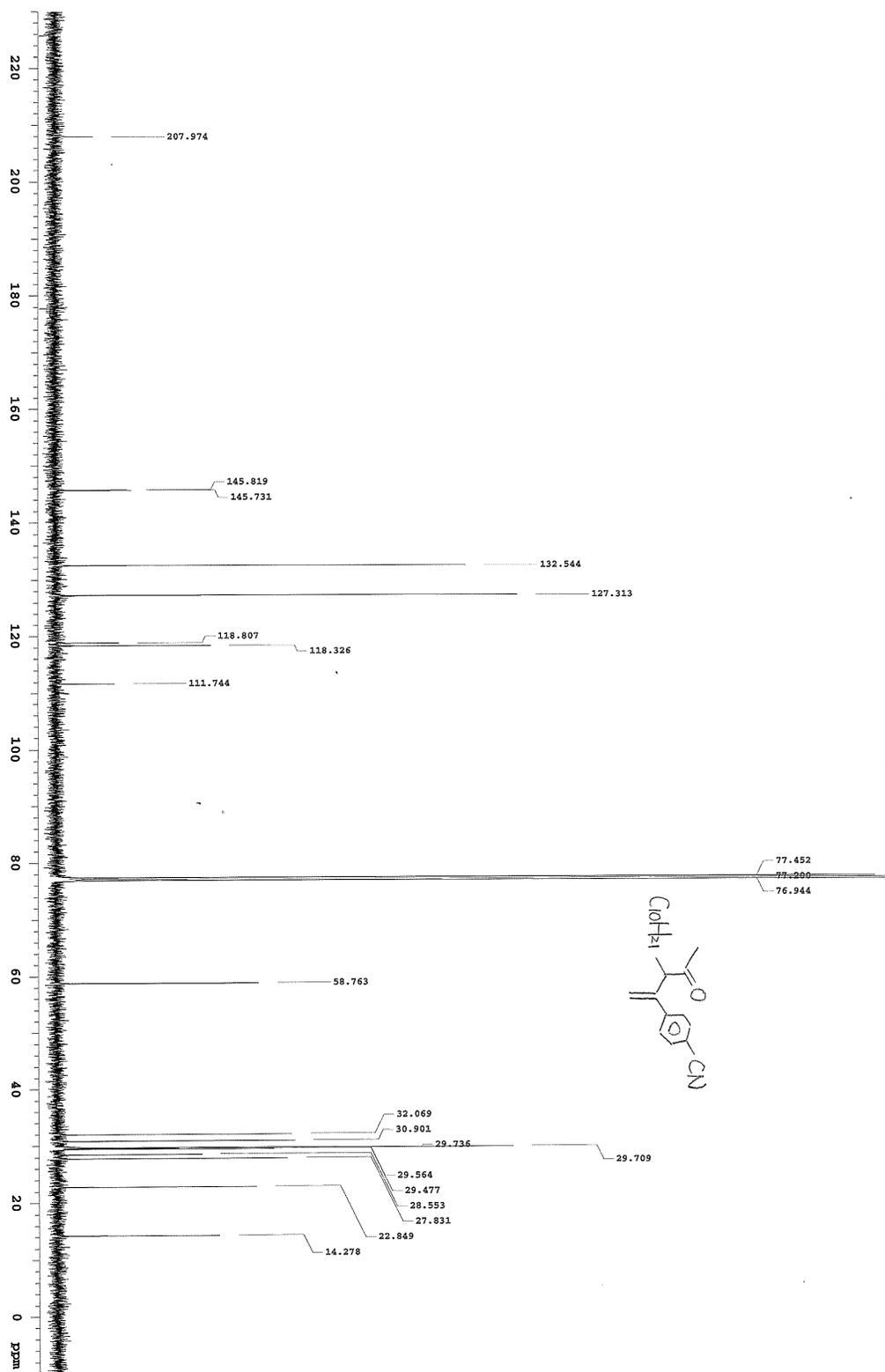


Figure S45. ¹H NMR spectrum of **5a**

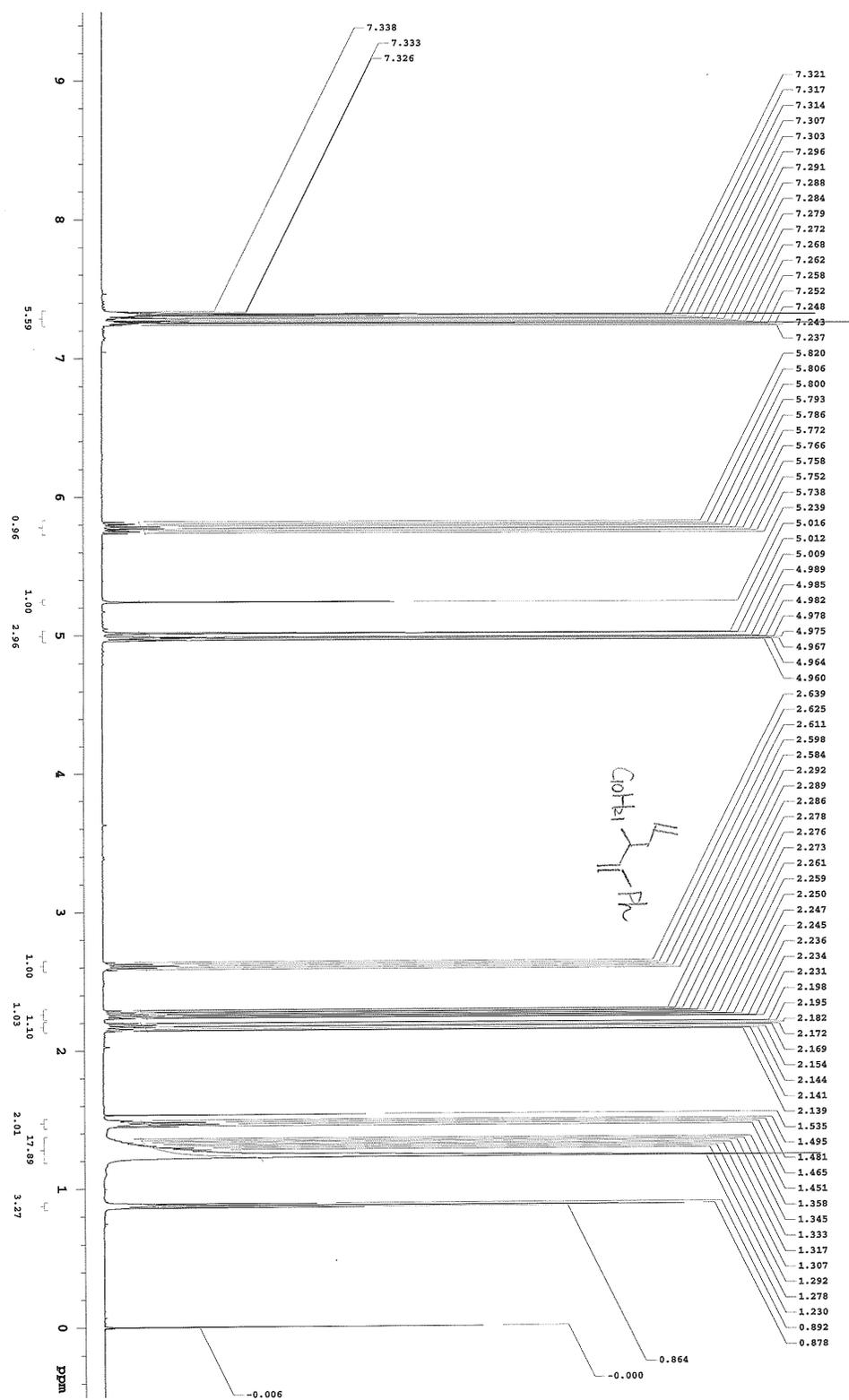


Figure S46. ^{13}C NMR spectrum of **5a**

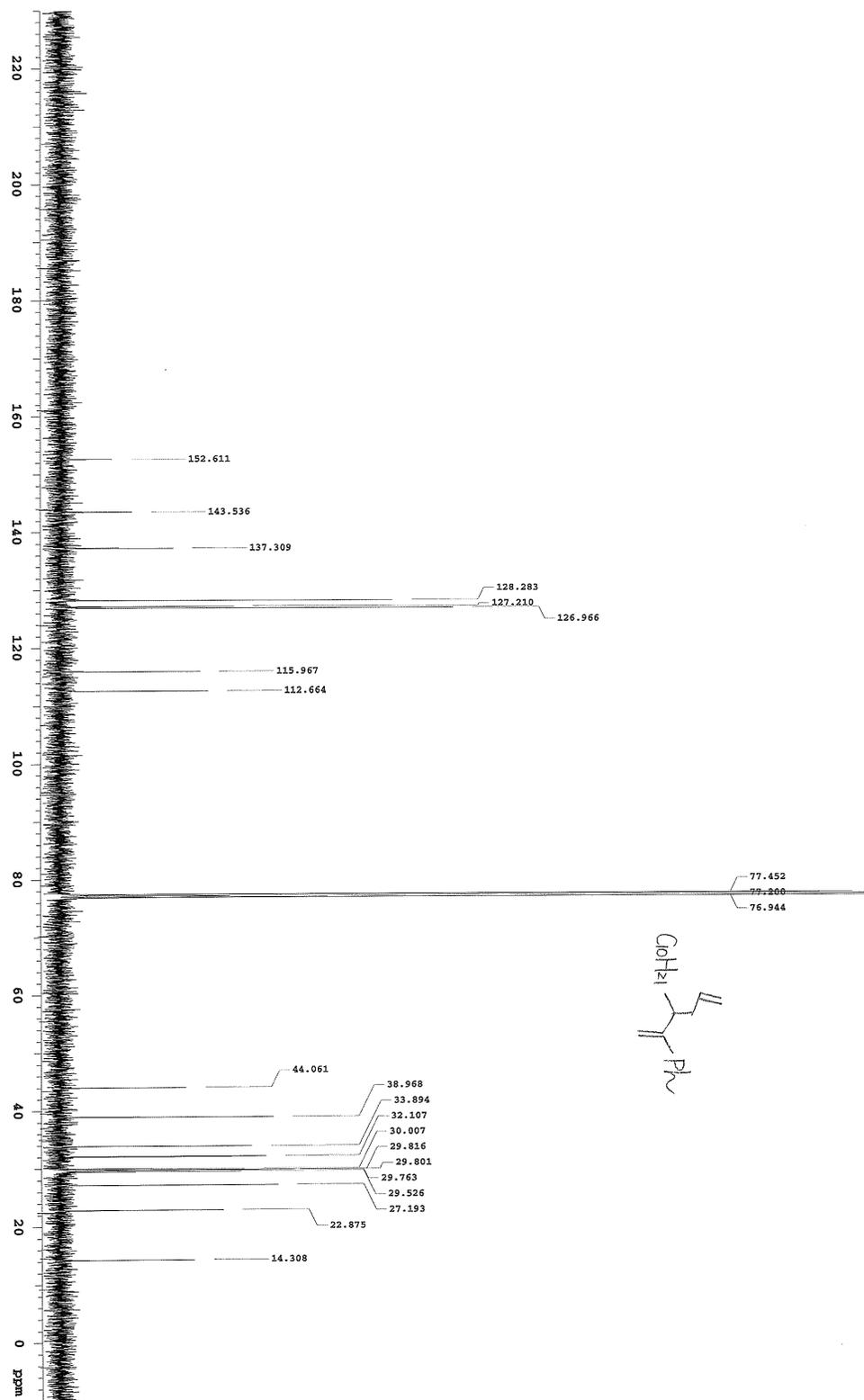


Figure S47. ^1H NMR spectrum of **5b**

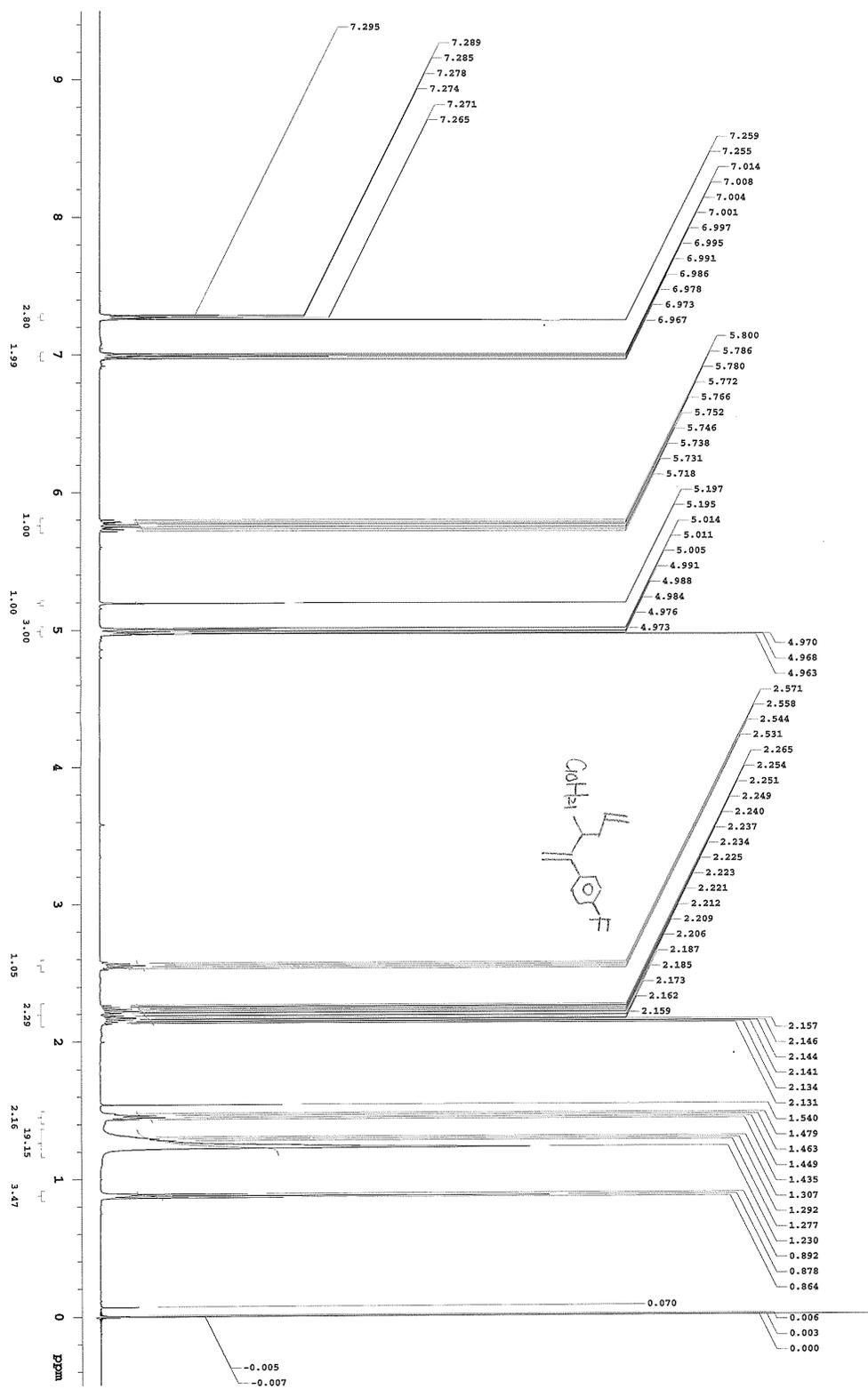


Figure S48. ^{13}C NMR spectrum of **5b**

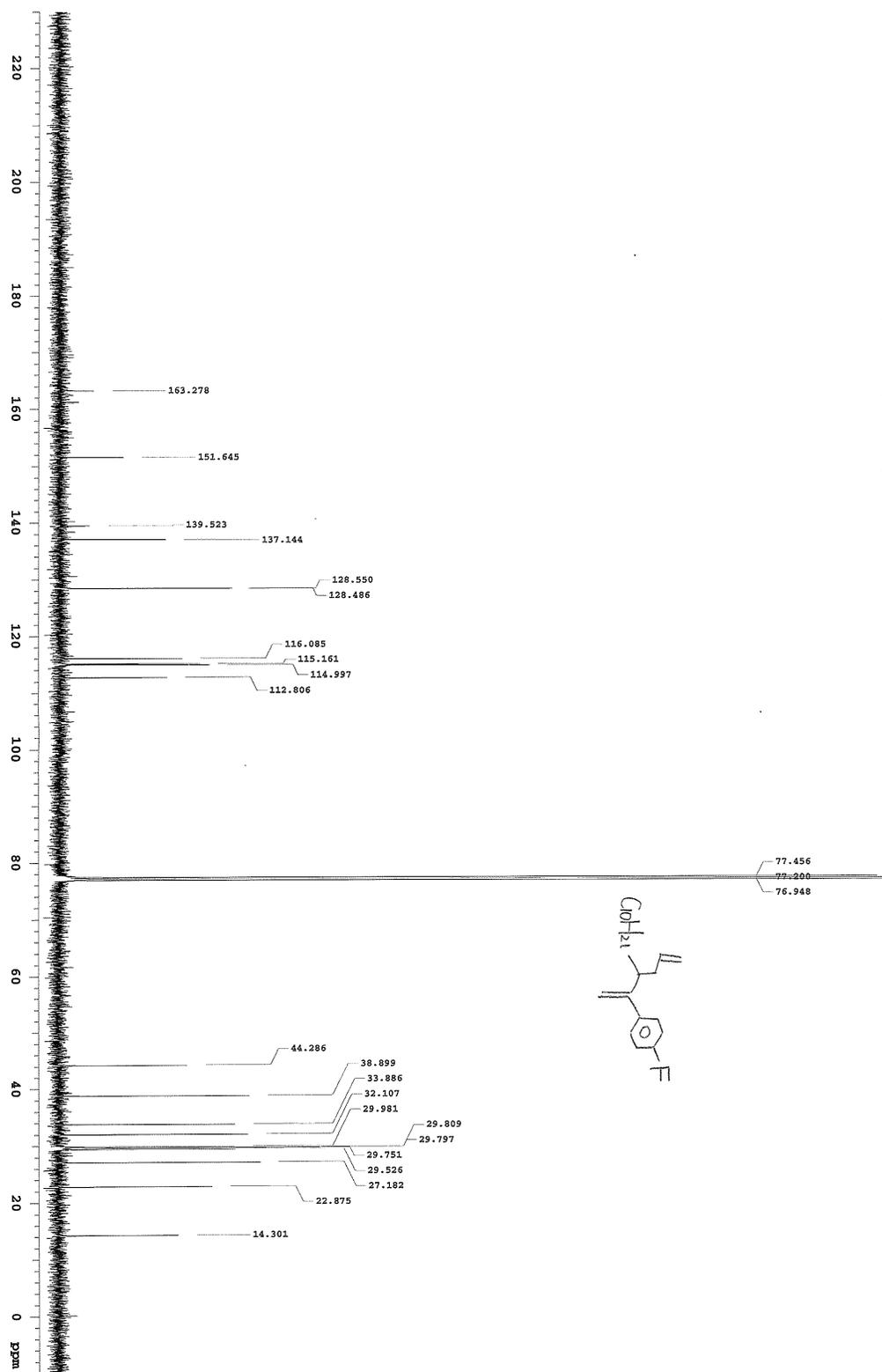


Figure S49. ¹H NMR spectrum of **6a**

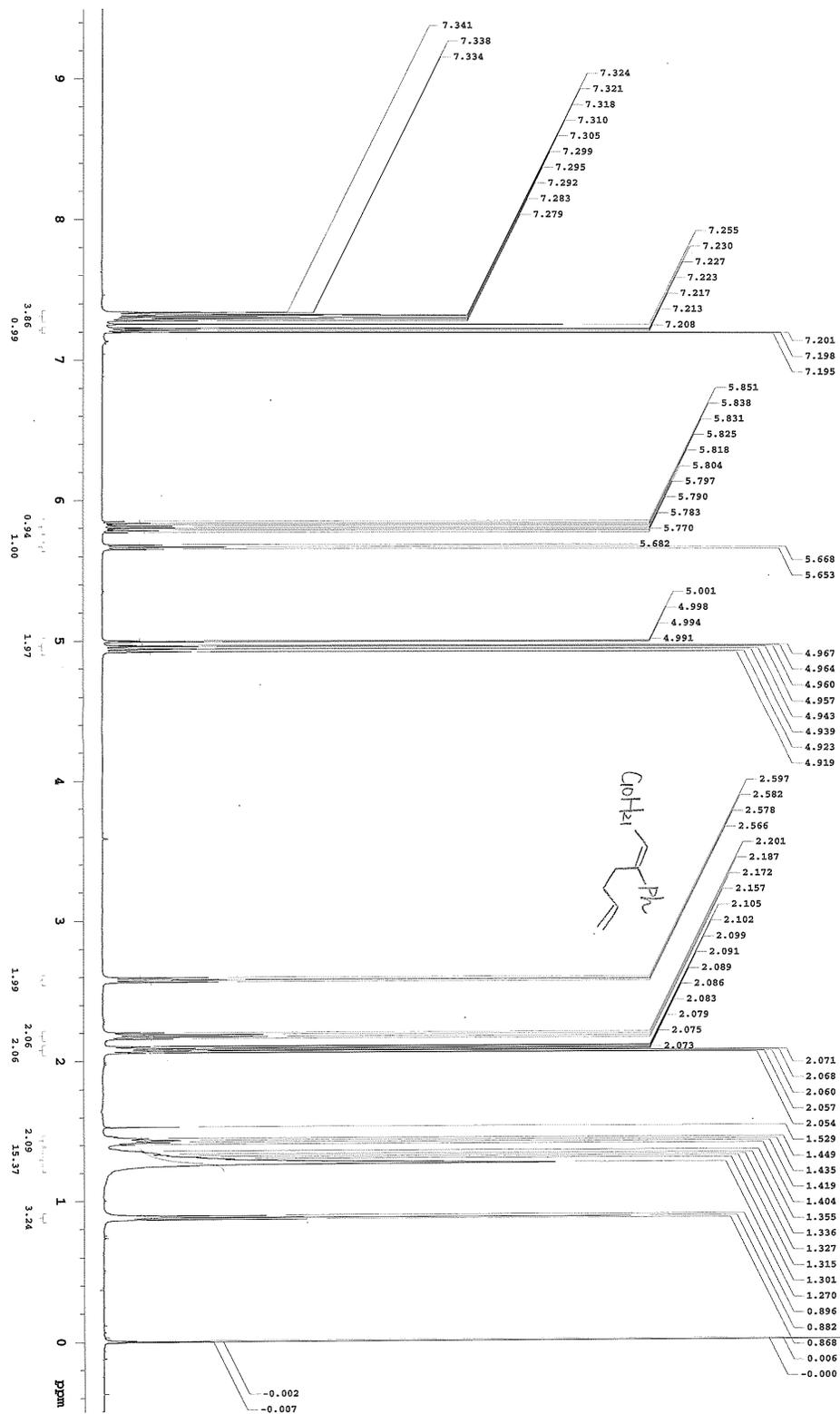


Figure S50. ^{13}C NMR spectrum of **6a**

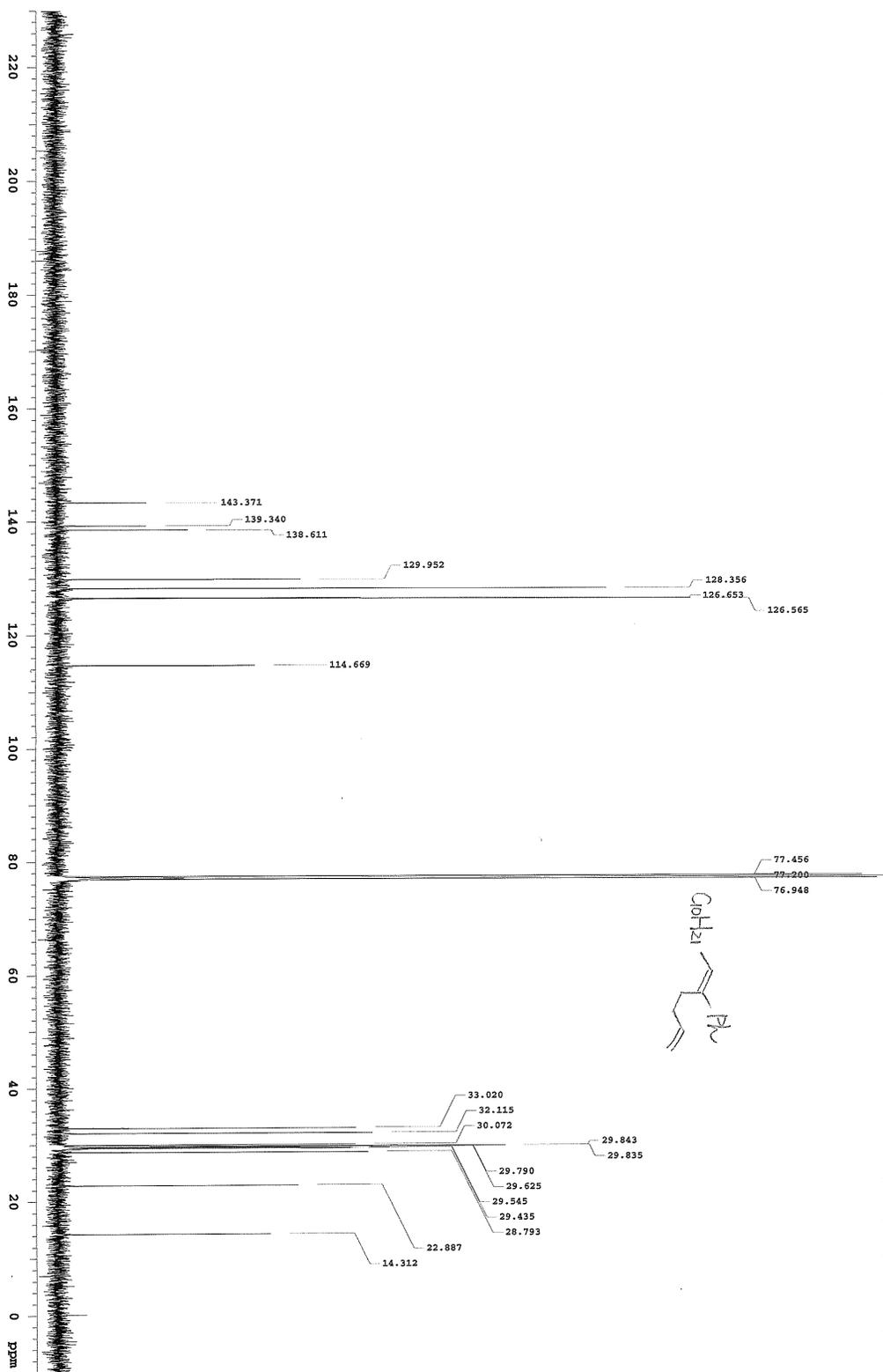


Figure S51. ¹H NMR spectrum of **6b**

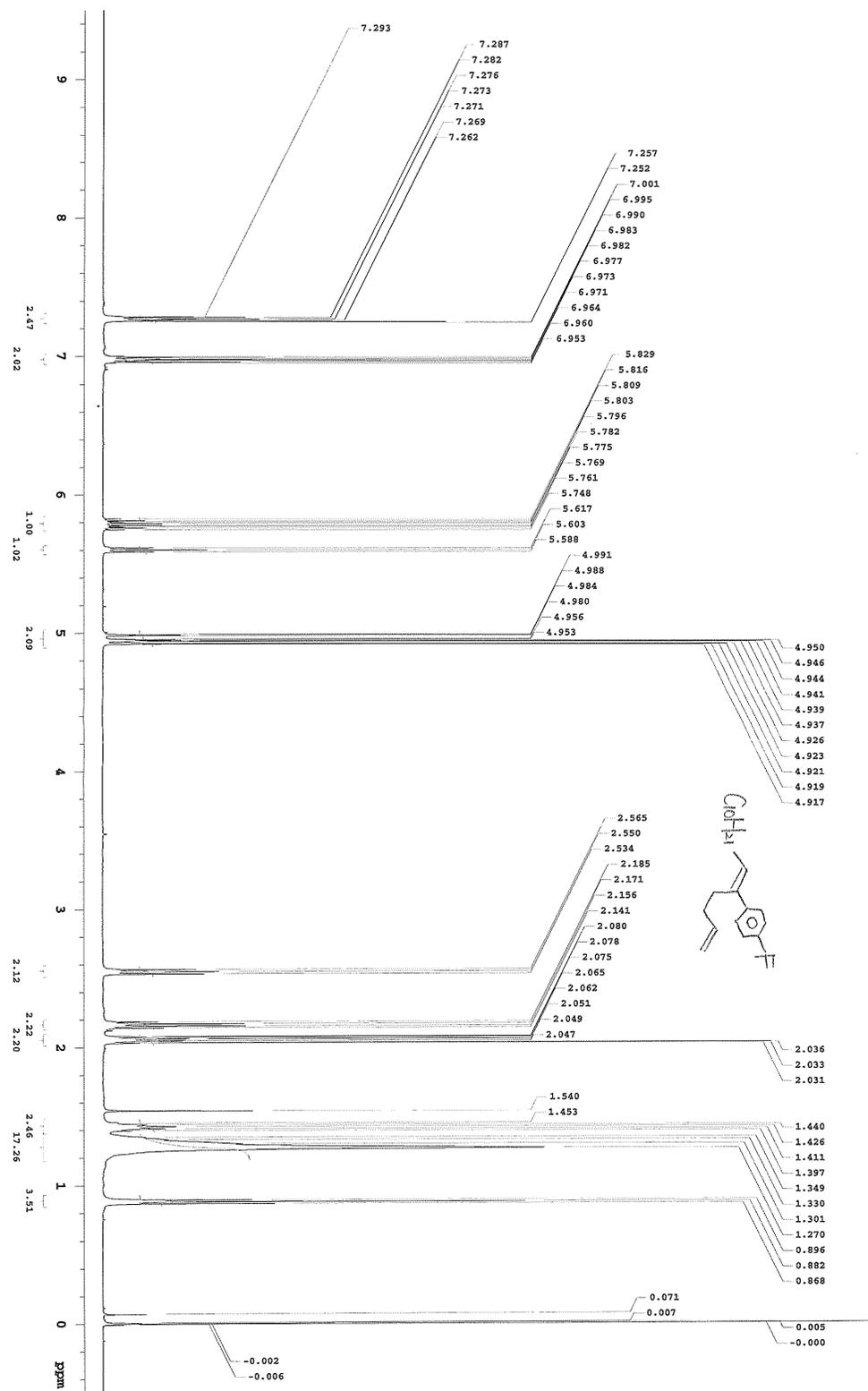


Figure S52. ^{13}C NMR spectrum of **6b**

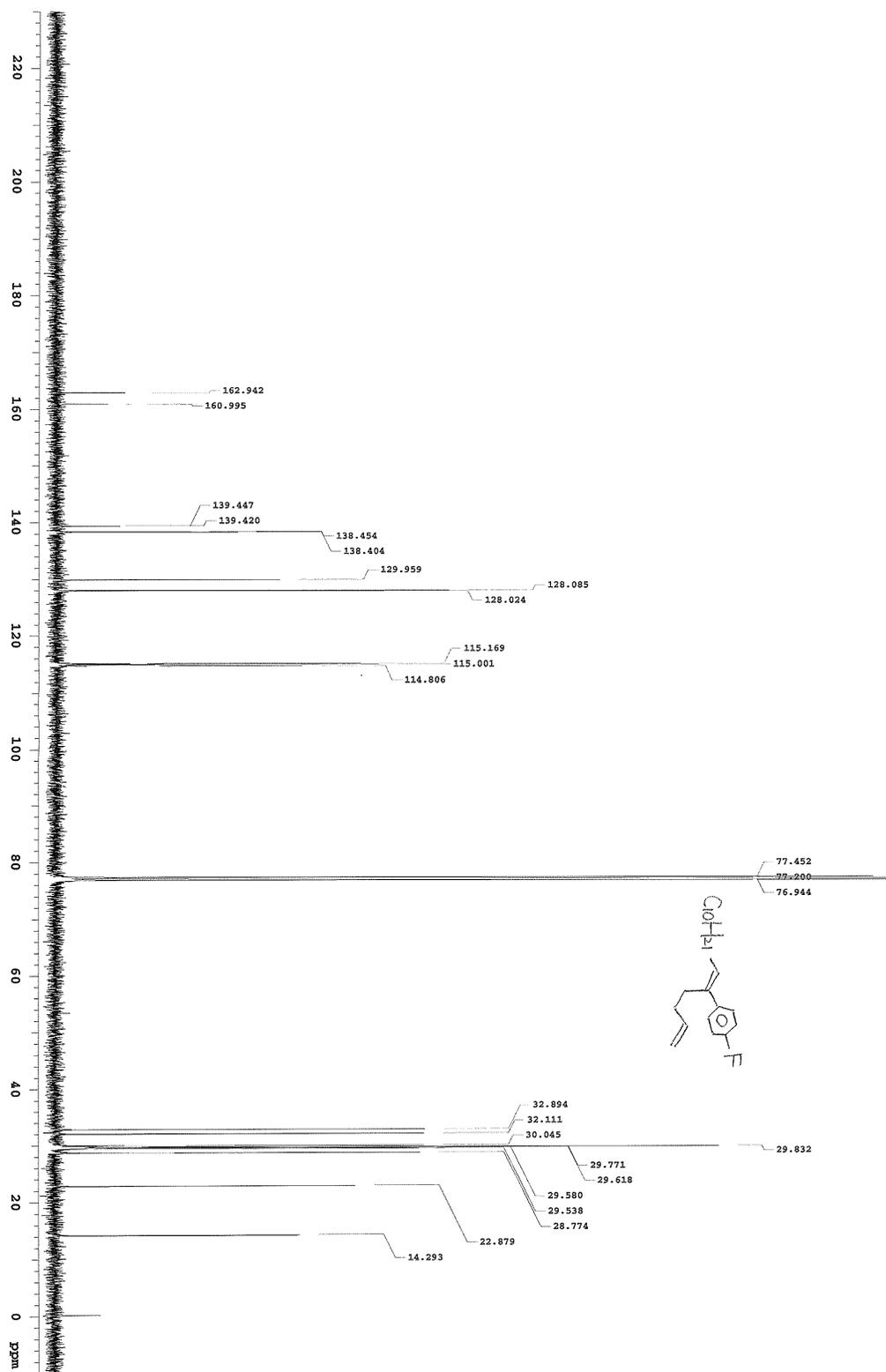


Figure S53. ¹H NMR spectrum of **7**

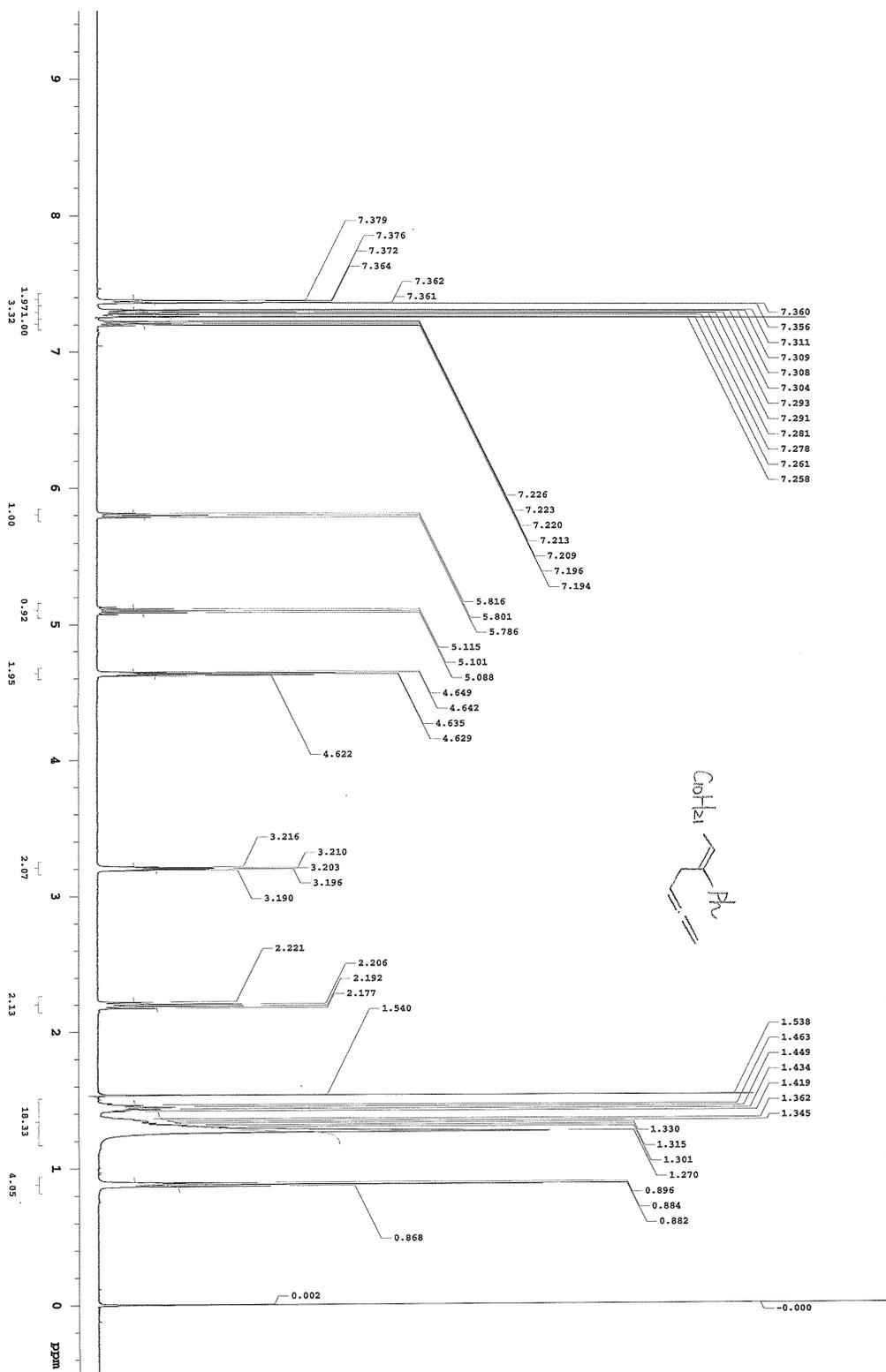


Figure S54. ^{13}C NMR spectrum of **7**

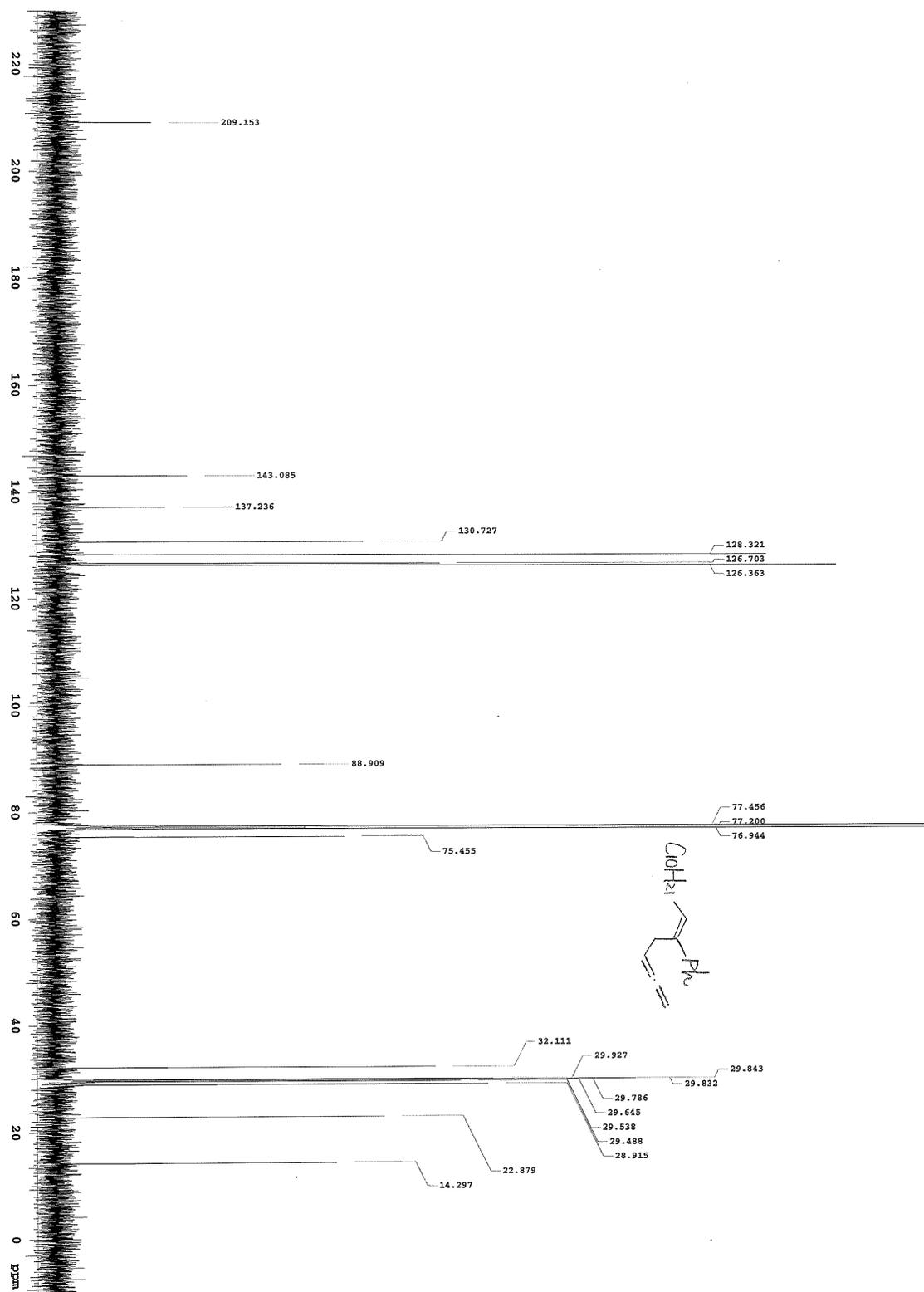


Figure S55. ¹H NMR spectrum of **8**

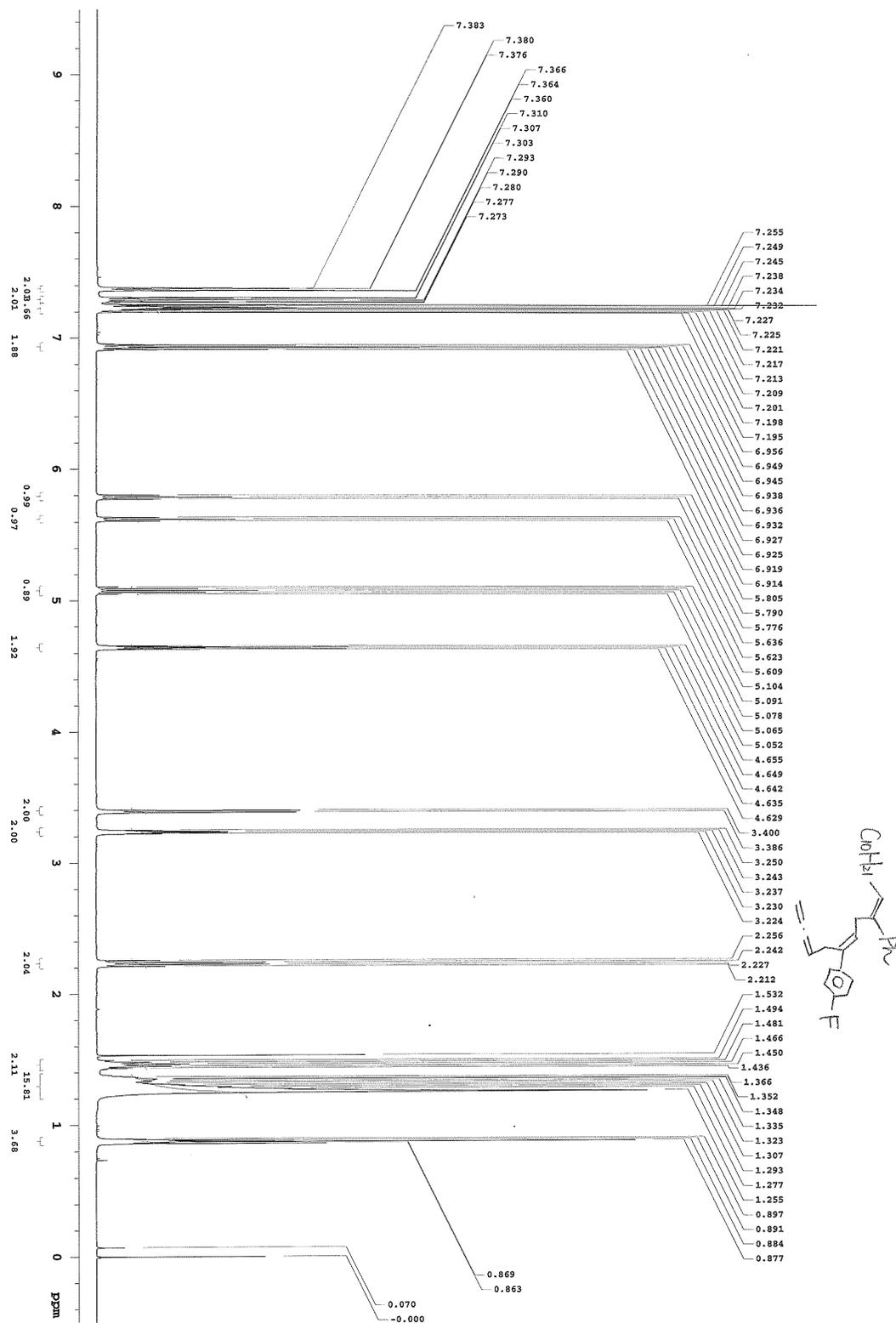


Figure S56. ^{13}C NMR spectrum of **8**

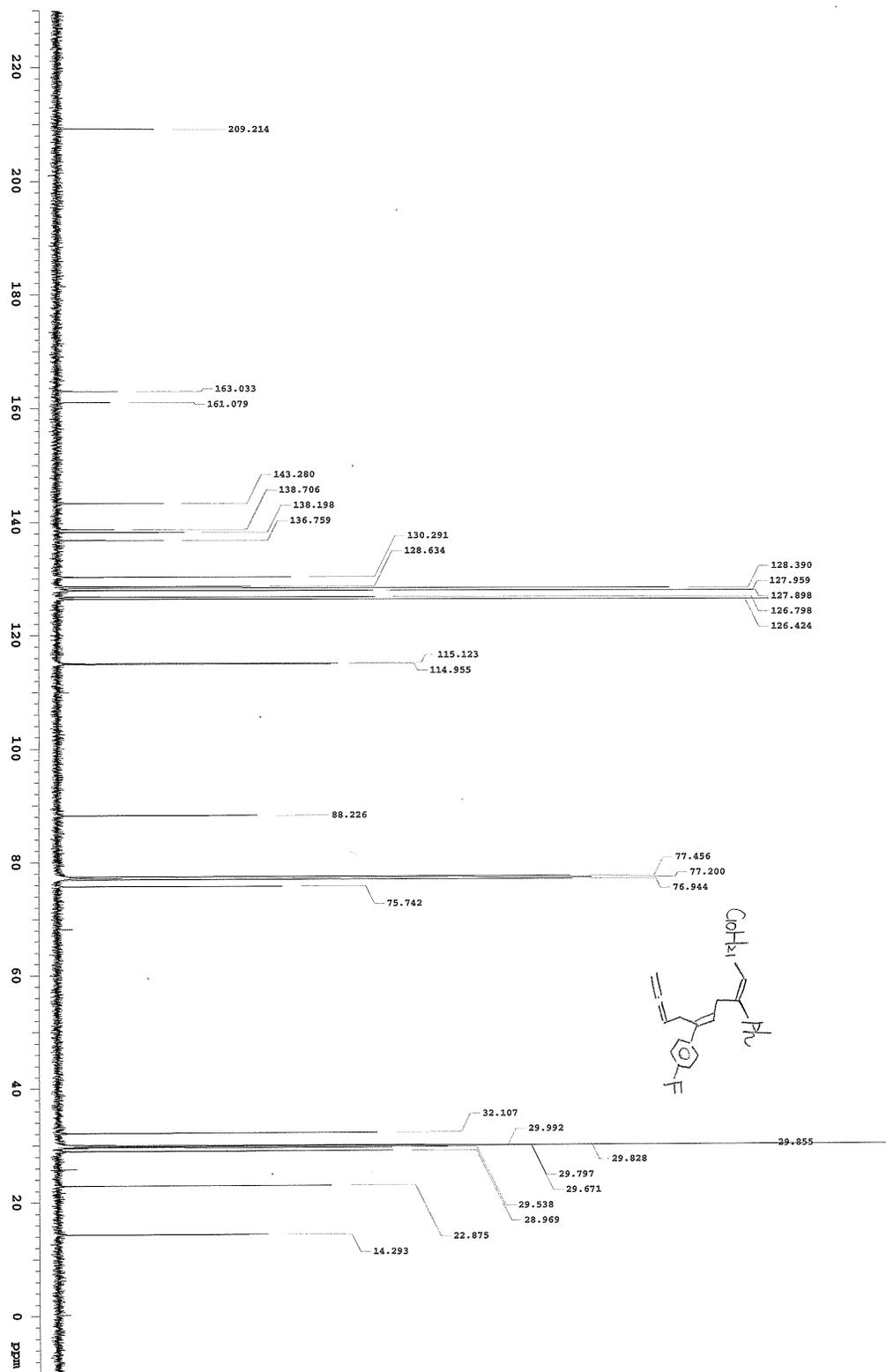


Figure S57. ¹H NMR spectrum of **9**

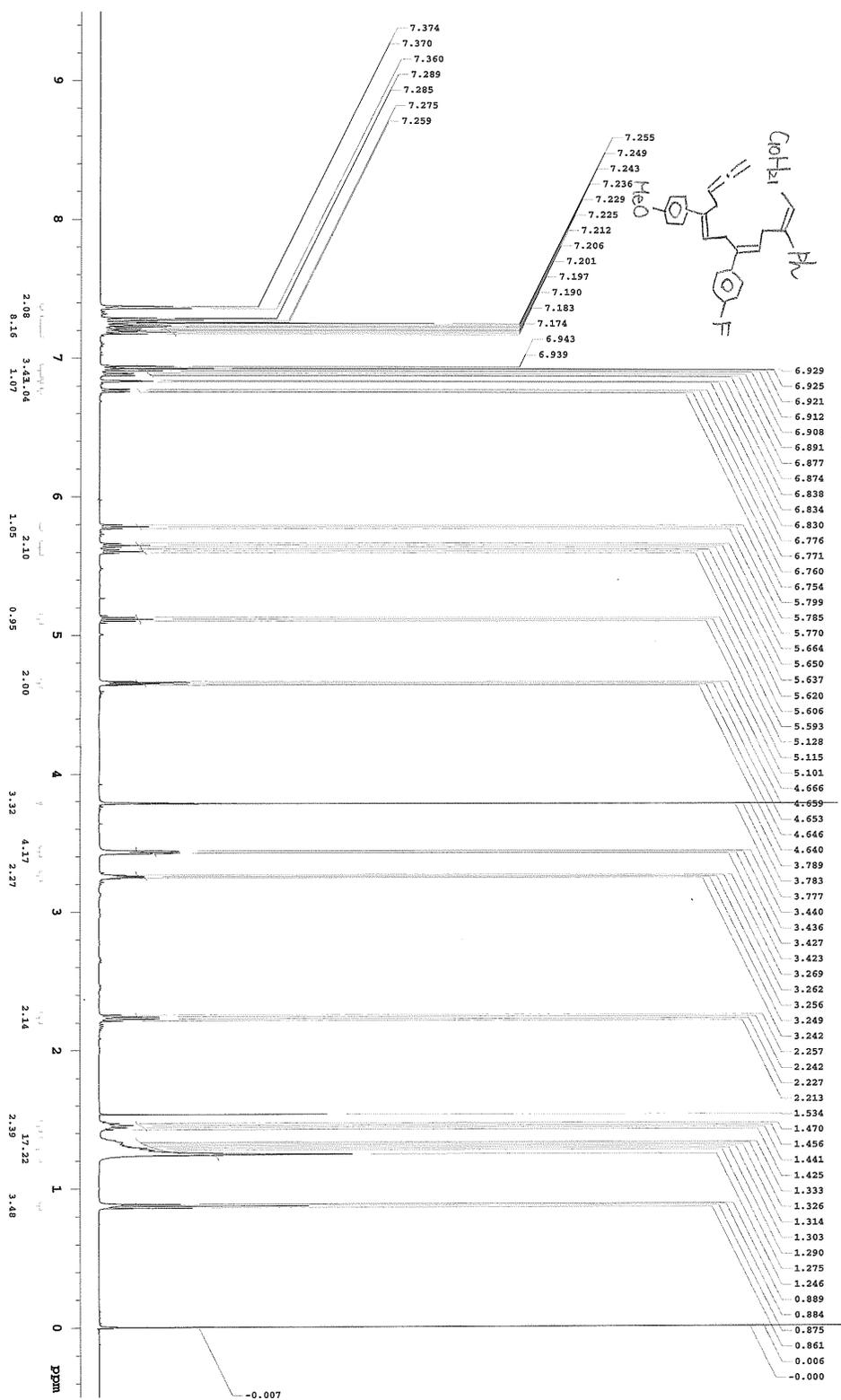


Figure S58. ^{13}C NMR spectrum of **9**

