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Supplementary Information

Generation and reaction of alkyl radicals in open reaction vessels

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General Experimental:

CAUTION: Although we had no incidents relating to the stability of the diazonium salts used in this work, care was taken to avoid the use of metal needles and spatulas.

All reactions were performed at room temperature under air using plastic tubing, plastic syringes, and oven dried glassware. Dimethylsulfoxide (DMSO) was dried over 3 Å molecular sieves. Diazonium salts (**1a**–**f**) and dibenzyl Hantzsch esters (**2** and **2a**) were synthesized using literature procedures.[1] Alkyl iodides were generated from the corresponding alcohols in the standard fashion or were commercially available. All other solvents and reagents were used as received from commercial sources. Melting points were determined using a Stanford Research Systems Optimelt automated melting point system and are uncorrected. Infrared spectra were acquired on a Bruker ALPHA FT-IR spectrometer as thin films, or neat. Absorption maxima are expressed in wavenumbers (cm^{-1}). ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker AVANCE III 500, a Bruker AVANCE III 400 spectrometer and Bruker AVANCE III 300 spectrometer (^{1}H frequencies 500, 400 and 300; ^{13}C frequencies 125, 100 and 75 MHz respectively). ^{1}H chemical shifts are expressed as parts per million (ppm) with residual chloroform (δ 7.26) as an internal reference and are reported as chemical shift (δ_H); relative integral; multiplicity (s = singlet, br = broad, d = doublet, t = triplet, dd = doublet of doublets, ddd = doublet of doublet of doublets, m = multiplet); and coupling constants (*J*) reported in Hz. ^{13}C NMR chemical shifts are expressed as parts per million (ppm) with residual chloroform (δ 77.1) as internal reference and are reported as chemical shift (δ_C). High resolution mass spectra were recorded on a Bruker Apex II Fourier Transform Ion Cyclotron Resonance mass spectrometer with a 7.0 T magnet, fitted with an off-axis Analytica electrospray source. Column chromatography was performed using Grace Davison, Merck, or Scharlau 40-60 μm (230-400 mesh) silica gel using commercial solvents. Analytical thin layer chromatography was performed using preconditioned plates (Merck TLC silica gel 60 F_{254} on aluminium) and visualised using UV light (254 nm and 365 nm), ethanolic anisaldehyde and potassium permanganate solutions.

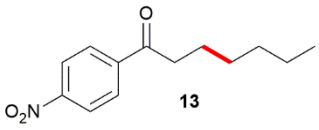
General Procedure 1

To a solution of corresponding alkyl iodide (0.35 mmol) and acceptor (0.70 mmol) in dry DMSO (0.2 mL) the solutions of 2,4,6-trimethylbenzenediazonium tetrafluoroborate (0.70 mmol) in dry DMSO (0.4 mL) and dibenzyl Hantzsch ester (0.70 mmol) in dry DMSO (0.4 mL) were added by syringes in one portion at room temperature under air. The resulting reaction mixture was stirred for 5 minutes and diluted with ethyl acetate (10 mL), water (20 mL) was added and the mixture was extracted with ethyl acetate (3 × 15 mL). The combined organic layers were dried over anhydrous sodium sulfate, concentrated under vacuum and the desired product was purified by column chromatography on silica get using hexane/ethyl acetate as eluent.

General Procedure 2

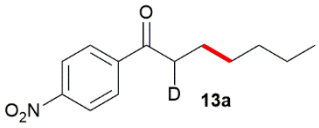
To a solution of butyl iodide (0.35 mmol), quinoline (0.70 mmol) and trifluoroacetic acid (3.5 mmol) in dry DMSO (0.1 mL) the solutions of 4-chlorophenyl tetrafluoroborate (0.35 mmol) in dry DMSO (0.3 mL) and dibenzyl Hantzsch ester (0.35 mmol) in dry DMSO (0.3 mL) were added by syringe pumps (0.1ml/min) at room temperature under air. The resulting reaction mixture was diluted with ethyl acetate (10 mL), added 1M NaOH (20 mL) extracted with ethyl acetate (3 × 15 mL). The combined organic layers were washed with water and dried over anhydrous sodium sulfate,

concentrated under vacuum and the desired product was purified by column chromatography on silica get using hexane/ethyl acetate as eluent.



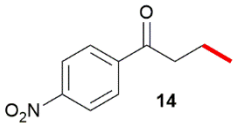
13

1-(4-Nitrophenyl)heptan-1-one (13)[2]: Isolated as an orange oil (72%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 9 (2H, d, J = 8.8 Hz), 8.09 (2H, d, J = 8.8 Hz), 3.00 (2H, t, J = 7.4 Hz), 1.70-1.77 (2H, m), 1.27-1.40 (6 H, m), 0.89 (3H, p, J = 3.5 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 198.9, 150.3, 141.5, 129.1, 123.9, 39.2, 31.6, 28.9, 24.0, 22.5, 14.0; ν_{max}/cm^{-1}: 2929, 2858, 1692, 1524, 1344, 1196, 853; HRMS (APPI) calcd for $C_{13}H_{17}NO_3$ (M^+) 235.12029, found 235.12029.



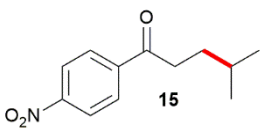
13a

1-(4-Nitrophenyl)heptan-1-one-2-*d* (13a): Isolated as an orange oil (35%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.30 (2H, d, J = 8.7 Hz), 8.09 (2H, d, J = 8.7 Hz), 2.89-3.04 (1H, m), 1.73 (2H, q, J = 7.3 Hz), 1.29-1.38 (6H, m), 0.88 (3H, m); ^{13}C NMR (101 MHz; $CDCl_3$): 198.9, 150.3, 141.5, 129.0, 123.8, 38.3-39.2 (m), 31.6, 28.9, 23.9, 22.5, 14.0; ν_{max}/cm^{-1}: 2955, 2928, 2857, 1690, 1604, 1524, 1344, 1317, 1239, 851, 741,710; HRMS (APPI) calcd for $C_{13}H_{16}DNO_3$ (M^+) 236.1267, found 236.12655.



14

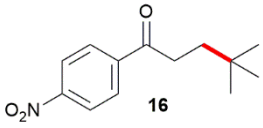
1-(4-Nitrophenyl)butan-1-one (14)[3]: Isolate as a yellow solid (65%); m.p 54-57 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.34 (2H, d, J = 8.7 Hz), 8.7 Hz), 3.02 (2H, t, J = 7.2 Hz), 1. 81 (2H, s, J = 7.3 Hz), 1.05 (3H, t, J = 7.4 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 198.7, 141.5, 129.0, 123.8, 64.4, 41.0, 25.3, 17.44, 13.7; ν_{max}/cm^{-1}: 2959, 2924, 2854, 1692, 1524, 1344, 1205, 853, 736; HRMS (APCI) HRMS (APCI) calcd for $C_{10}H_{11}NO_3$ (M^-) 193.07444, found 193.07548.



15

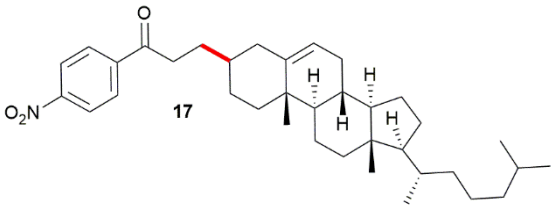
4-Methyl-1-(4-nitrophenyl)pentan-1-one (15): Isolated as an orange oil (41%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.30 (2H, d, J = 8.8 Hz), 8.11 (2H, d, J = 8.8 Hz), 3.00 (2H, t, J = 7.2 Hz), 1.62-1.67 (3H, m), 0.95 (6H, d, J = 6.4 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 199.1, 150.4, 141.6, 129.2, 124.0, 37.3, 33.0, 27.9,

22.5; ν_{max}/cm^{-1}: 2956, 2927, 2870, 1692, 1603, 1524, 1344,1201, 854, 740. HRMS (APPI) calcd for $C_{10}H_{11}NO_3$ (M^+) 221.10464, found 221.10482.



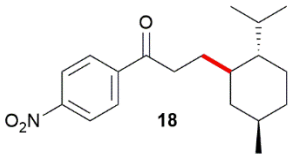
16

4,4-Dimethyl-1-(4-nitrophenyl)pentan-1-one (16): Isolated as orange oil (21%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.24 (2H, J = 8.6 Hz), 8.03 (2H, d, J = 8.5 Hz), 2.90 (2H, t, J = 8.0 Hz), 1.59 (2H, t, J = 8.1 Hz), 0.90 (9H, s); ^{13}C NMR (101 MHz; $CDCl_3$): 199.3, 150.3, 141.5, 129.1, 129.0, 123.9, 37.8, 34.9, 30.2, 29.2; ν_{max}/cm^{-1}: 2955, 2927, 2865, 1692, 1525, 1344, 1197, 855, 739. HRMS (APPI) calcd for $C_{13}H_{17}NO_3$ (M^+) 235.12029, found 235.12025.



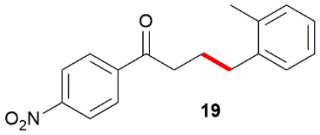
17

3-(3'-Cholesteryl)-1-(4'-nitrophenyl)propan-1-one (17): Isolated as a yellow oil (24%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 (2H, d, J = 8.6 Hz), 8.09 (2H, d, J = 8.6 Hz), 5.26-5.28 (1H, m), 3.00-3.04 (1H, m), 2.85-2.92 (1H, m), 2.50-2.53 (1H, m), 1.93-2.01 (3H, m), 1.77-1.88 (4H, m), 1.65-1.71 (3H, m), 1.45-11.59 (8H, m), 1.32-1.34 (4H, m), 1.25-1.29 (5H, m), 1.07-1.15 (6H, m), 1.01 (3H, s), 0.90 (3H, d, J = 6.3 Hz), 0.85 (6H, d, J = 6.3 Hz), 0.67 (3H, s); 199.1, 150.3, 141.5, 140.2, 129.1, 123.8, 121.6, 56.8, 56.2, 50.5, 42.3 39.8, 39.5, 37.5, 37.4, 36.7, 36.2, 35.8, 34.1, 34.0, 31.9, 31.9, 28.2, 28.0, 26.3, 25.4, 24.3, 23.8, 22.8, 20.8, 19.4, 18.7, 11.9; ν_{max}/cm^{-1}: 2929, 2866, 1687, 1523, 1346, 853, 737; HRMS (ESI+) calcd for $C_{36}H_{53}N_1O_3$ (M+H) 547.40200, found 547.40208.



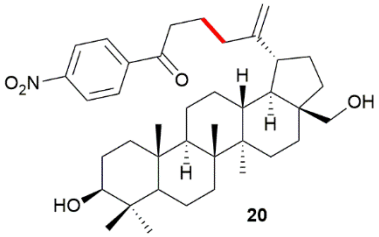
18

3-(2-Isopropyl-5-methylcyclohexyl)-1-(4-nitrophenyl)propan-1-one (18): Isolated as an orange oil (40%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 (2H, d, J = 8.6 Hz), 8.09 (2H, d, J = 8.7 Hz), 2.85-3.04 (2H, m), 1.97-2.03 (2H, m), 1.62-1.74 (4H, m), 1.37-1.53 (3H, m), 0.83-0.89 (9H, m), 0.74 (2H, d, J = 7.3 Hz), 0.69-0.74 (1H, m); ^{13}C NMR (101 MHz; $CDCl_3$): 199.3, 199.2, 150.3, 141.6, 129.1, 123.9, 48.3, 46.7, 41.2, 38.4, 38.2, 37.8, 35.9, 35.8, 35.3, 34.9, 32.8, 29.8, 29.3, 26.9, 26.6, 26.0, 25.2, 24.3, 22.8, 21.7, 21.6, 20.8, 19.7, 15.3; ν_{max}/cm^{-1}: 2951, 2917, 2864, 1694, 1525, 1455, 1344, 1201, 854, 738. HRMS (APPI) calcd for $C_{19}H_{27}NO_3$ (M^+) 317.19855, found 317.19851.



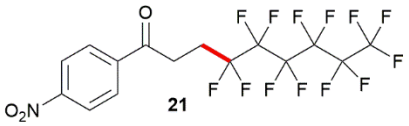
19

1-(4-Nitrophenyl)-4-(*o*-tolyl)butan-1-one (19): Isolated as a yellow solid (64%). m.p 86-88 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.30 (2H, d, J = 8.8 Hz), 8.06 (2H, d, J = 8.8 Hz), 7.12-7.16 (4H, m), 3.07 (2H, t, J = 7.0 Hz), 2.72 (2H, t, J = 7.8 Hz), 2.33 (3H, s), 2.06 (2H, p, J = 7.4 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 198.4, 150.4, 141.5, 139.5, 136.1, 130.45, 129.1, 129.0, 126.4, 126.1, 123.9, 38.5, 32.5, 24.2, 19.4; ν_{max}/cm^{-1}: 2953, 2887, 1690, 1525, 1344, 1319, 1196, 747. HRMS (APCI) calcd for $C_{17}H_{17}NO_3$ (M^+) 283.12029, found 283.12028.



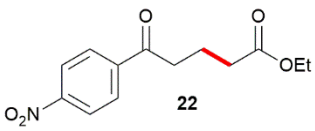
20

5-(9-Hydroxy-3a-(hydroxymethyl)-5a,5b,8,8,11a-pentamethylicosahydro-1H-cyclopenta[a]chrysen-1-yl)-1-(4-nitrophenyl)hex-5-en-1-one (20): Isolated as yellow oil (42%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.30 (2H, d, J = 8.5 Hz), 8.10 (2H, d, J = 8.7 Hz), 4.83 (1H, s), 4.67 (1H, s), 3.79 (1H, d, J = 10.7 Hz), 3.31 (1H, d, J = 10.9 Hz), 3.16-3.19 (1H, m), 3.00-3.10 (2H, m), 2.31 (1H, brs), 2.03-2.09 (3H, m), 1.93-1.98 (3H, m), 1.52-1.63 (8H, m), 1.32-1.43 (8H, m), 1.17-1.28 (6H, m), 1.03-1.08 (2H, m), 1.01 (3H, s), 0.96 (6H, s), 0.81 (3H, s), 0.75 (3H, s); ^{13}C NMR (101 MHz; $CDCl_3$): 198.5, 153.9, 150.3, 141.5, 129.0, 123.9, 107.9, 79.0, 60.5, 55.3, 50.4, 49.5, 47.8, 42.7, 40.9, 38.9, 38.8, 38.7, 37.3, 37.2, 34.3, 33.9, 29.7, 29.3, 28.0, 27.4, 27.1, 21.8, 20.9, 18.3, 16.1, 16.0, 15.4, 14.8; ν_{max}/cm^{-1}: 2940, 2870, 1693, 1528, 1345, 1031; HRMS (ESI+) calcd for $C_{39}H_{57}NO_5$ (M+Na) 642.41290, found 642.41241.



21

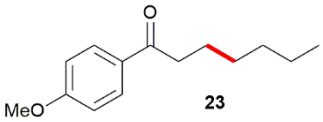
4,4,5,5,6,6,7,7,8,8,9,9,9-Tridecafluoro-1-(nitrophenyl)nonan-1-one (21): Isolated as a light-yellow solid (28%). m.p 100-102 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.34 (2H, d, J = 8.6 Hz), 8.15 (2H, d, J = 8.6 Hz), 3.36 (2H, t, J = 7.6 Hz), 2.64 (2H, m); ^{13}C NMR (101 MHz; $CDCl_3$): 194.9, 150.7, 140.4, 129.1, 124.0, 30.2, 25.4 (1C, t, J = 21.7 Hz); ν_{max}/cm^{-1}: 2926, 1691, 1527, 13320, 1224, 1186, 1139, 1100, 1053, 981, 860, 742, 700, 648, 566, 529. HRMS (APCI) calcd for $C_{15}H_8F_{13}NO_3$ (M^-) 497.03021, found 497.03394.



22

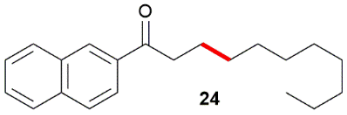
Ethyl 5-(4-nitrophenyl)-5-oxopentanoate (22)[4]: Isolated as an orange oil (75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 (2H, d, J = 8.7 Hz), 8.10 (2H, d, J = 8.7 Hz), 4.13 (2H, q, J = 7.1 Hz), 3.10 (2H, t, J = 7.1 Hz),

2.44 (2H, t, J = 7.0 Hz), 2.03-2.09 (2H, m), 1.23-1.26 (3H, m); ^{13}C NMR (101 MHz; $CDCl_3$): 197.8, 173.1, 150.3, 141.2, 129.1, 123.9, 60.5, 38.0, 33.1, 29.7, 19.1, 14.2; ν_{max}/cm^{-1}: 2928, 1727, 1692, 1524, 1345, 1317, 1202, 1149, 854, 741; LRMS (ESI) m/z: 288.06 (M+Na)



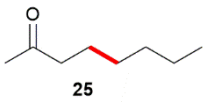
23

1-(4-Methoxyphenyl)heptan-1-one (23)[5]: Isolated as a yellow oil (56%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.93 (2H, d, J = 9.2 Hz), 6.92 (2H, d, J = 8.8 Hz), 3.85 (3H, s), 2.89 (2H, t, J = 7.4 Hz), 1.67-1.75 (2H, m), 1.30-1.38 (6H, m), 0.89 (2H, m); ^{13}C NMR (101 MHz; $CDCl_3$): 199.3, 163.4, 130.4, 113.8, 55.5, 38.4, 31.8, 29.2, 24.7, 22.6, 14.1; ν_{max}/cm^{-1}: 2957, 2931, 1669, 1600, 1576, 1252, 1237, 1175, 1033, 836, 798, 603; LRMS (APCI) m/z: 221.08 [M+H].



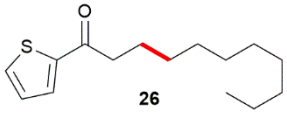
24

1-(Naphthalen-2-yl)undecane-1-one (24): Isolated as a yellow solid (51%). m.p 42-44 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.47 (1H,s), 8.03 (1H, dd, J_1 = 1.6 Hz, J_2 = 8.6 Hz), 7.96 (1H, J = 8.0 Hz), 7.87 (2H, t, J = 7.5 Hz), 7.52-7.61 (2H, m), 3.09 (2H, d, J = 7.4 Hz), 1.80 (2H, p, J = 7.4 Hz), 1.27-1.42 (14 H, m), 0.87-0.90 (3H, m); ^{13}C NMR (101 MHz; $CDCl_3$): 200.6, 135.5, 134.5, 132.5, 129.6, 129.5, 128.4, 128.3, 127.8, 126.7, 124.0, 38.7, 31.9, 29.61, 29.55, 29.5, 29.4, 24.6, 22.7, 14.1 ; ν_{max}/cm^{-1}: 2918, 2849, 1667, 1467, 1375, 1183, 826, 745, 478; HRMS (ESI+) calcd for $C_{21}H_{28}O_1$ (M+H) 297.22129, found 297.22104



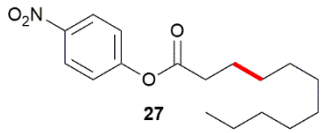
25

Octan-2-one (25)[6]: Isolated as a yellow oil (40%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 2.22 (2H, t, J = 7.4 Hz), 1.91 (3H, s), 1.36 (2H, p, J = 7.1Hz), 1.05-1.12 (6H, m), 0.67 (3H, t, J = 6.7 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 208.7, 43.5, 31.4, 29.5, 28.7, 23.6, 22.3, 13.7; ν_{max}/cm^{-1}: 2985, 2926, 2855, 1708, 1469, 1439, 1043, 1021, 747, 660; LRMS (APCI) m/z: 129.08 [M+H].

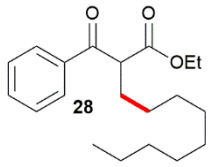


26

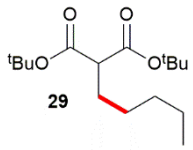
1-(Thiophen-2-yl)undecan-1-one (26)[7]: Isolated as a yellow oil (41%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.70 (1H, d, J = 3.7 Hz), 7.60 (1H, d, J = 4.9 Hz), 7.12 (1H, t, J = 4.3 Hz), 2.89 (2H, t, J = 7.5 Hz), 1.74 (2H, J = 7.3 Hz), 1.26-1.33 (14H, m), 0.88 (3H, t, J = 6.7 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 193.6, 144.6, 133.3, 131.6, 128.0, 39.5, 31.9, 29.6, 29.5, 29.4, 29.34, 29.3, 24.9, 22.7, 14.1; ν_{max}/cm^{-1}: 2924, 2853, 1664, 1416, 1236, 720; LRMS (APCI) m/z: 253.11 [M+H].



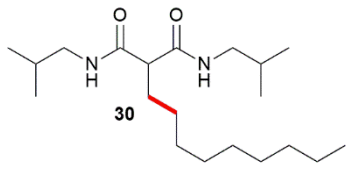
4-Nitrophenyl undecanoate (27)[8]: Isolated as an orange oil (40%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.18 (2H, d, J = 9.0 Hz), 7.20 (2H, d, J = 9.0 Hz), 2.52 (2H, t, J = 7.5 Hz), 1.69 (2H, p, 7.4 Hz), 1.20-1.34 (14H, m), 0.81 (3H, t, J = 6.5 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 171.3, 155.5, 145.3, 125.2, 122.4, 34.3, 31.9, 29.5, 29.4, 29.3, 29.2, 20.0, 24.7, 22.7, 14.1; ν_{max}/cm^{-1}: 2924, 2854, 1767, 1525, 1346, 1209, 1161, 1132, 1103, 864 ;LRMS (APCI) m/z: 308.21 [M+H].



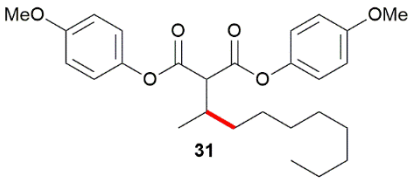
Ethyl 2-benzoylundecanoate (28): Isolated as an orange oil (80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.98 (2H, d, J = 7.4 Hz), 7.58 (1H, t, J = 7.4 Hz), 7.47 (2H, t, J = 7.6 Hz), 4.29 (1H, t, J = 7.2 Hz), 4.14 (2H, q, J = 6.9 Hz), 1.98-2.03 (2H, m), 1.24-1.33 (14H, m), 1.65 (3H, t, J = 7.1 Hz), 0.87 (3H, t, J = 6.8 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 195.3, 170.1, 136.4, 133.4, 128.7, 128.6, 61.3, 54.4, 31.9, 29.5, 29.4, 29.3, 29.2, 29.0, 27.6, 22.7, 14.1, 14.0; ν_{max}/cm^{-1}: 2924, 2854, 1735, 1686, 1448, 1231, 1182, 690; HRMS (ESI+) calcd for $C_{20}H_{30}O_3$ (M+H) 319.22677, found 319.22649



Di-tert-butyl 2-pentylmalonate (29)[9]: Isolated as a light-yellow liquid (75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 3.09 (1H, t, J = 7.6 Hz), 1.78 (2H, m), 1.44 (18H, s), 1.26-1.31 (6H, m), 0.86 (3H, t, J = 6.8 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 169.1, 81.2, 54.1, 31.5, 28.6, 28.0, 28.0, 26.9, 22.5, 14.0 ; ν_{max}/cm^{-1}: 2977, 2932, 1726, 1367, 1286, 1248, 1136, 848; HRMS (ESI+) calcd for $C_{16}H_{30}O_4$ (M+Na) 309.20363, found 309.20330.

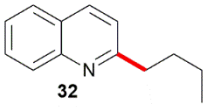


***N,N'*-Diisobutyl-2-nonylmalonamide (30):** Isolated as yellow oil (61%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.80 (2H, brs), 3.07 (4H, m), 2.97 (1H, t, J = 7.3 Hz), 1.82-1.85 (2H, m), 1.74-1.81 (2H, m), 1.24-1.28 (14H, m), 0.86 -0.91 (15H); ^{13}C NMR (101 MHz; $CDCl_3$): 171.2, 55.7, 46.9, 33.0, 31.9, 29.7, 29.5, 29.4, 29.2, 28.4, 27.7, 22.6, 20.0, 20.0, 14.0; ν_{max}/cm^{-1}: 3298, 2956, 2922, 2870, 2853, 1665, 1655, 1466; HRMS (ESI+) calcd for $C_{20}H_{40}N_2O_2$ (M+Na) 363.30001, found 363.29975.



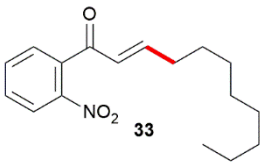
31

Bis(4-methoxyphenyl) 2-(decan-2-yl)malonate (31): Isolated as orange oil (26%).[1]H NMR (400 MHz, $CDCl_3$) δ: 7.05 (24, d, J = 9.0 Hz), 6.90 (4H, d, J = 9.0 Hz), 3.80 (6H, s), 3.72 (1H, d, J = 7.3 Hz), 2.42-2.50 (1H, m), 1.38-1.47 (4H, m), 1.26-1.28 (10H, m), 1.20 (3H, d, J = 6.8 Hz), 0.88 (3H, t, J = 6.8 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 167.7, 167.5, 157.5, 144.0, 122.1, 122.1, 114.6, 57.3, 55.6, 34.5, 33.6, 31.9, 29.6, 29.5, 29.3, 27.0, 22.7, 17.1, 14.1; ν_{max}/cm^{-1}: 2926, 2854, 1751, 1505, 1249, 1190, 1118, 1034; HRMS (ESI+) calcd for $C_{27}H_{36}O_6$ (M+Na) 479.24041, found 479.24001.



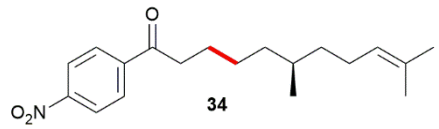
32

2-Butylquinoline (32)[10]: Isolated as orange oil (51%). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.08 (2H, d, J = 8.4 Hz), 7.76 (1H, d, J = 8.0 Hz), 7.67 (1H, t, J = 7.2 Hz), 7.47 (1H, t, J = 7.4 Hz), 7.29 (1H, d, J = 8.4 Hz), 2.97 (2H, t, J = 7.9 Hz), 1.75-1.85 (2H, m), 1.44 (2H, s, J = 7.4 Hz), 0.96 (3H, t, J = 7.3 Hz); ^{13}C NMR (75 MHz; $CDCl_3$): 163.1, 147.7, 136.3, 129.3, 128.7, 127.4, 126.7, 125.7, 121.4, 38.9, 32.2, 22.6, 14.0; ν_{max}/cm^{-1}: 2959, 2932, 2872, 1602, 1506, 1458, 1376, 1363, 828, 757, 625, 478. HRMS (APPI) calcd for $C_{13}H_{15}N$ (M+H) 186.12773, found 186.12782.



33

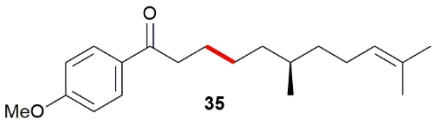
(*E*)-1-(2-Nitrophenyl)undec-2-en-1-one (33): Isolated as yellow oil (57%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.06 (1H, d, J = 1.2 Hz), 7.70 (1H, t, J = 7.4 Hz), 7.56-7.60 (1H, m), 7.47 (1H, dd, J_1 = 1.8 Hz, J_2 = 7.4 Hz), 7.26-7.36 (2H, m), 2.58 (2H, q, J = 6.9 Hz), 1.24-1.32 (12 H, m), 0.88 (3H, t, J = 6.6 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 190.9, 150.5, 137.3, 132.9, 129.5, 127.2, 124.7, 123.3, 30.8, 28.8, 28.7, 28.3, 28.1, 28.0, 21.6, 13.1; ν_{max}/cm^{-1}: 2924, 2854, 1675, 1530, 1347, 1259, 1021, 790; LRMS (APCI) m/z: 290.19 [M+H].



34

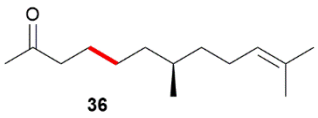
(*S*)-6,10-Dimethyl-1-(4-nitrophenyl)undec-9-en-1-one (34): Isolated as orange oil (40%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.30 (2H, d, J = 8.8 Hz), 8.01 (2H, d, J = 8.8 Hz), 5.07-5.5.11 (1H, m), 3.01 (2H, t, J = 7.3 Hz), 1.89-2.00 (2H, m), 1.72-1.75 (1H, m), 1.67 (3H, s), 1.59 (3H, s), 1.21-1.44 (6H, m), 1.12-1.1 (2H, m), 0.87 (3H, d, J = 6.5 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 198.9, 150.3, 141.6, 131.2, 129.1, 125.0,

123.9, 39.3, 37.2, 36.8, 32.4, 26.8, 25.8, 25.6, 24.4, 19.6, 17.8; ν_{max}/cm^{-1}: 2922, 2855, 1694, 1525, 1344, 1196, 745; HRMS (APPI) calcd for $C_{19}H_{27}NO_3$ (M^+) 317.19855, found 317.19850.



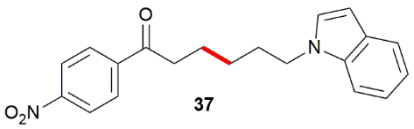
35

(*S*)-6,10-Dimethyl-1-(4-methoxyphenyl)undec-9-en-1-one (35): Isolated as an orange oil (37%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.93 (2H, d, J = 8.8 Hz), 6.92 (2H, d, J = 8.8 Hz), 5.09 (1H, t, J = 1.2 Hz), 3.89 (3H, s), 2.90 (2H, t, J = 7.4 Hz), 2.03-1.87 (2H, m), 1.74-1.69 (1H, m), 1.67 (3H, s), 1.59 (3H, s), 1.43-1.25 (6H, m), 1.19-1.08 (2H, m), 0.86 (3H, d, J = 6.48 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 199.2, 163.4, 131.0, 130.3, 125.1, 113.7, 55.5, 38.4, 37.2, 36.7, 32.3, 26.9, 25.8, 25.6, 25.0, 19.6, 17.7; ν_{max}/cm^{-1}: 2922, 2855, 1676, 1599, 1509, 1458, 1255, 1168, 1031, 833; HRMS (ESI+) calcd for $C_{20}H_{30}O_2$ (M+H) 303.23186, found 303.23162.



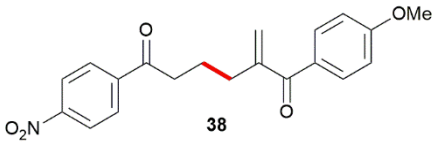
36

(*S*)-7,11-Dimethyldodec-10-en-2-one (36)[11]: Isolated as a yellow oil (26%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 5.08 (1H, t, J = 6.6 Hz), 2.41 (2H, t, J = 7.4 Hz), 2.12 (3H, s), 1.90-1.99 (2H, m), 1.67 (3H, s), 1.59 (3H, s), 1.53-1.55 (2H, m), 1.36-1.39 (1H, m), 1.23-1.32 (4H, m), 1.07-1.13 (2H, m), 0.85 (3H, d, J = 6.4 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 209.3, 131.0, 125.0, 43.8, 37.1, 36.7, 32.3, 29.8, 26.6, 25.7, 25.5, 24.2, 19.5, 17.6; ν_{max}/cm^{-1}: 2924, 2855, 1717, 1457, 1375, 1165; LRMS (APCI) m/z: 221.11 [M+H].



37

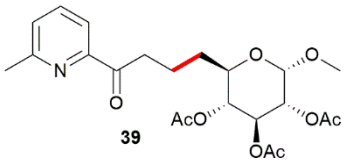
6-(1 *H*-Indol-1-yl)-1-(4-nitrophenyl)hexan-1-one (37): Isolated as a yellow solid (66%). m.p 121-123 ^{o}C ; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.27 (2H, d, J = 8.4 Hz), 8.02 (2H, d, J = 8.8 Hz), 7.64 (1H, d, J = 7.6 Hz), 7.34 (1H, d, J = 8.0 Hz), 7.20 (1H, t, J = 7.6 Hz), 7.10 (2H, t, J = 6.0 Hz), 6.49 (1H, d, J = 2.8 Hz), 4.15 (2H, t, J = 6.8 Hz), 2.94 (2H, t, J = 7.2 Hz), 1.91 (2H, p, J = 7.3 Hz), 1.78 (2H, p, J = 7.5 Hz), 1.40 (2H, p, J = 7.8 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 198.3, 150.3, 141.3, 135.9, 129.0, 128.6, 127.8, 123.8, 121.0, 119.3, 109.3, 101.1, 46.1, 38.8, 30.1, 26.5, 23.5; ν_{max}/cm^{-1}: 2945, 2853, 1688, 1519, 1339, 1318, 1189, 745, 729, 720, 703; HRMS (ESI+) calcd for $C_{20}H_{20}N_2O_3$ (M+H) 337.15467, found 337.15432.



38

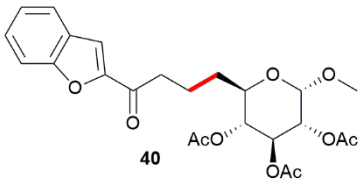
1-(4-methoxyphenyl)-2-methylene-6-(4-nitrophenyl)hexane-1,6-dione (38): Isolated as yellow oil (33%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 (2H, d, J = 8.7 Hz), 8.08 (2H, d, J = 8.7 Hz), 7.80 (2H, d, J = 8.8 Hz), 6.92 (2H, d, J = 8.8 Hz), 5.79 (1H, s), 5.57 (1H, s), 3.87 (3H, s), 3.08 (2H, t, J = 7.3 Hz), 2.56 (2H, t, J =

7.6 Hz), 1.98 (2H, p, *J* = 7.5 Hz); ^{13}C NMR (101 MHz; $CDCl_3$): 198.2, 196.9, 163.3, 150.3, 147.5, 141.4, 132.0, 130.0, 129.0, 123.9, 123.8, 113.6, 55.5, 38.5, 32.1, 22.6; ν_{max}/cm^{-1}: 2955, 2923, 2852, 1693, 1600, 1525, 1345, 1257, 1167; LRMS (APCI) m/z: 354.08 [M+H].



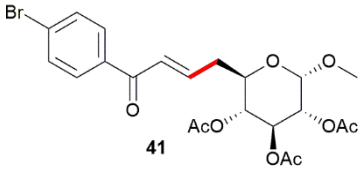
39

2-Methoxy-6-(4-(6-methylpyridin-2-yl)-4-oxobutyl) tetrahydro-2H-pyran-3,4,5-triyl triacetate (39): Isolated as an orange oil (62%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 (1H, d, *J* = 7.6 Hz), 7.69 (1H, t, *J* = 7.8 Hz), 7.31 (1H, d, *J* = 7.6 Hz), 5.44 (1H, t, *J* = 9.8 Hz), 4.84-4.90 (3H, m), 3.79-3.3.84 (1H, m), 3.41 (3H, s), 3.24 (2H, t, *J* = 7.4 Hz), 2.60 (3H, s), 2.07 (3H, s), 2.02 (3H, s), 2.00 (3H, s), 1.72-1.77 (2H, m), 1.59-1.64 (2H, m); ^{13}C NMR (101 MHz; $CDCl_3$): 201.9, 170.2, 170.1, 169.8, 157.9, 153.0, 136.9, 126.7, 118.7, 96.4, 72.4, 71.2, 70.3, 68.3, 55.3, 37.1, 30.7, 24.4, 20.7, 19.7; ν_{max}/cm^{-1}: 2945, 1746, 1695, 1367, 1217, 1032, 932; HRMS (ESI+) calcd for $C_{22}H_{29}N_1O_9$ (M+Na) 452.19151, found 452.19104.



40

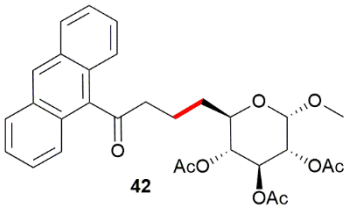
2-(4-(Benzofuran-2-yl)-4-oxobutyl)-6-methoxytetrahydro-2H-pyran-3,4,5-triyl triacetate (40): Isolated as yellow solid (45%). m.p 152-154 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.72 (1H, d, *J* = 7.6 Hz), 7.58 (1H, d, *J* = 8.4 Hz), 7.47-7.51 (2H, m), 7.28-7.34 (1H, m), 5.46 (1H, t, *J* = 9.8 Hz), 4.85-4.92 (3H, m), 3.79-3.85 (1H, m), 3.42 (3H, s), 2.98-2.3.03 (2H, m), 2.08 (3H, s), 2.04 (3H, s), 2.01 (3H, s), 1.79-1.87 (1H, m), 1.63-1.71 (2H, m), 1.54-1.61 (1H, m); ^{13}C NMR (101 MHz; $CDCl_3$): 190.8, 170.2, 170.1, 169.9, 155.6, 152.6, 128.2, 127.0, 123.9, 123.3, 112.5, 112.4, 96.5, 72.3, 71.2, 70.3, 68.3, 55.3, 38.5, 30.6, 20.7, 20.7, 19.8; ν_{max}/cm^{-1}: 2940, 1745, 1681, 1368, 1217, 1032, 931, 734; HRMS (ESI+) calcd for $C_{24}H_{28}O_{10}$ (M+Na) 499.15747, found 499.15703.



41

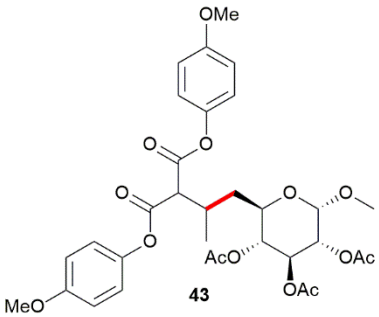
(*E* and *Z*)-2-(4-(4-Bromophenyl)-4-oxobut-2-en-1-yl)-6-methoxytetrahydro-2H-pyran-3,4,5-triyl triacetate (41): Isolated as orange oil (37%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.77 (4H, m, *E* and *Z*), 7.59 (4H, m, *E* and *Z*), 7.00-7.05 (1H, m, *E*), 6.89-6.92 (2 H, d, *J (E)* = 15 Hz, *J (Z)* = 10 Hz), 6.45 (1 H (*Z*)), dt, *J* = 11.6 , 7.0 Hz), 6.42-5.49 (2H, m, *E* and *Z*), 4.84-4.92 (6H, m, *E* and *Z*), 3.93-3.97 (2H, m, *E* and *Z*), 3.39 (3H, s, *Z*), 3.37 (3H, s, *E*), 2.91 (2H, m, *E*), 2.46-2.59 (2H, m, *Z*), 2.07 (6H, s), 2.02 (3H, s), 2.00 (6H, s), 1.99 (3H, s) ; ^{3}C NMR (101 MHz; $CDCl_3$): 190.31, 188.91, 170.19, 170.13, 170.05, 170.00, 169.79, 169.71, 143.82, 143.76, 137.07, 136.33, 131.92, 131.88, 130.07, 129.84, 128.06, 128.04, 127.98, 125.78, 96.68,

72.15, 71.54, 71.09, 70.97, 70.19, 70.05, 68.07, 67.57, 55.53, 55.32, 34.7, 30.72, 20.71, 20.69, 20.64; ν_{max}/cm^{-1}: 2989, 1740, 1434, 1369, 1210, 1091, 1040, 1010, 964, 601; HRMS (ESI+) calcd for $C_{22}H_{25}{}^{79}Br_1O_9$ (M+Na) 535.05742 and $C_{22}H_{25}{}^{81}Br_1O_9$ (M+Na) 537.05544, found 535.05695 and 537.05496.



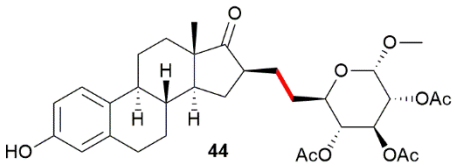
42

2-(4-(Anthracen-9-yl)-4-oxobutyl)-6-methoxytetrahydro-2H-pyran-3,4,5-triyl triacetate (42): Isolated as an orange oil (47%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.50 (1H, s), 8.04 (2H, m), 7.78 (2H, m), 7.52 (4H, m), 5.47 (1H, t, J = 9.6 Hz), 4.86-4.92 (3H, m), 3.81-3.85 (1H, m), 3.37 (3H, s), 3.10 (2H, t, J = 7.4 Hz), 2.09 (3H, s), 2.04 (3H, s), 2.02 (3H, s), 1.69-1.77 (2H, m), 1.60-1.67 (2H, m); ^{13}C NMR (101 MHz; $CDCl_3$): 209.8, 170.2, 170.1, 169.9, 136.5, 131.1, 128.9, 128.1, 126.9, 126.8, 125.5, 124.3, 96.5, 72.4, 71.2, 70.3, 68.4, 55.3, 45.9, 30.5, 29.7, 20.7, 19.7; ν_{max}/cm^{-1}: 2924, 2854, 1748, 1693, 1371, 1288, 1256, 1136, 733; HRMS (ESI+) calcd for $C_{38}H_{32}O_9$ (M+Na) 559.19385, found 559.19316.



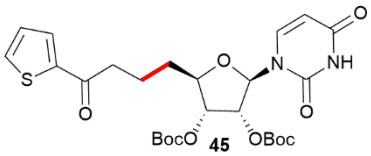
43

Bis(4-methoxyphenyl) 2-(1-(3,4,5-triacetoxy-6-methoxytetrahydro-2H-pyran-2-yl)propan-2-yl)malonate (43): Isolated as an orange oil (27%).(dr 1:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.03 (8H, dd, J_1 = 3.2 Hz, J_2 = 9.2 Hz), 6.89 (8H, d, J = 8.8 Hz), 5.45 (2H, t, J = 9.6 Hz), 4.92 (2H, d, J = 3.6 Hz), 4.83-4.89 (4H, m), 3.95 (2H, m), 3.80 (6H, s), 3.46 (3H, s), 3.44 (3H, s), 2.74-2.77 (1H, m), 2.63-2.74 (1H, m), 2.07 (6H, s), 2.07 (3H, s), 2.01 (6H, s), 2.00 (3H, s), 1.80 (-1.94 (2H, m), 1.59-1.70 (2H, m), 1.22-1.29 (6H, m); ^{13}C NMR (101 MHz; $CDCl_3$): 170.19, 170.15, 170.10, 170.03, 169.95, 169.80, 167.41, 167.27, 167.09, 167.02, 157.61, 157.59, 143.93, 143.89, 143.84, 122.04, 122.02, 114.59, 96.86, 96.76, 72.63, 72.57, 71.05, 70.19, 70.06, 67.58, 66.35, 57.06, 55.90, 55.82, 55.78, 55.61, 35.85, 35.78, 311.02, 29.88, 20.71-20.61 (6C), 18.28, 16.57; ν_{max}/cm^{-1}: 2932, 2843, 1749, 1506, 1246, 1224, 1194, 1036 ; HRMS (ESI+) calcd for $C_{32}H_{38}O_{14}$ (M+Na) 669.21538, found 669.21455.



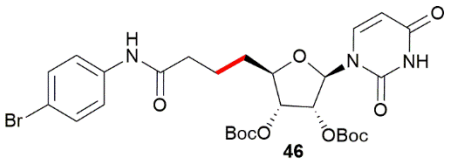
44

2-(2-(3-Estronyl)ethyl)-6-methoxytetrahydro-2H-pyran-3,4,5-triyl triacetate (44): Isolated as a yellow oil (35%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.12 (1H, d, J = 8.4 Hz), 6.62 (1H, d, J = 8.4 Hz), 6.58 (1H, s), 5.43 (1H, t, J = 9.6 Hz), 4.81-4.88 (3H, m), 3.73 (1H, t, J = 9.2 Hz), 3.63 (3H, s), 2.84-2.86 (2H, m), 2.21-2.35 (4H, m), 2.06 (3H, s), 2.04 (3H, s), 2.00 (3H, s), 188-2.01 (2H, m), 1.41-1.61 (8H, m), 1.19-1.33 (4H, m), 0.86 (3H, s); ^{13}C NMR (101 MHz; $CDCl_3$): 222.31, 170.3, 170.1, 170.0, 153.8, 137.9, 131.9, 126.4, 115.3, 112.9, 96.5, 72.4, 71.2, 68.1, 55.3, 49.0, 48.9, 48.5, 44.0, 37.9, 31.9, 29.9, 28.6, 28.3, 26.7, 25.8, 20.8, 20.7, 20.6, 14.1; ν_{max}/cm^{-1}: 2925, 2857, 1748, 1500, 1453, 1369, 1220, 1037, 733; HRMS (ESI+) calcd for $C_{32}H_{42}O_{10}$ (M+Na) 609.26702, found 609.26617.



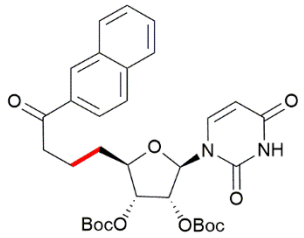
45

Di-tert-butyl (2-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-5-(4-oxo-4-(thiophen-2-yl)butyl)tetrahydrofuran-3,4-diyl) bis(carbonate) (45): Isolated as a yellow oil (56%).^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.1 (1H, s), 7.71 (1H, d, J = 3.7 Hz), 7.62 (1H, d, J = 5.8 Hz), 7.27 (1H, d, J = 8.1 Hz), 7.09-7.11 (1H, m), 5.83 (1H, d, J = 4.4 Hz), 5.74-5.77 (1H, m), 5.52 (1H, t, J = 5.1 Hz), 4.97 (1H, t, J = 5.9 Hz), 4.11-4.16 (1H, m), 2.93-2.99 (2H, m), 1.78-1.97 (4H, m), 1.47 (9H, s), 1.45 (9H, s); ^{13}C NMR (101 MHz; $CDCl_3$): 192.4, 163.0, 152.2, 152.1, 149.8, 144.2, 140.5, 133.5, 131.8, 128.1, 103.1, 89.2, 83.6, 83.4, 80.1, 75.2, 75.1, 38.4, 32.3, 27.7, 27.6, 20.4; ν_{max}/cm^{-1}: 2980, 2927, 1746, 1689, 1370, 1251, 1133, 848, 732; HRMS (ESI+) calcd for $C_{26}H_{34}N_2O_{10}$ (M+Na) 589.18264, found 589.18206.



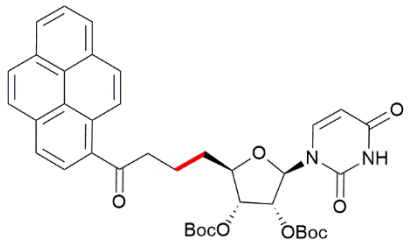
46

2-(4-((4-Bromophenyl)amino)-4-oxobutyl)-5-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)tetrahydrofuran-3,4-diyl di-tert-butyl bis(carbonate) (46): Isolates as a white solid (41%); m.p 152-154 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 11.44 (1H, brs), 10.00 (1H, brs), 7.75 (1H, d, J = 8.1 Hz), 7.55-7.61 (2H, m), 7.42-7.47 (2H, m), 5.76-5.80 (1H, m), 5.63-5.69 (1H, m), 5.31-5.36 (1H, m), 5.06-5.10 (1H, m), 3.98-3.99 (1H, m), 2.30-2.36 (2H, m), 1.64-1.80 (4H, m), 1.38-1.44 (2H, m), 1.41 (9H, s), 1.40 (9H, s); ^{13}C NMR (101 MHz; DMSO-d_6): 171.5, 171.2, 163.5, 152.2, 152.0, 150.7, 142.7, 139.1, 138.9, 131.9, 131.8, 121.5, 115.3, 115.0, 102.8, 88.7, 83.2,83.0, 82.9, 80.4, 75.0, 74.9,42.6, 36.3, 32.0, 27.7, 21.4; ν_{max}/cm^{-1}: 2982, 2933, 1747, 1682, 1531, 1489, 1371, 1287, 1251, 1135, 1073, 908, 822, 728; HRMS (ESI+) calcd for $C_{28}H_{36}{}^{79}Br_1N_3O_{10}$ (M+Na) 676.14763 and $C_{28}H_{36}{}^{81}Br_1N_3O_{10}$ (M+Na) 678.14611, found 676.14716 and 678.14523.



47

Di-tert-butyl (2-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-5-(4-(naphthalen-2-yl)-4-oxobutyl)tetrahydrofuran-3,4-diyl) bis(carbonate) (47): Isolated as a yellow oil (52%).^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.93 (1H, brs), 8.44 (1H, s), 8.00 (1H, d, J = 8.8 Hz), 7.94 (1H, d, J = 8.2 Hz), 7.80-7.88 (2H, m), 7.50-7.60 (2H, m), 7.27 (1H, d, J = 8.8 Hz), 5.85 (1H, d, J = 4.3 Hz), 5.73 (1H, d, J = 8.3 Hz), 5.28 (1H, t, J = 5.0 Hz), 5.00 (1H, t, J = 5.8 Hz), 4.18-4.20 (1H, m), 3.14-3.19 (2H, m), 1.85-1.90 (4H, m), 1.48 (9H, s), 1.46 (9H, s); ^{13}C NMR (101 MHz; $CDCl_3$): 199.4, 162.8, 152.3, 152.2, 149.8, 140.5, 135.6, 134.2, 132.5, 129.6, 129.5, 128.5, 127.8, 127.7, 126.8, 123.8, 103.0, 89.1, 83.6, 83.4, 80.8, 75.2, 75.1, 37.7, 32.4, 27.7, 27.6, 20.2; ν_{max}/cm^{-1}: 2980, 2935, 1747, 1680, 1458, 1370, 1254, 1133, 909, 728; HRMS (ESI+) calcd for $C_{32}H_{38}N_2O_{10}$ (M+Na) 633.24187, found 633.24153.



48

Di-tert-butyl (2-(4-(4,5a1-dihydropyren-1-yl)-4-oxobutyl)-5-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)tetrahydrofuran-3,4-diyl) bis(carbonate) (48): Isolated as a yellow solid (57%). m.p 86-88 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.92 (1H, d, J = 9.4 Hz), 8.81 (1H, brs), 8.31 (1H, d, J = 8.0 Hz), 8.24-8.8.27 (3H, m), 8.16-8.8.22 (3H, m), 8.05-8.1 (2H, m), 7.22 (1H, d, J = 8.1 Hz), 5.84 (1H, d, J = 4.4 Hz), 5.66 (1H, d, J = 7.9 Hz), 5.32 (1H, t, J = 5.0 Hz), 5.06 (1H, t, J = 5.9 Hz), 4.21-4.25 (1H, m), 3.27-3.32 (2H, m), 1.93-2.02 (4H, m), 1.52 (9H, s), 1.45 (9H, s); ^{13}C NMR (101 MHz; $CDCl_3$): 204.0, 162.7, 152.3, 152.2, 149.7, 140.5, 133.8, 132.3, 131.1, 130.5, 129.7, 129.5, 129.3, 127.1, 126.4, 126.3, 126.1, 126.0, 125.0, 124.7, 124.3, 124.0, 102.9, 89.3, 83.6, 83.4, 80.9, 75.2, 75.1, 41.6, 32.5, 27.7, 27.6, 20.8; ν_{max}/cm^{-1}: 2978, 2930, 1746, 1687, 1370, 1456, 1252, 1158, 1132, 845; HRMS (ESI+) calcd for $C_{38}H_{40}N_2O_{10}$ (M+Na) 707.25752, found 707.25764.

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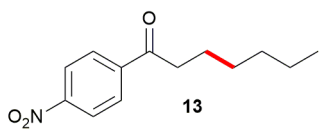
1-(4-nitrophenyl)heptan-1-one



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8.080

3.018
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1.341
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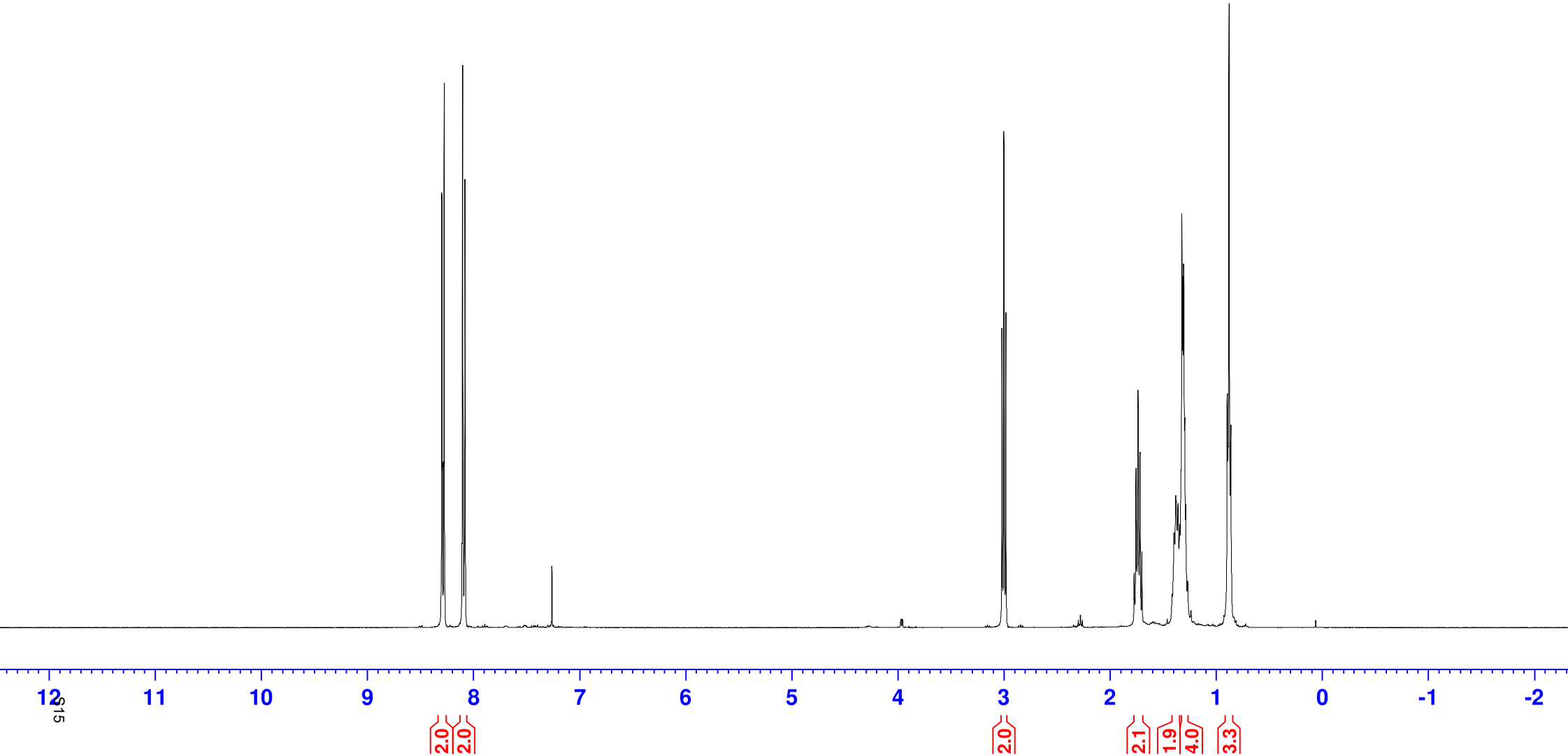
0.859



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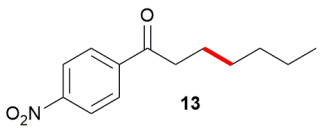
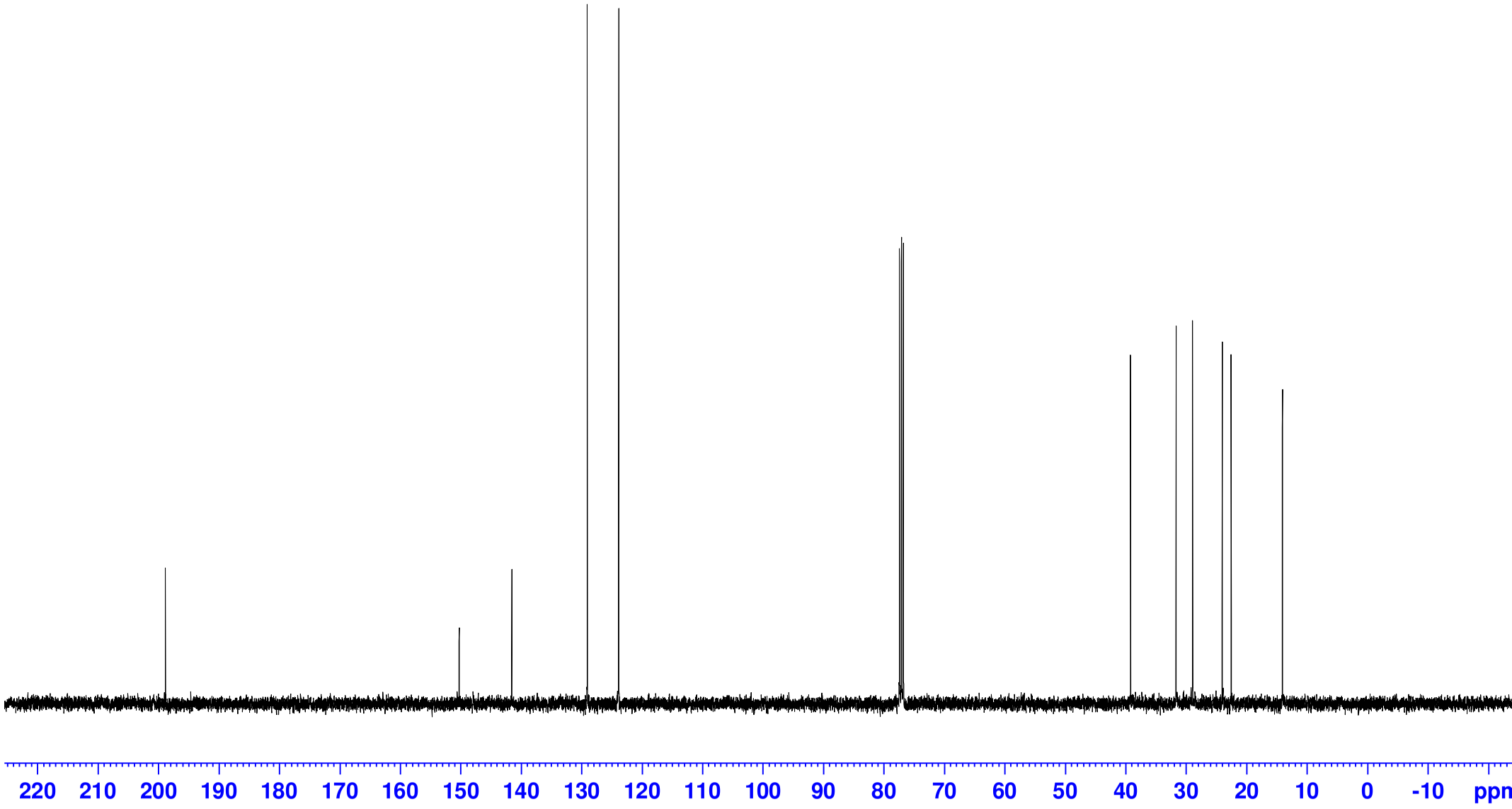
1-(4-nitrophenyl)heptan-1-one



198.87 150.26 141.54 129.06 123.86 39.19 31.63 28.92 24.00 22.52 14.04



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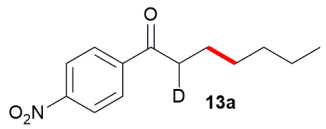

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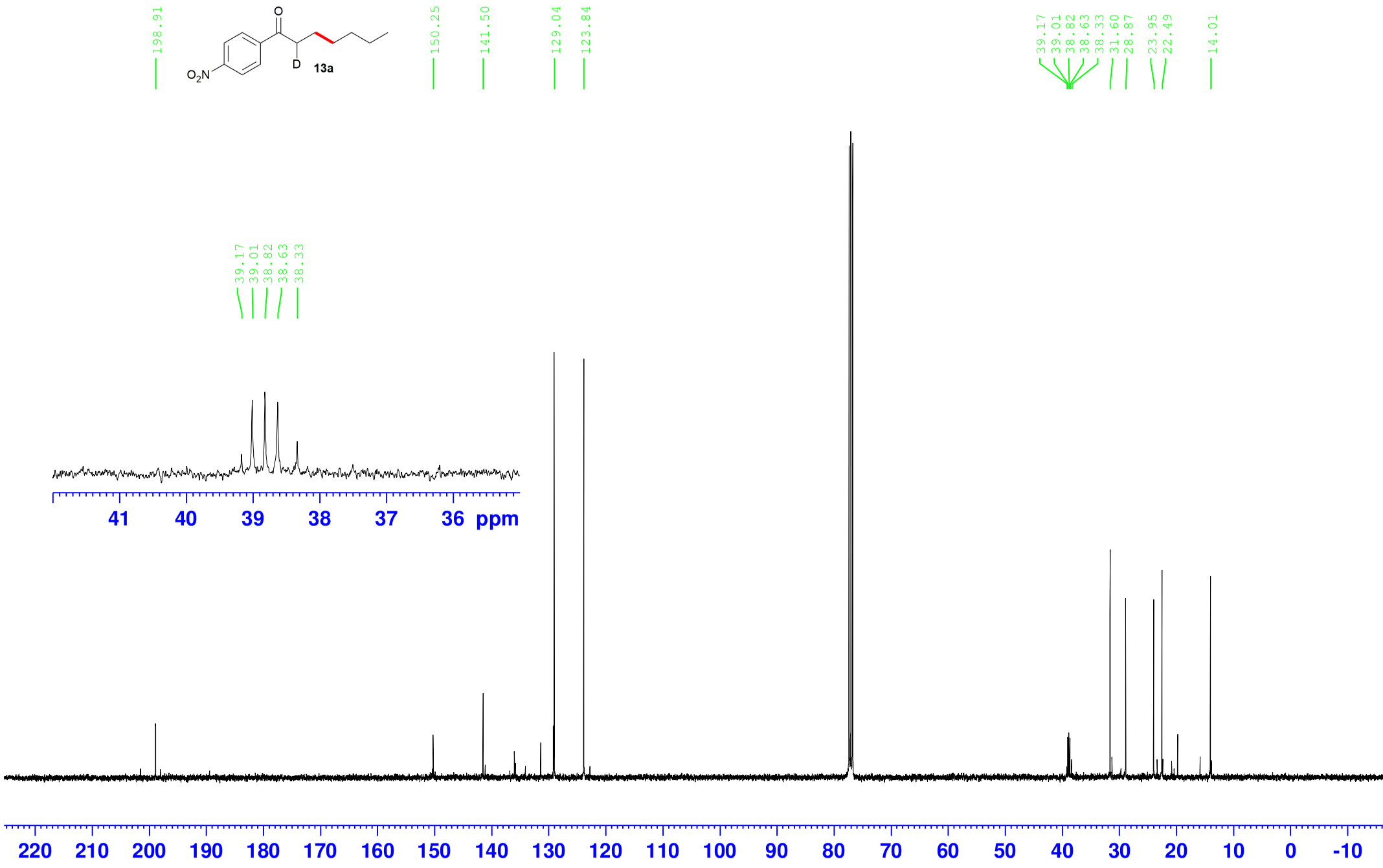

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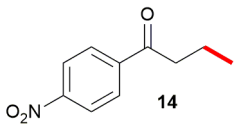

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8.086

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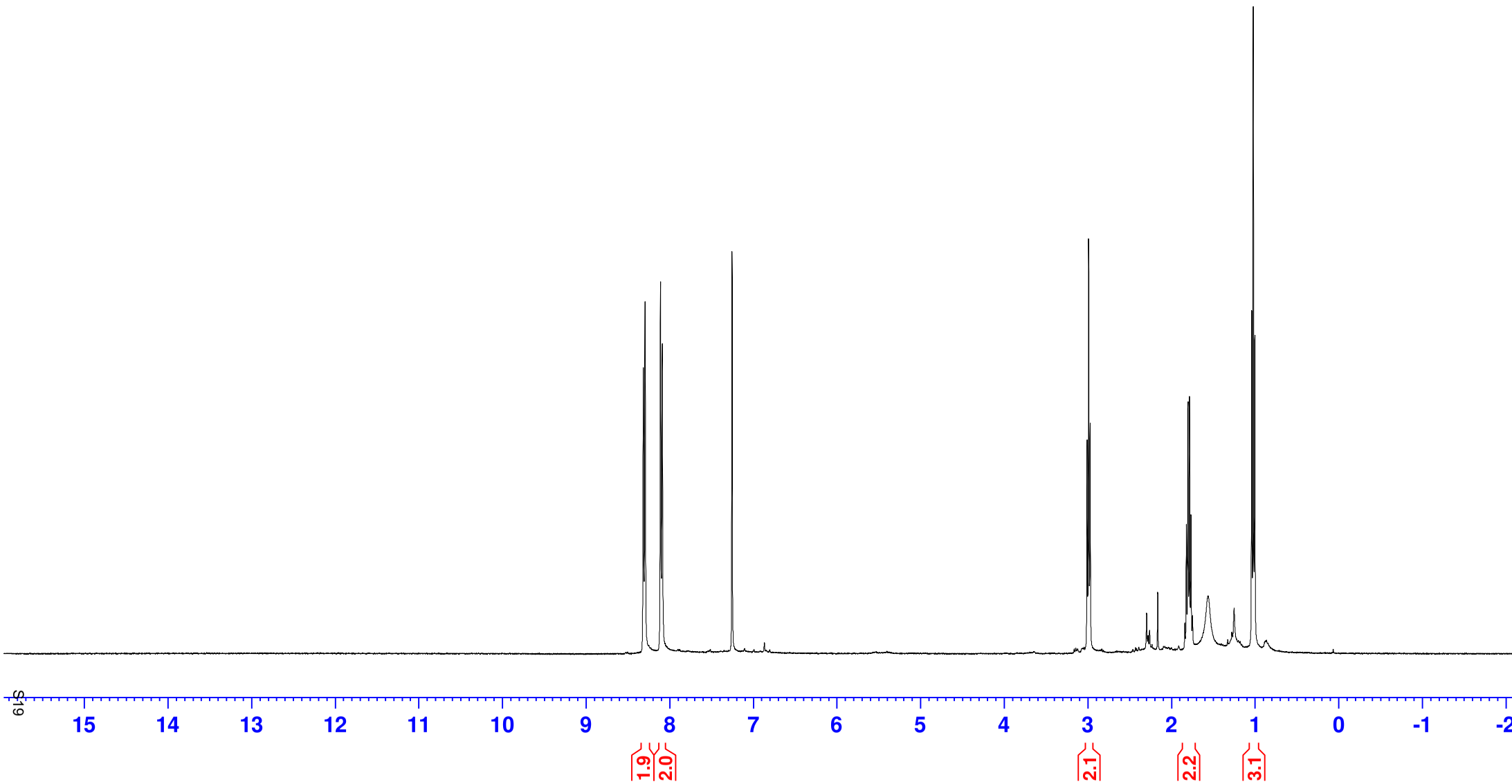
1.018
1.000



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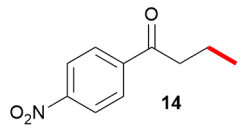

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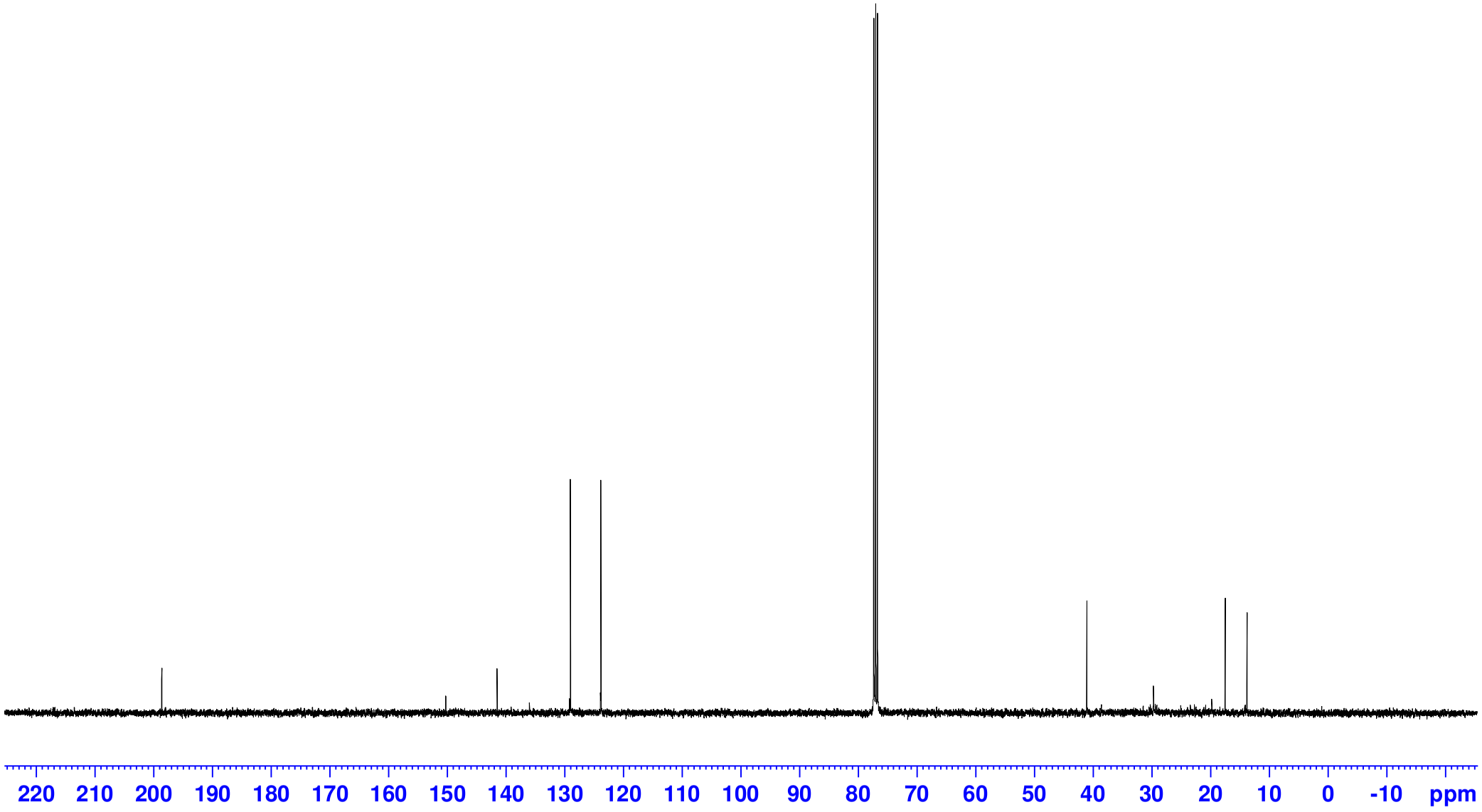

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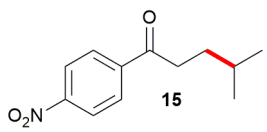

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1.626
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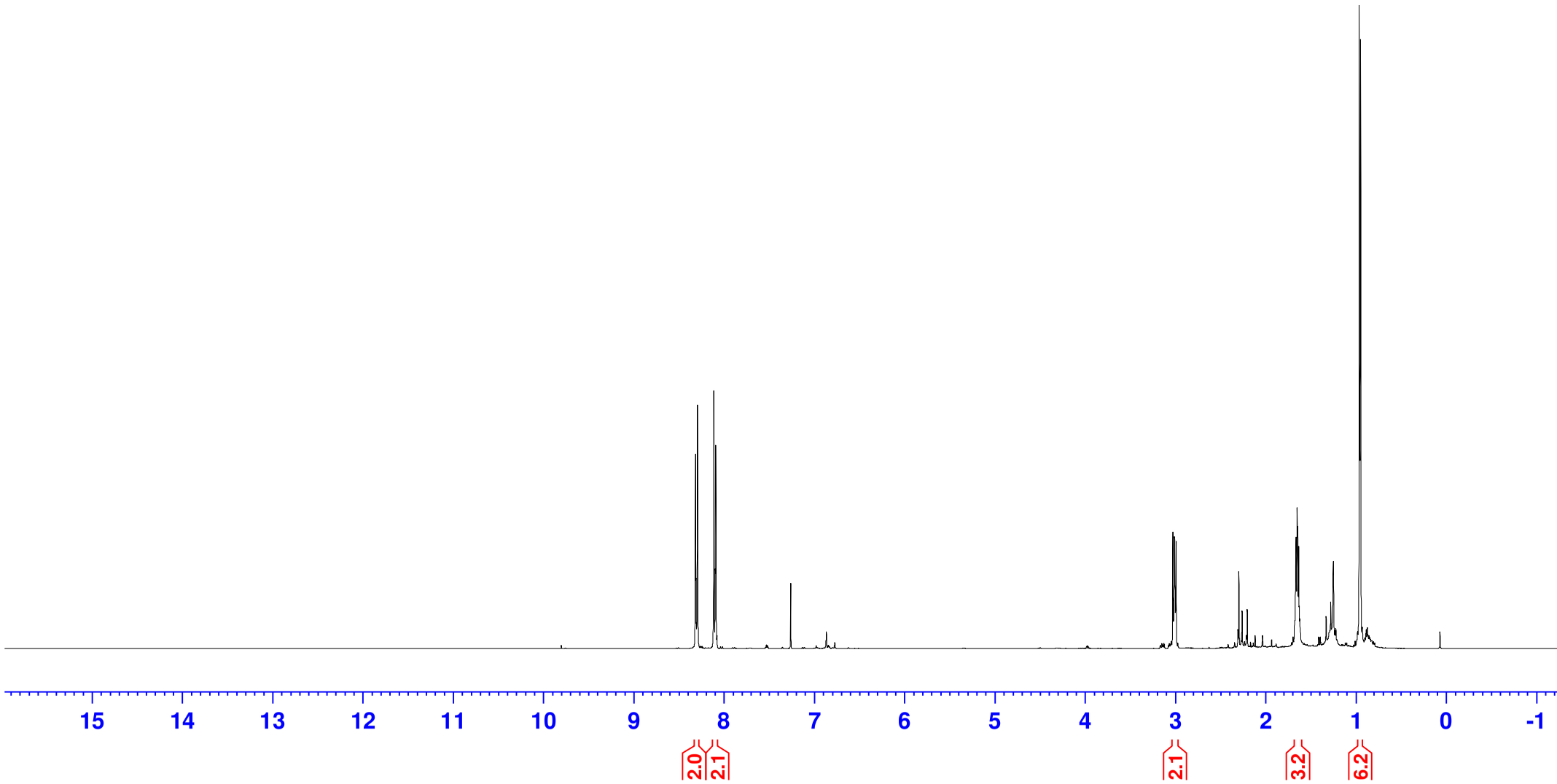
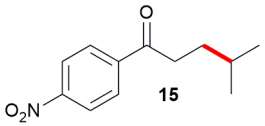
0.962
0.946



4-methyl-1-(4-nitrophenyl)pentan-1-one



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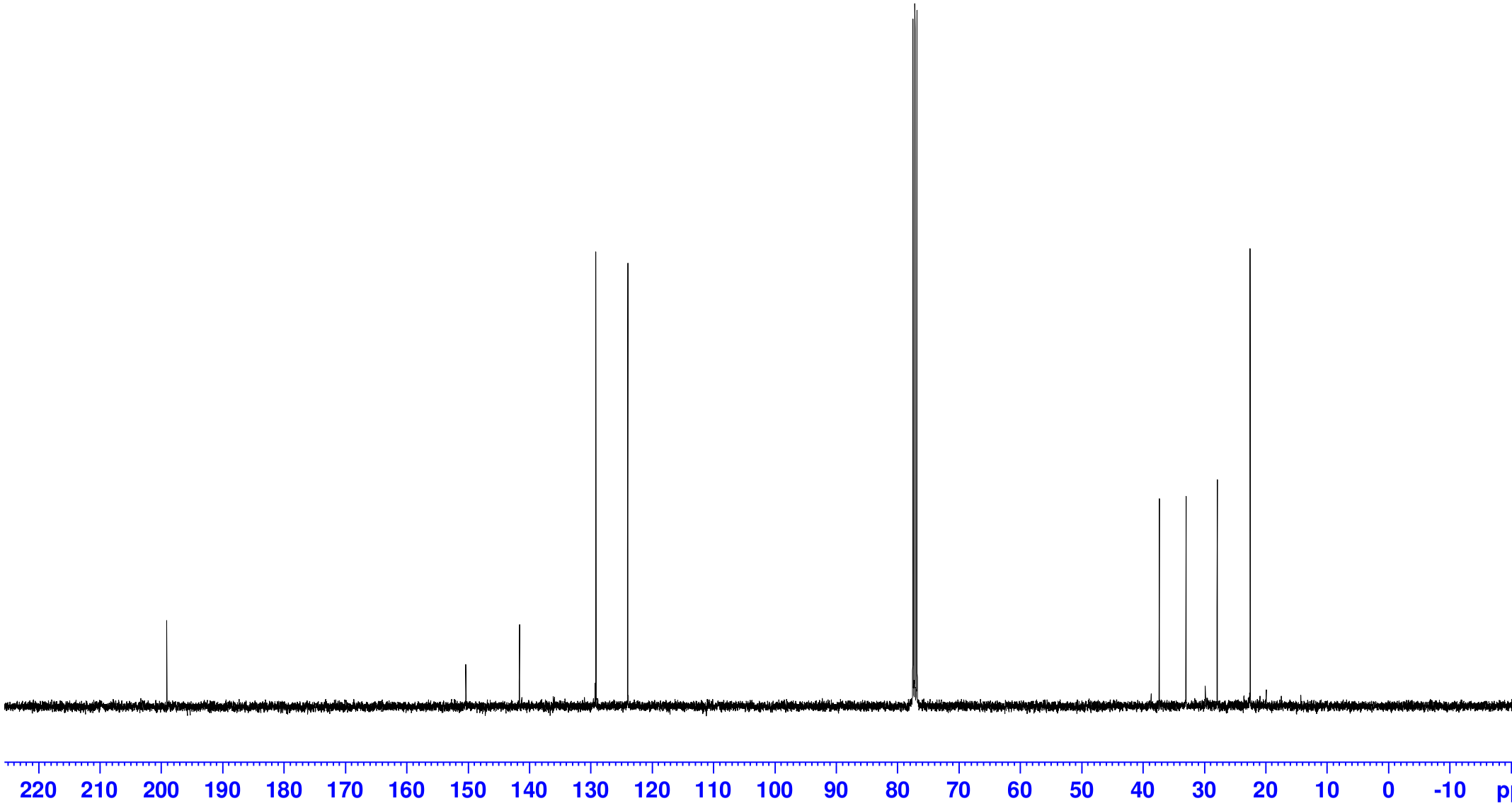
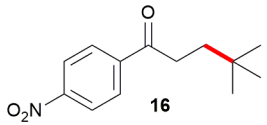
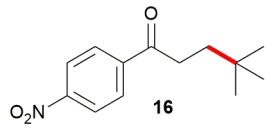
22.50



4,4-dimethyl-1-(4-nitrophenyl)pentan-1-one



4,4-dimethyl-1-(4-nitrophenyl)pentan-1-one



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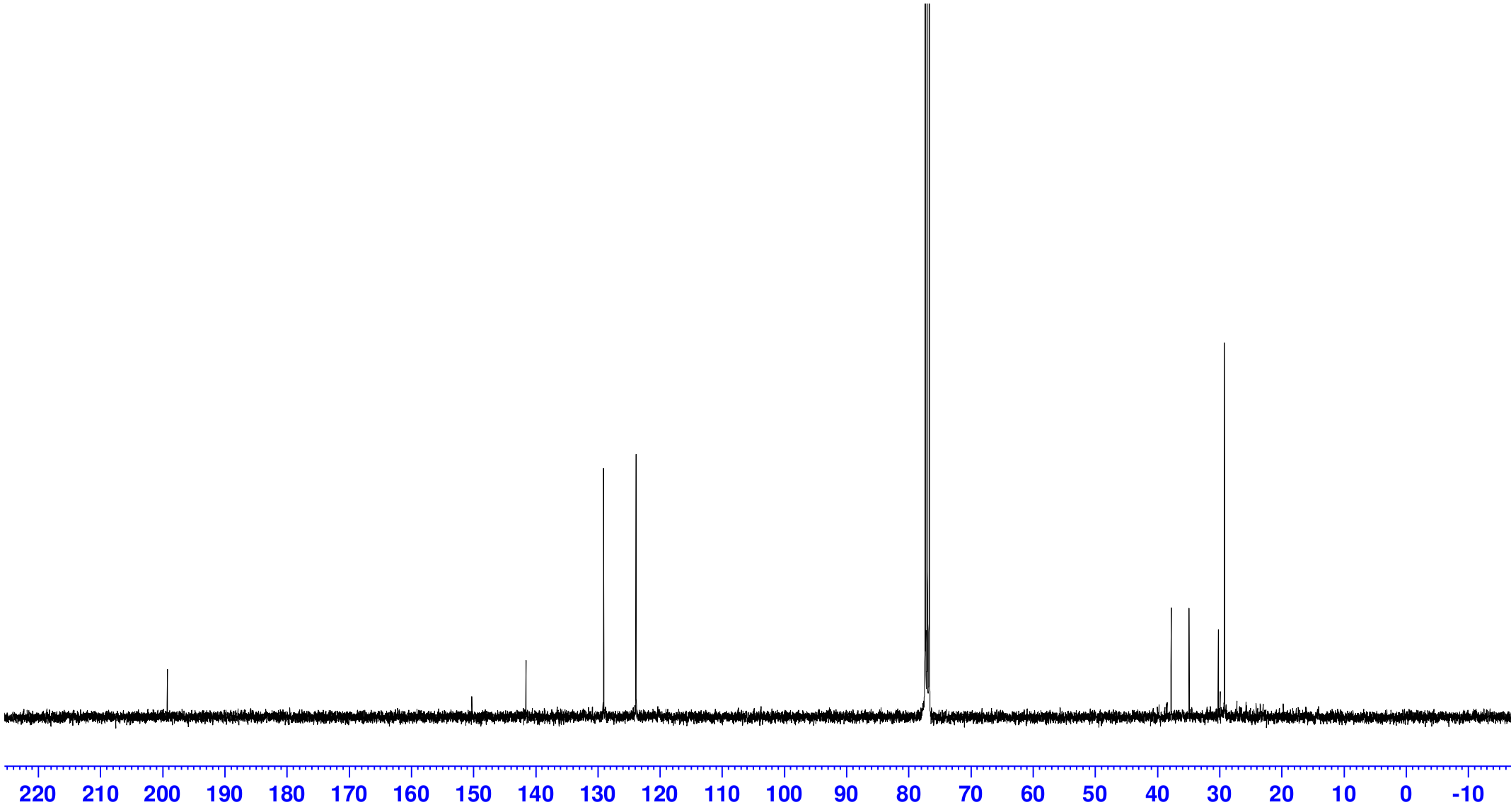
129.05
129.00
123.85
37.76
34.87
30.17
29.18



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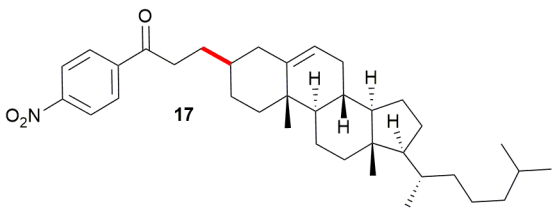
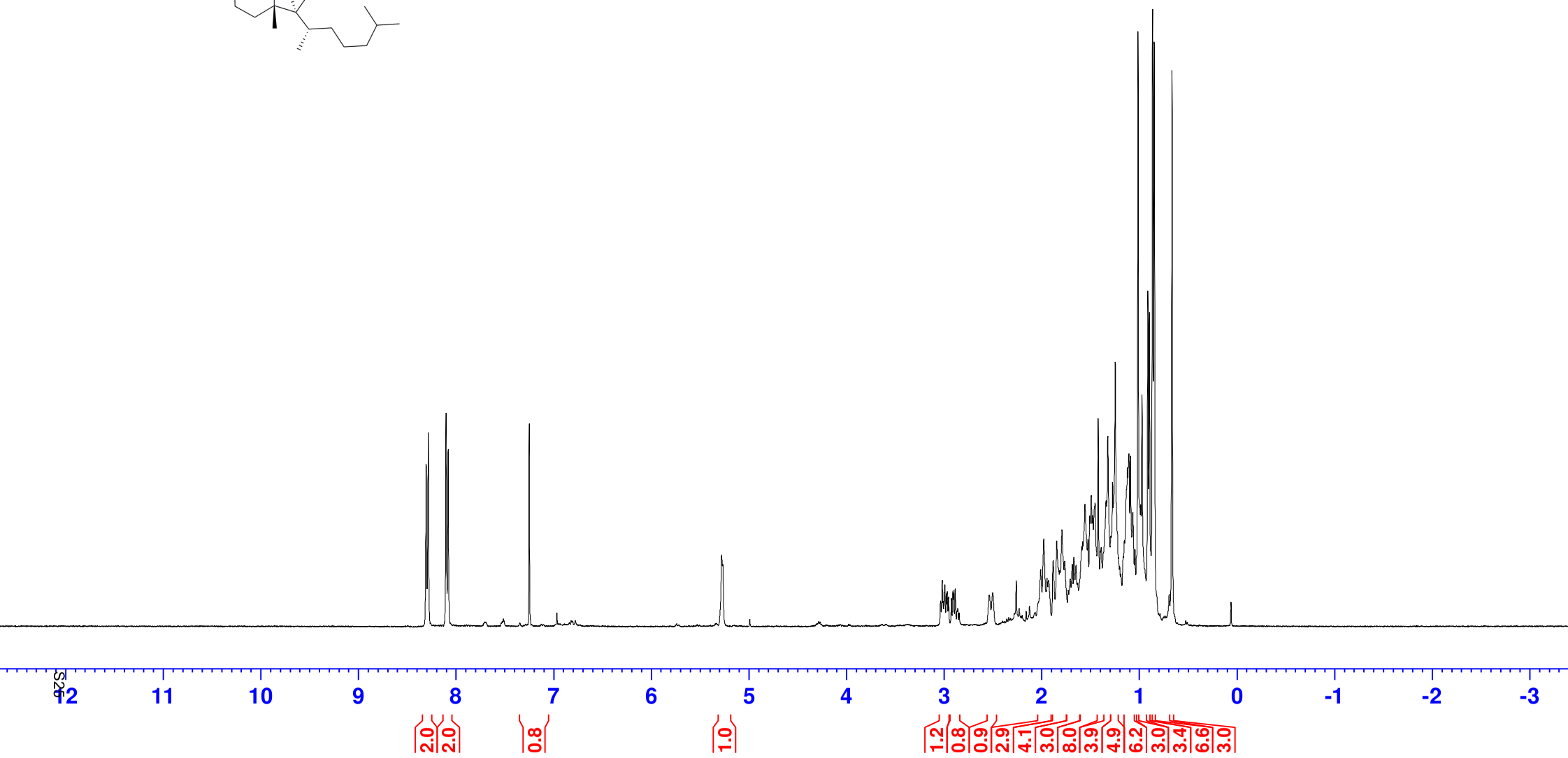
-1-(4-nitrophenyl)propan-1-one

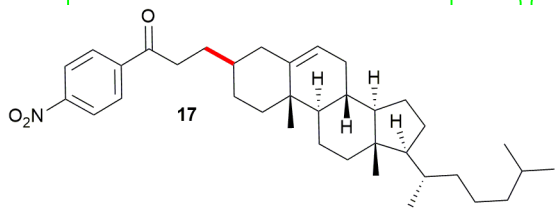


8.31 8.29 8.10 8.08

5.28 5.27 3.04 3.02 3.01 2.99 2.98 2.97 2.96 2.93 2.91 2.90 2.89 2.87 2.86 2.85 2.54 2.50 2.01 1.98 1.95 1.93 1.88 1.85 1.82 1.79 1.77 1.71 1.69 1.67 1.65 1.59 1.58 1.56 1.53 1.51 1.49 1.48 1.45 1.34 1.32 1.29 1.28 1.25 1.15 1.12 1.11 1.09 1.07 1.01 0.91 0.90 0.86 0.85 0.67



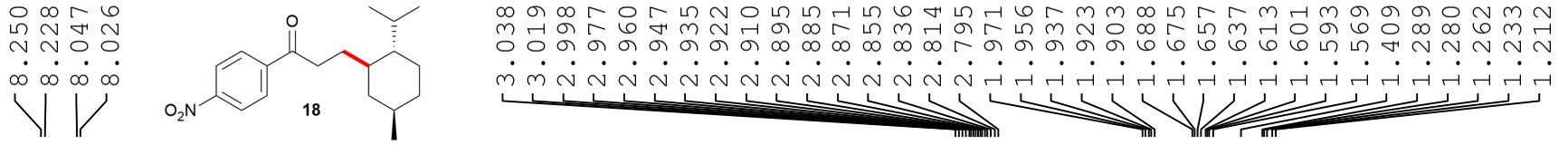
3-(10,13-dimethyl-17-(6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydrc
-1H-cyclopenta[a]phenanthren-3-yl)-1-(4-nitrophenyl)propan-1-one

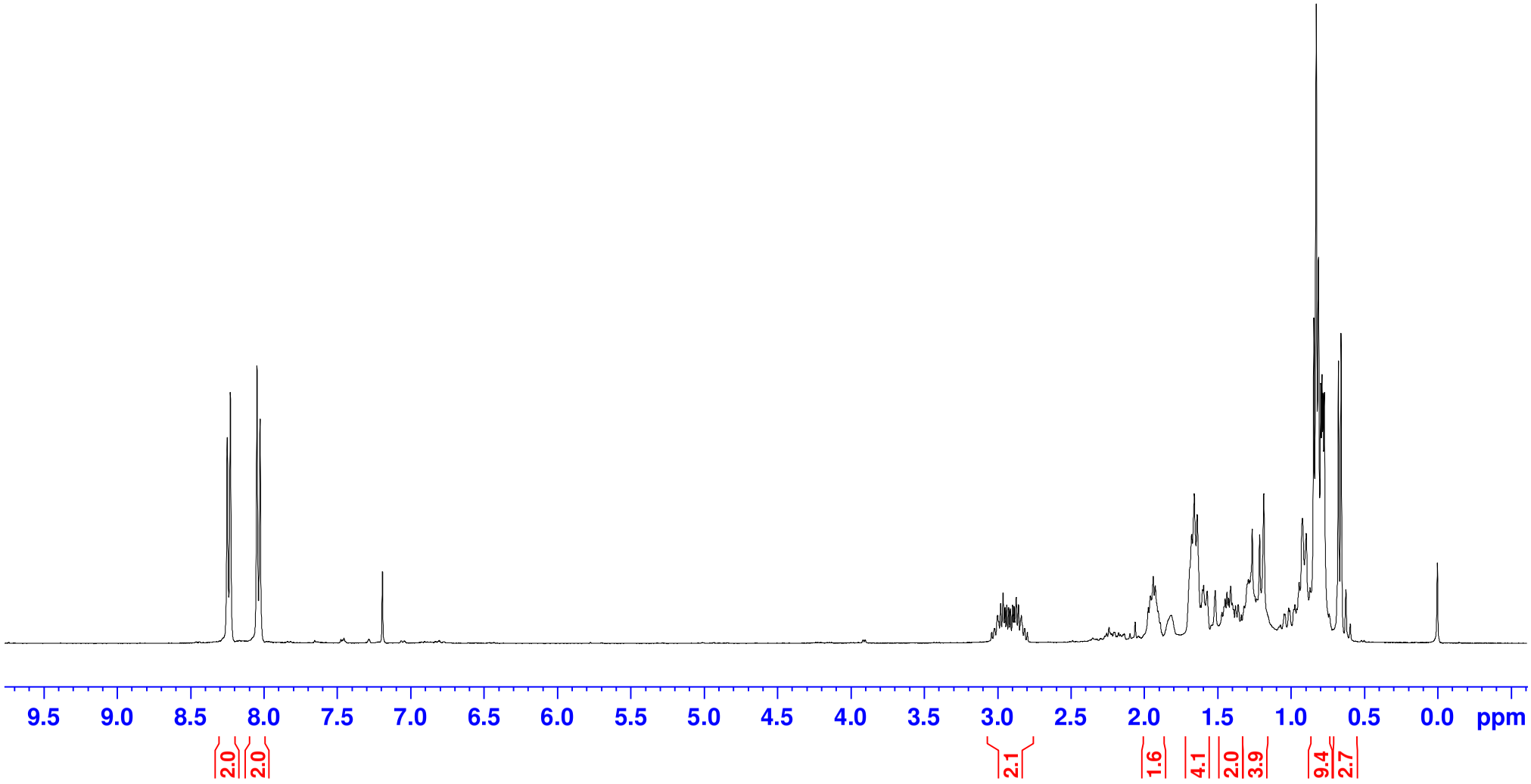


199.13
150.27
141.46
140.15
129.09
123.83
121.65

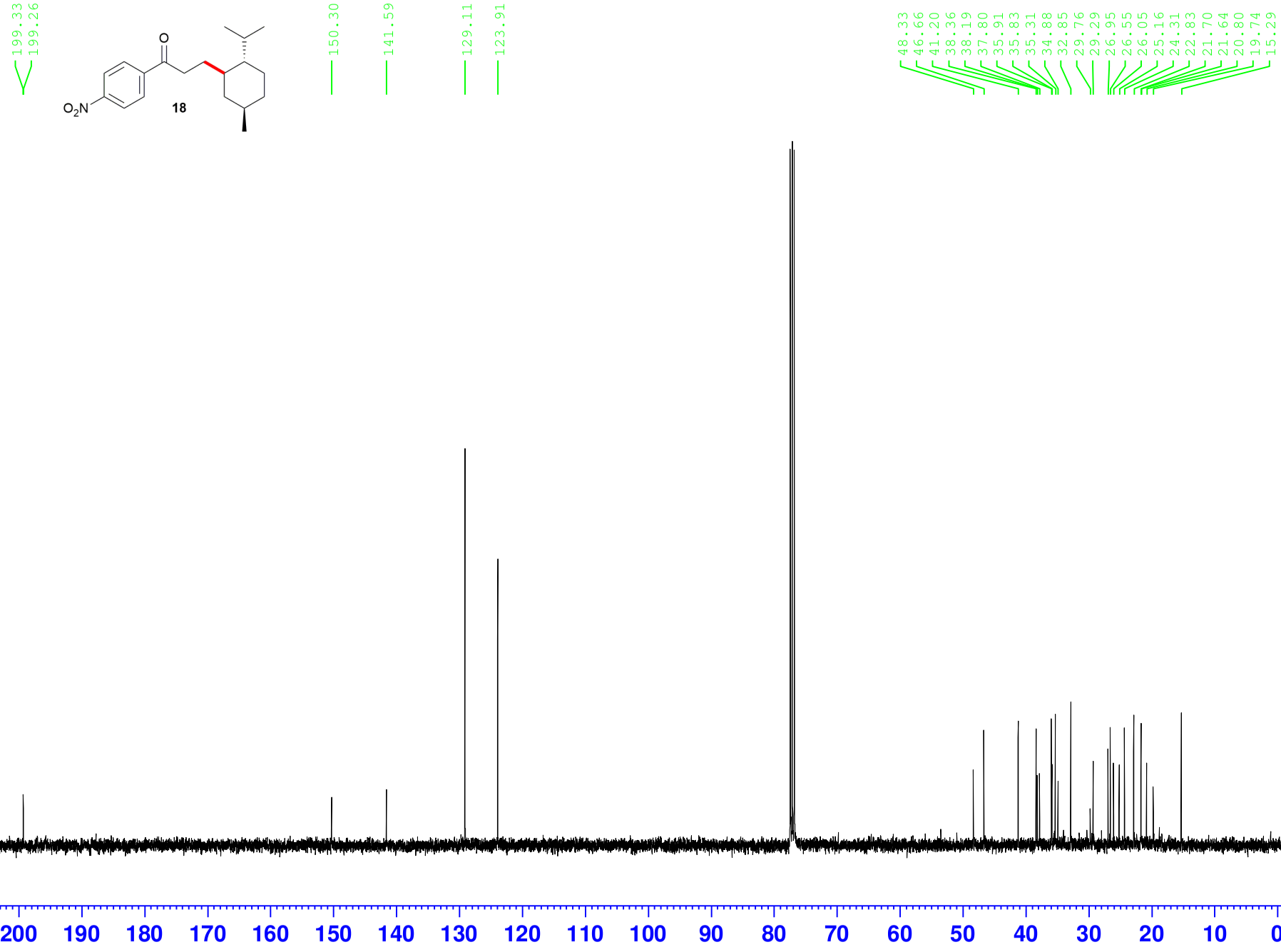
56.83
56.17
50.48
42.32
39.82
39.53
37.49
37.47
36.67
36.21
35.81
34.13
33.97
31.94
31.91
28.23
28.01
26.30
25.39
24.28
23.85
22.81
22.55
20.76
19.43
18.72
11.86

3-(2-isopropyl-5-methylcyclohexyl)-1-(4-nitrophenyl)propan-1-one

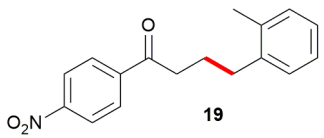




3-(2-isopropyl-5-methylcyclohexyl)-1-(4-nitrophenyl)propan-1-one



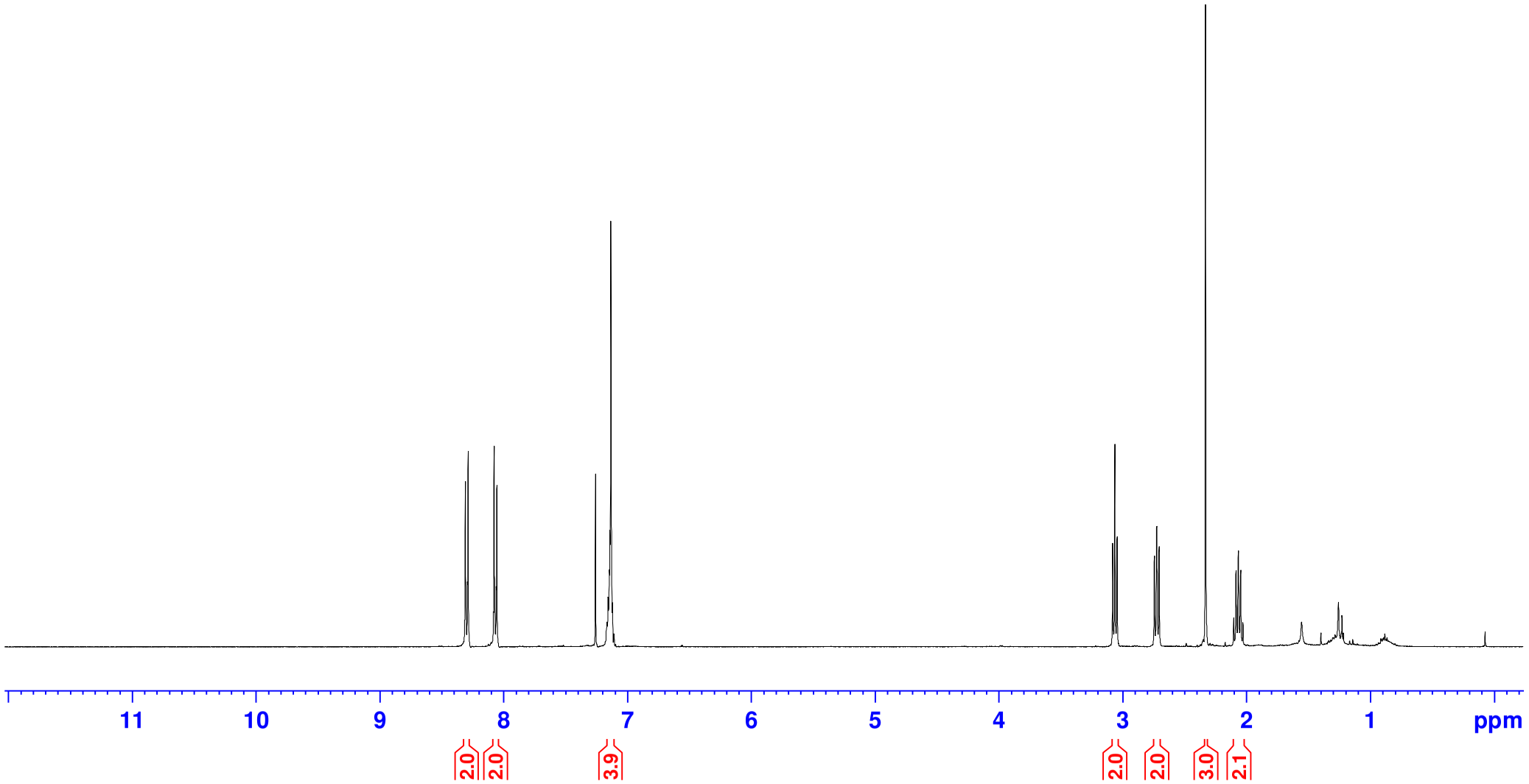
1-(4-nitrophenyl)-4-(o-tolyl)butan-1-one



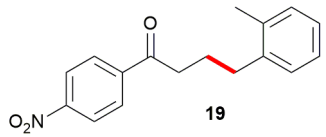
19

8.309
8.287
8.077
8.055
7.156
7.146
7.140
7.134
7.120

3.079
3.062
3.044
2.743
2.724
2.704
2.330
2.101
2.083
2.064
2.045
2.027



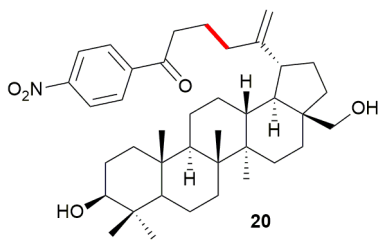
1-(4-nitrophenyl)-4-(o-tolyl)butan-1-one



19

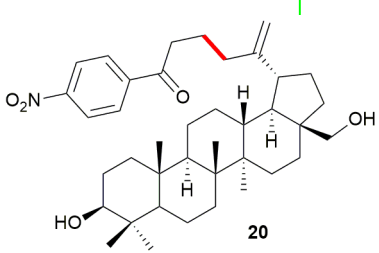
198.42
150.36
141.45
139.51
136.09
130.45
129.06
129.01
126.35
126.08
123.92
38.50
32.47
24.15
19.35

5-(9-hydroxy-3a-(hydroxymethyl)-5a,5b,8,8,11a-pentamethylicosahydro-1H-cyclopent
-1-(4-nitrophenyl)hex-5-en-1-one



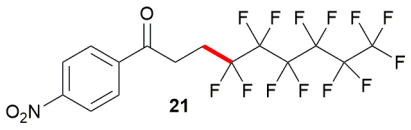
20

5-(9-hydroxy-3a-(hydroxymethyl)-5a,5b,8,8,11a-pentamethylicosahydro-1H-cyclopenta[a]chrysen-1-yl)
-1-(4-nitrophenyl)hex-5-en-1-one

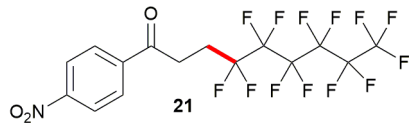


20

4,4,5,5,6,6,7,7,8,8,9,9,9-tridecafluoro-1-(4-nitrophenyl)nonan-1-one

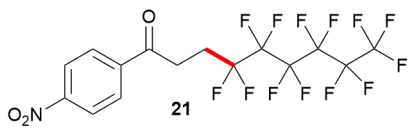


4,4,5,5,6,6,7,7,8,8,9,9,9-tridecafluoro-1-(4-nitrophenyl)nonan-1-one



21

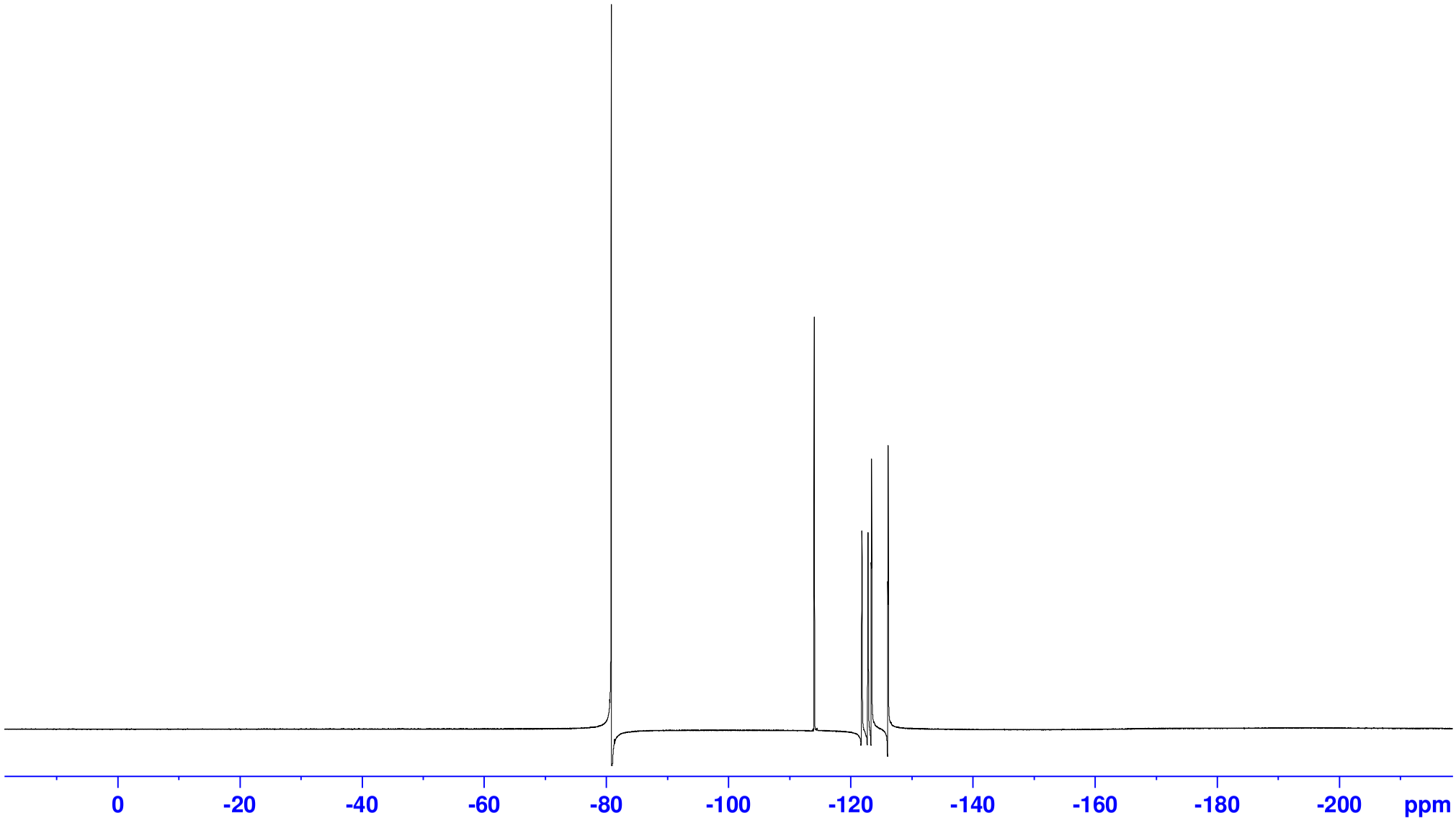
4,4,5,5,6,6,7,7,8,8,9,9,9-tridecafluoro-1-(4-nitrophenyl)nonan-1-one



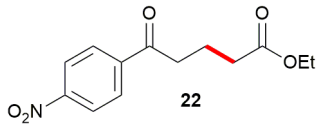
21

-80.78
-80.81
-80.84

-113.99
-114.00
-114.03
-114.06
-121.82
-121.85
-122.84
-123.36
-123.40
-123.41
-123.43
-126.08
-126.10
-126.12
-126.14
-126.16



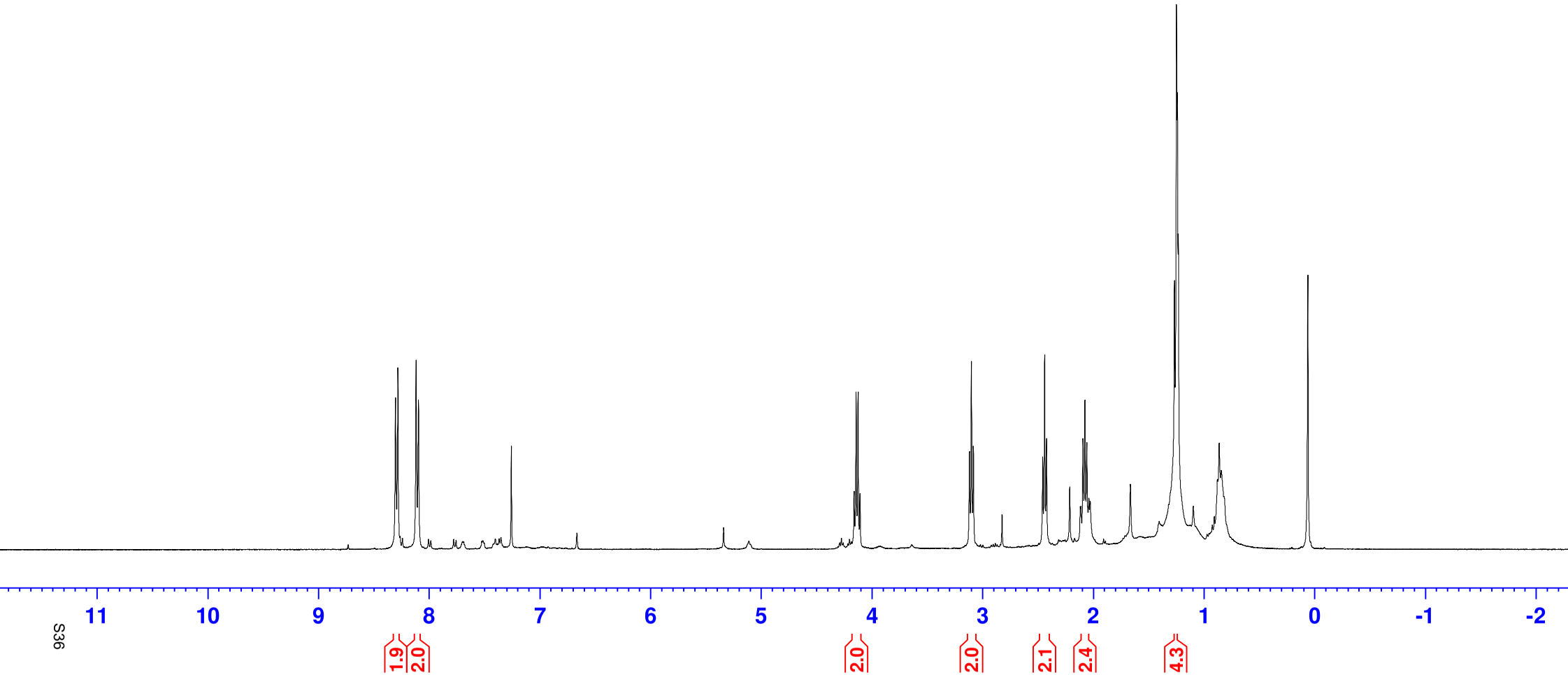
ethyl 5-(4-nitrophenyl)-5-oxopentanoate



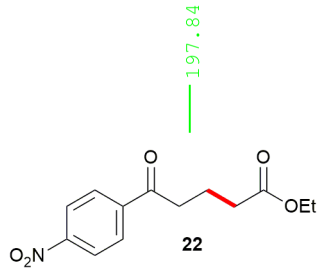
22

8.305
8.284
8.119
8.097

4.162
4.144
4.126
4.108
3.120
3.102
3.084
2.458
2.441
2.423
2.094
2.077
2.059
2.041
2.030
1.268
1.250
1.242
1.233

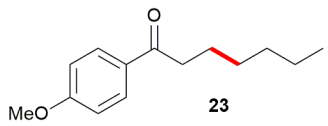


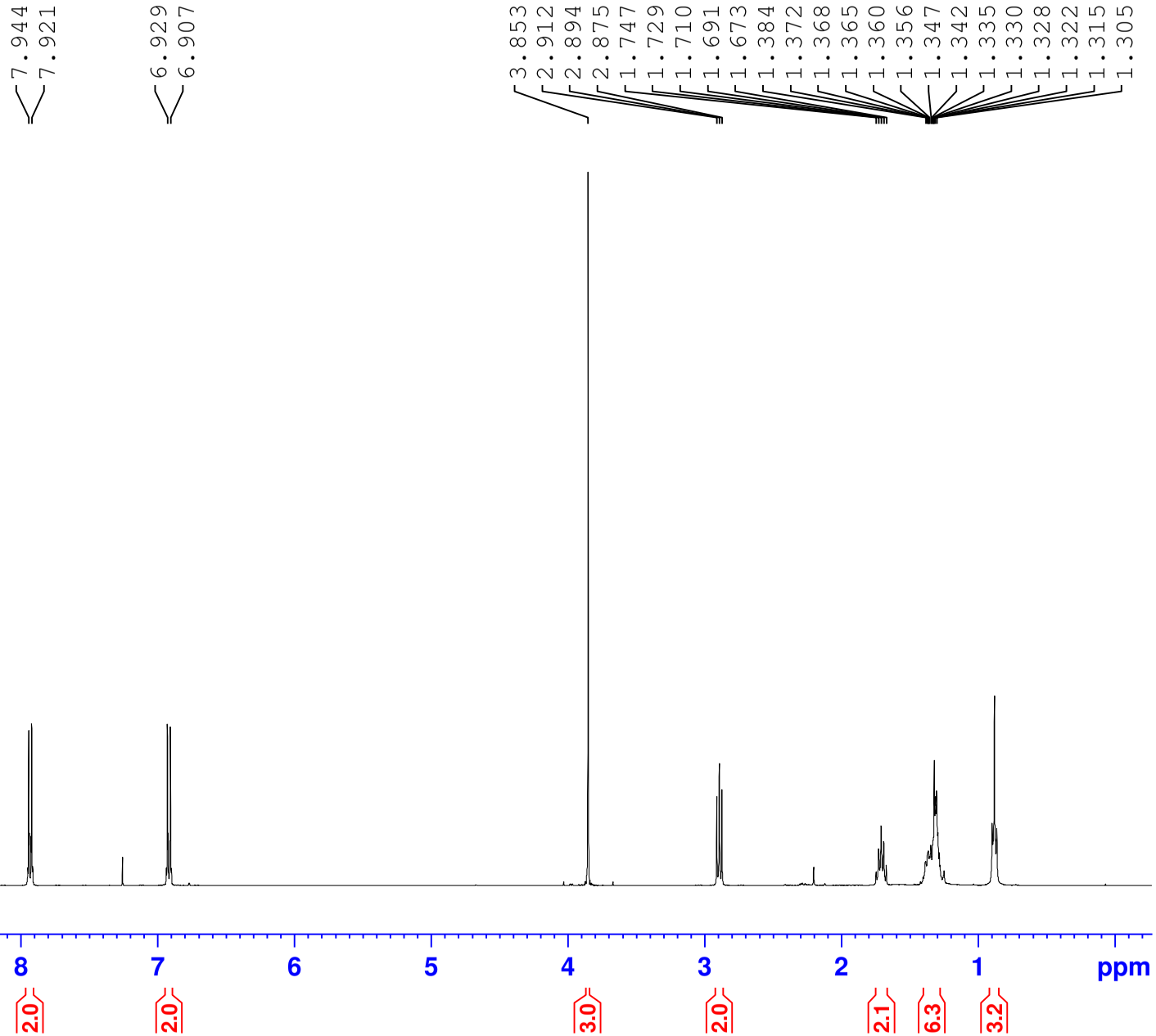
ethyl 5-(4-nitrophenyl)-5-oxopentanoate



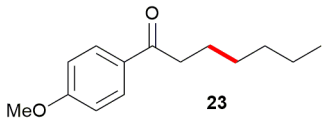
22

1-(4-methoxyphenyl)heptan-1-one





1-(4-methoxyphenyl)heptan-1-one

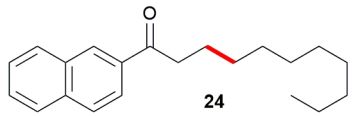


23

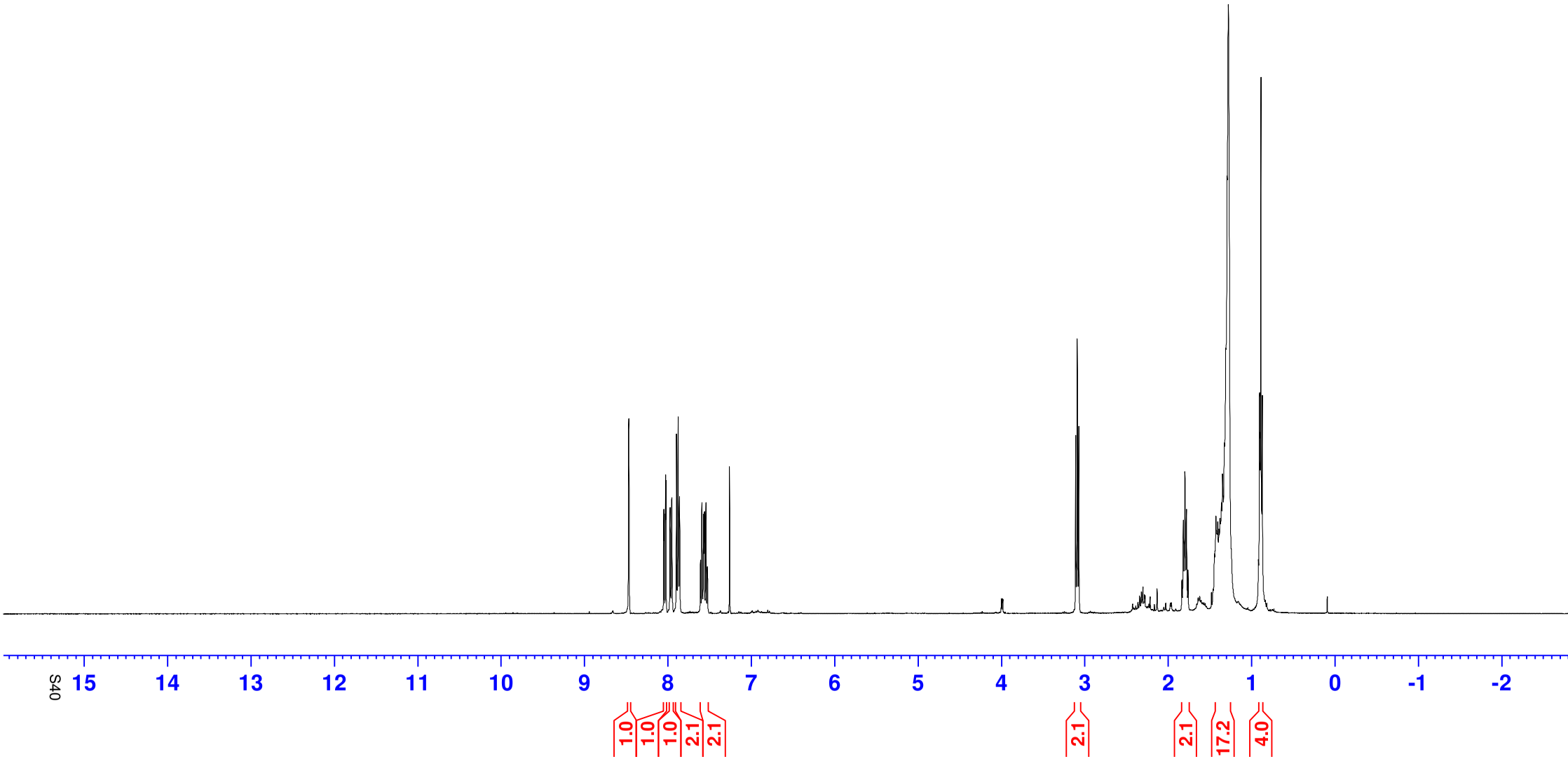
199.26
163.37
130.36
113.73
55.49
38.37
31.76
29.18
24.68
22.60
14.10

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

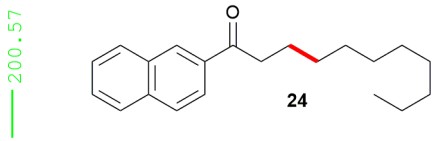
1-(naphthalen-2-yl)undecan-1-one



8.466
8.047
8.042
8.025
8.021
7.970
7.950
7.894
7.873
7.856
7.607
7.604
7.590
7.587
7.570
7.566
7.562
7.558
7.541
7.539
7.524
7.521
3.105
3.087
3.068
1.832
1.813
1.795
1.777
1.758
1.424
1.401
1.386
1.374
1.356
1.345
1.339
1.321
1.304
1.289
1.275
0.901
0.895
0.884
0.867



1-(naphthalen-2-yl)undecan-1-one

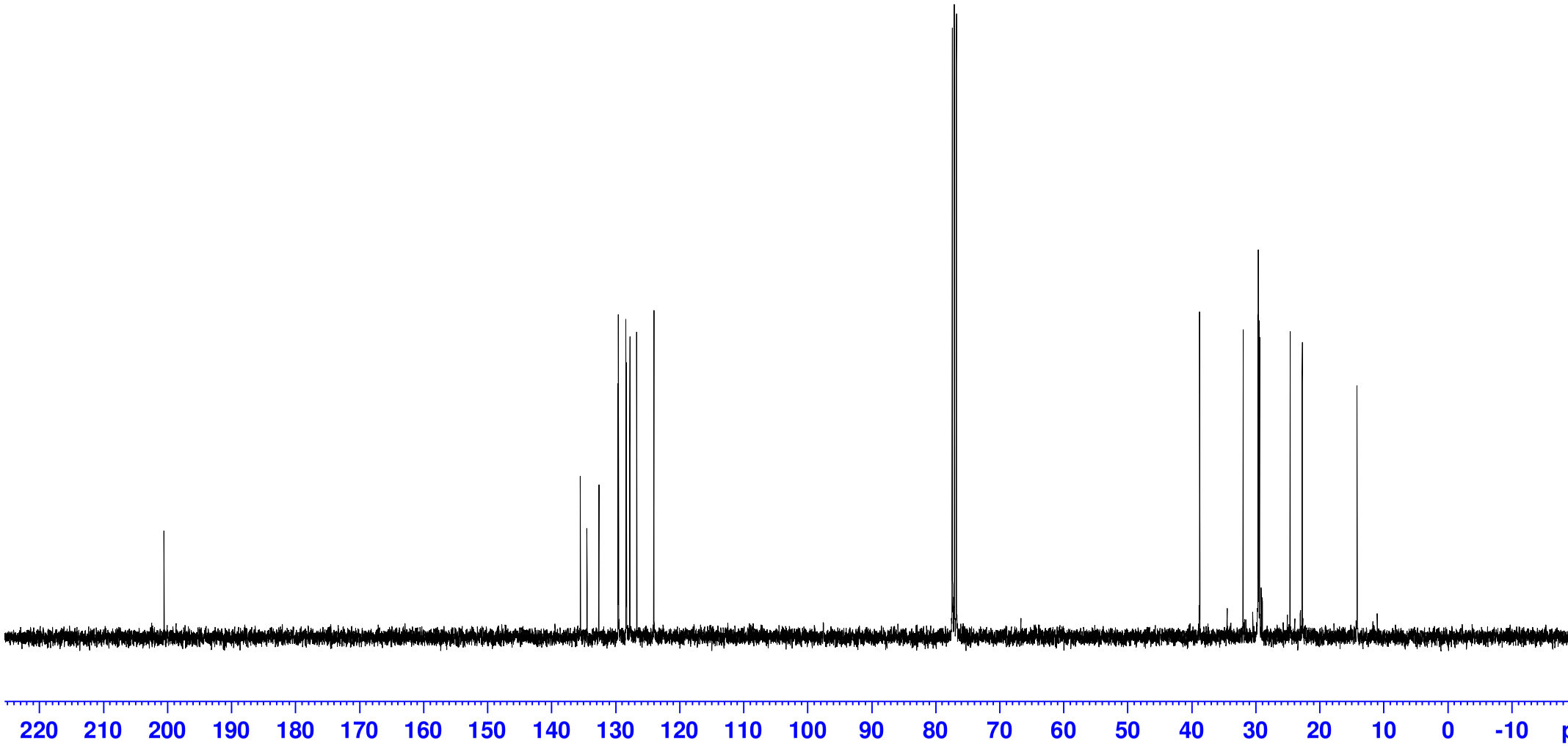


200.57

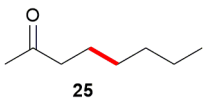
135.53
134.46
132.59
129.60
129.54
128.38
128.31
127.77
126.70
124.00

38.73
31.92
29.61
29.55
29.54
29.45
29.35
24.59
22.70

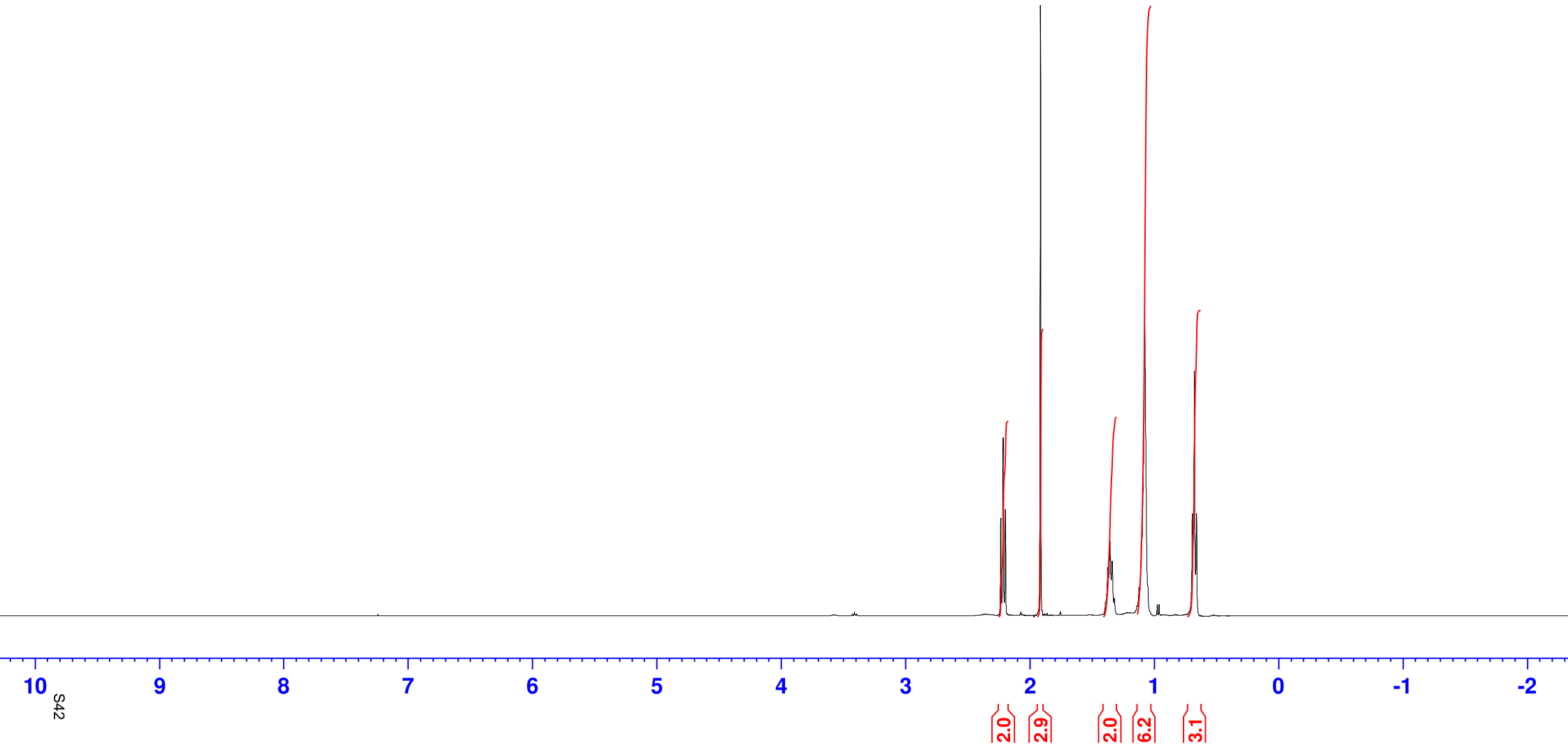
14.13



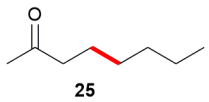
octan-2-one



2.235
2.216
2.198
1.916
1.392
1.375
1.357
1.339
1.321
1.124
1.104
1.079
1.053
0.691
0.675
0.657



octan-2-one

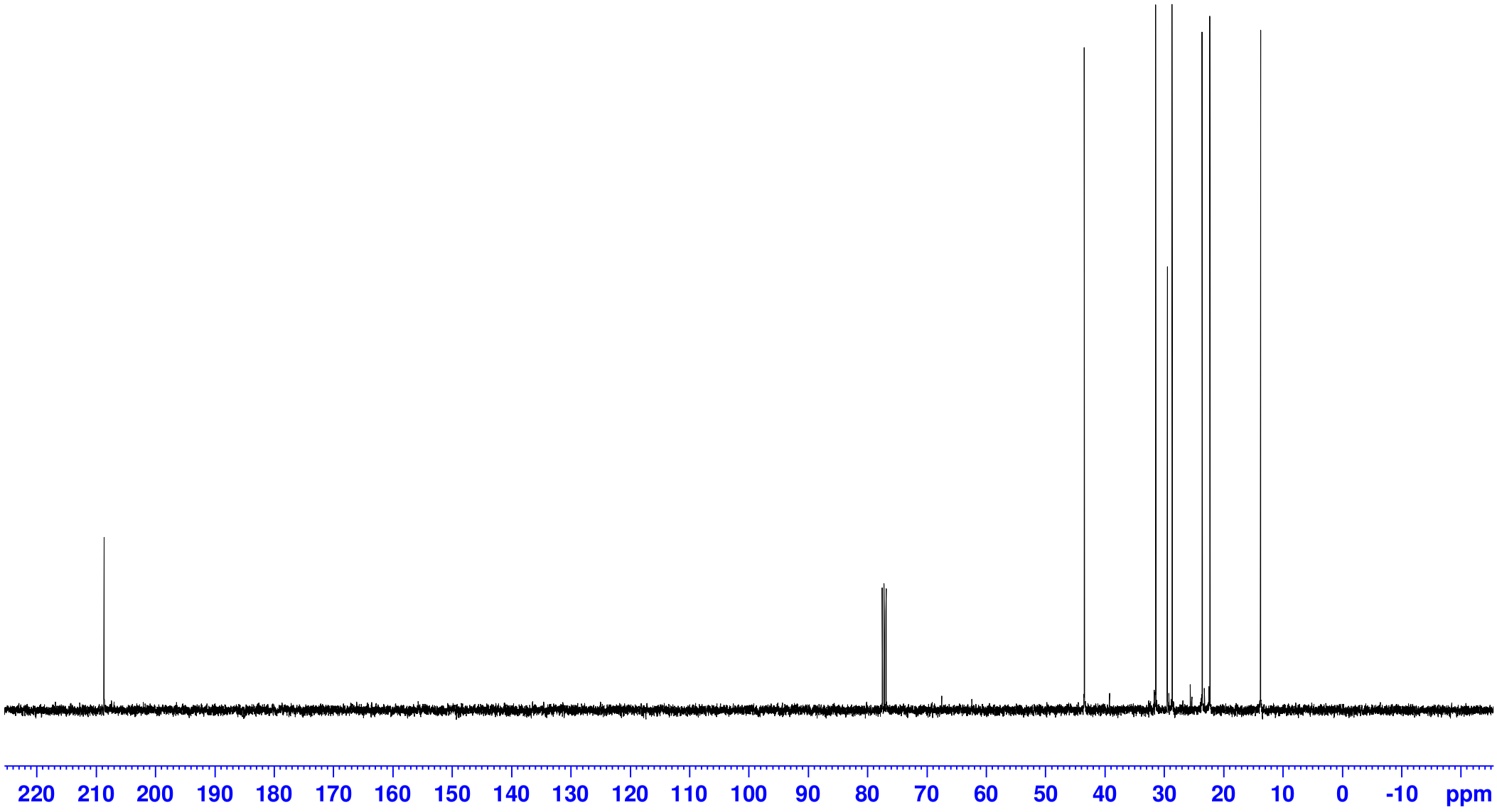


208.68

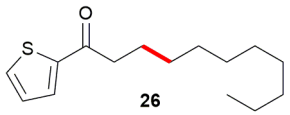
43.47

31.43
29.46
28.65
23.60
22.29

13.74



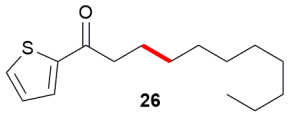
1-(thiophen-2-yl)undecan-1-one



7.707
7.697
7.615
7.602
7.128
7.117
7.106

2.901
2.882
2.864
1.776
1.758
1.740
1.722
1.703
1.331
1.311
1.258
0.892
0.875
0.858

1-(thiophen-2-yl)undecan-1-one

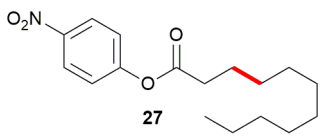


26

193.56
144.56
133.26
131.62
128.00
39.48
31.88
29.56
29.47
29.42
29.35
29.30
24.82
22.66
14.09

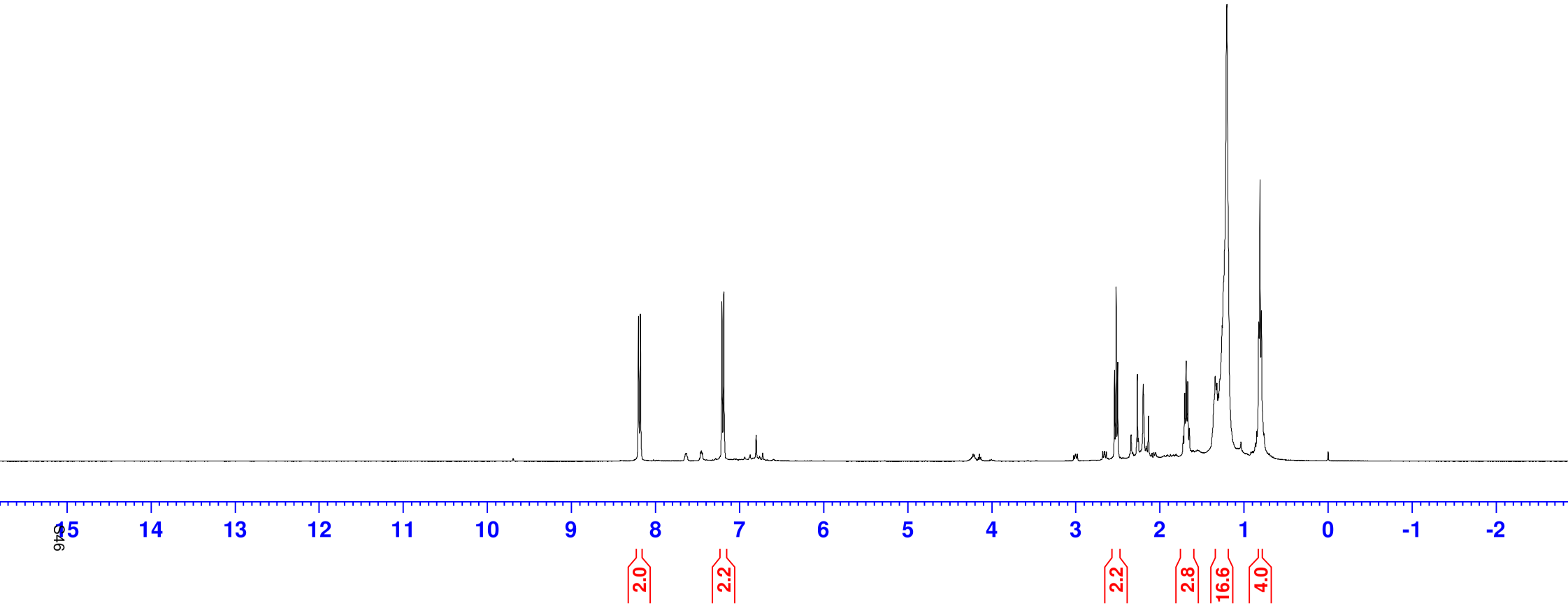
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

4-nitrophenyl undecanoate

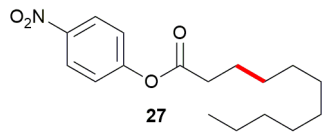


8.202
8.180
7.211
7.188

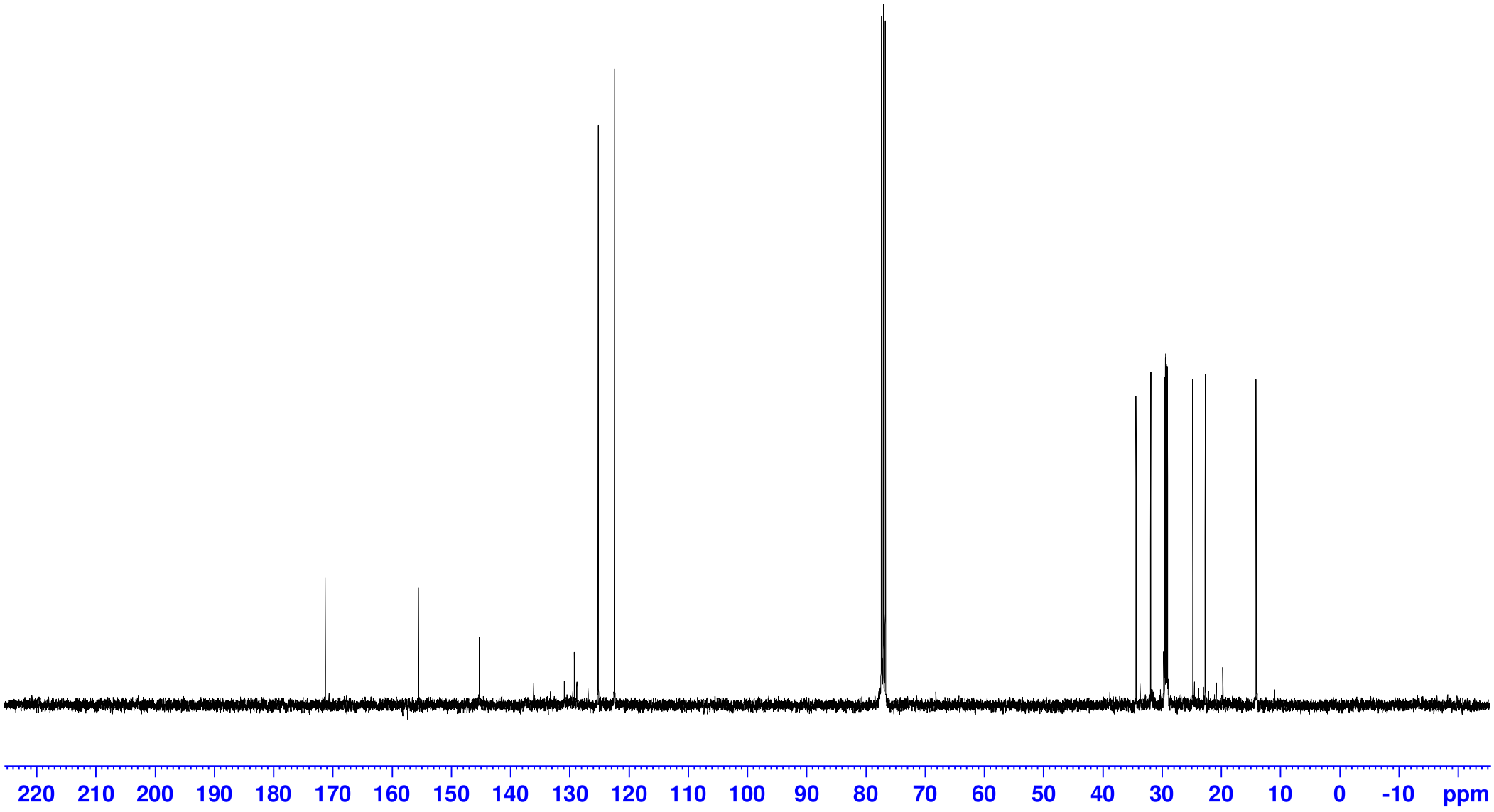
2.540
2.522
2.503
1.724
1.706
1.688
1.669
1.650
1.344
1.322
1.287
1.261
1.205
0.826
0.810
0.793



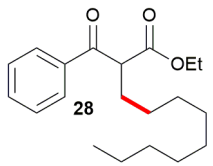
4-nitrophenyl undecanoate



171.28
155.55
145.27
125.16
122.41
34.34
31.87
29.52
29.42
29.27
29.20
29.04
24.74
22.66
14.07



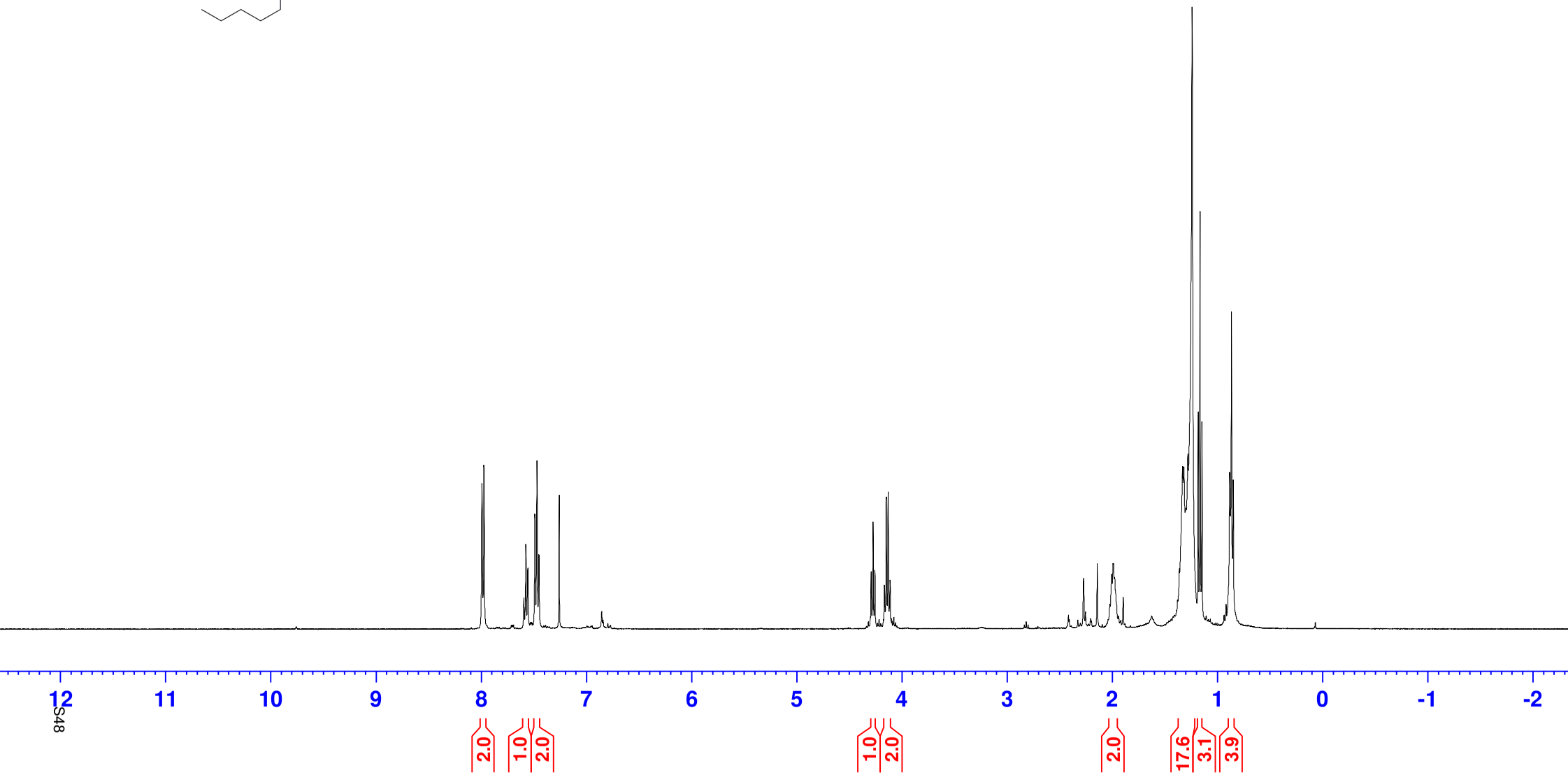
ethyl 2-benzoylundecanoate



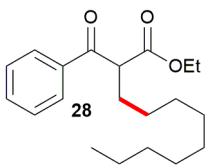
7.994
7.975
7.595
7.576
7.558
7.490
7.471
7.452

4.293
4.275
4.257
4.166
4.150
4.132
4.114

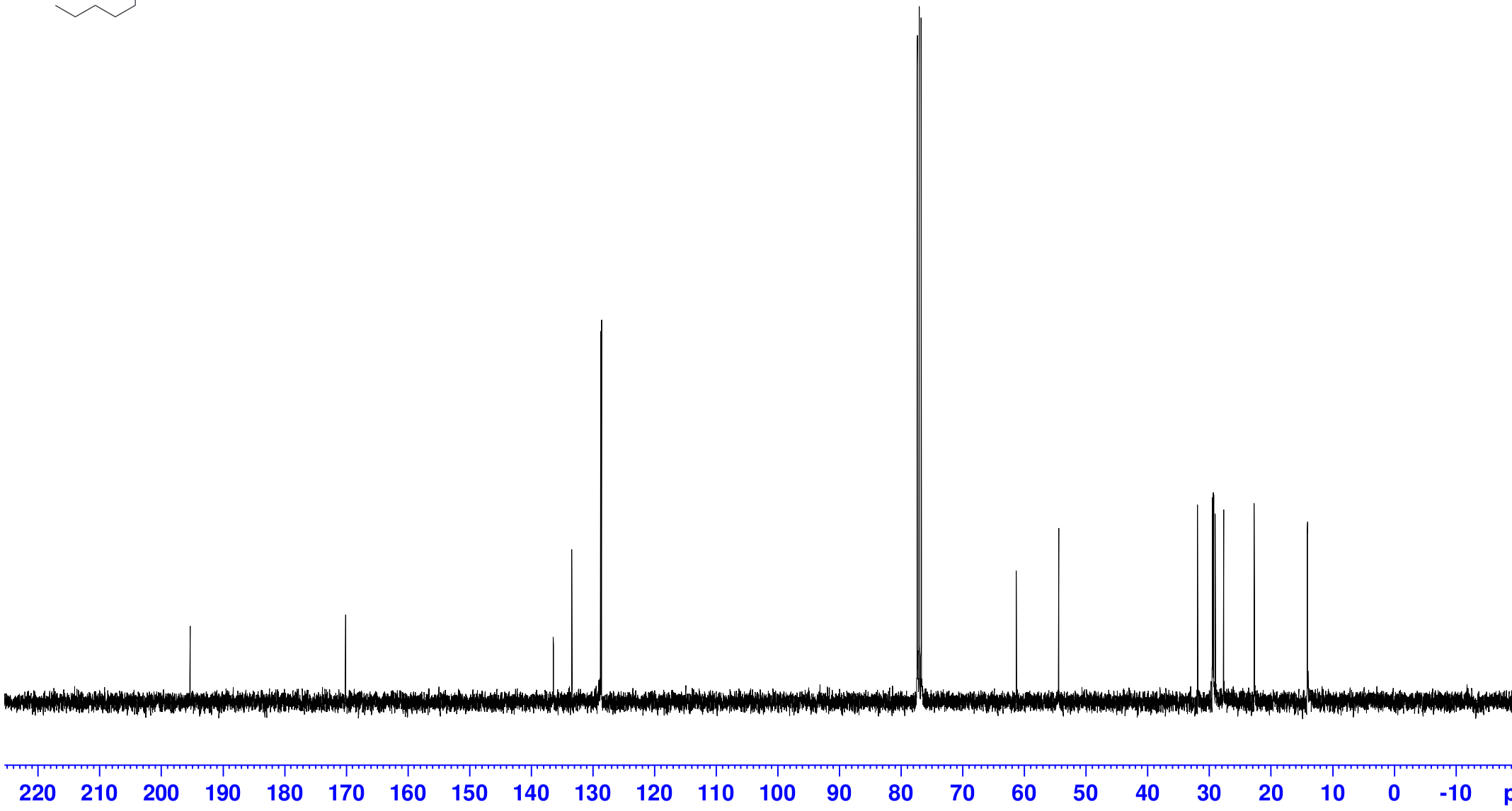
2.025
2.006
1.993
1.989
1.978
1.332
1.321
1.282
1.242
1.184
1.166
1.148
0.884
0.868
0.850



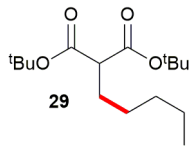
ethyl 2-benzoylundecanoate

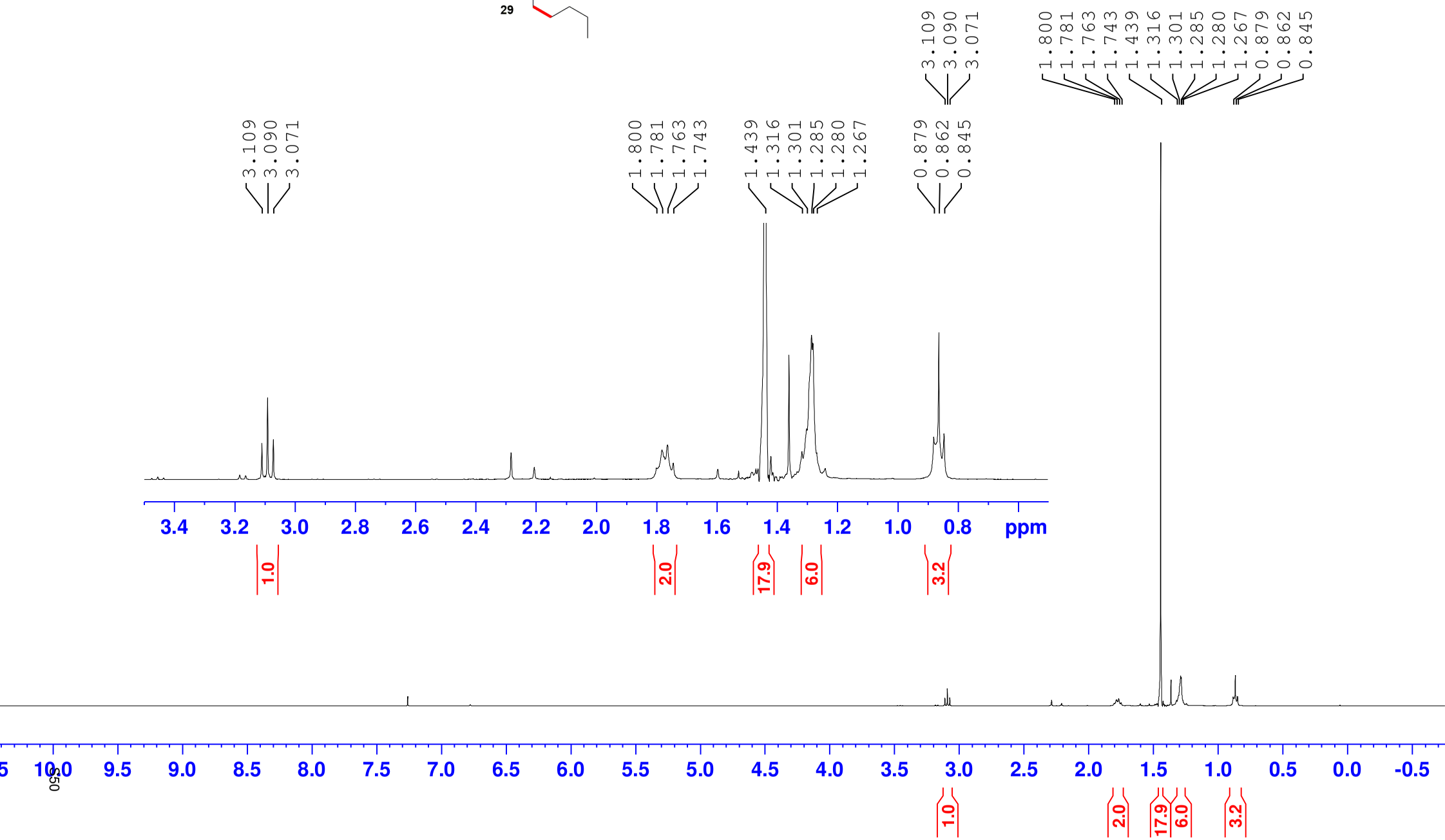


195.33
170.10
136.39
133.40
128.71
128.56
61.27
54.40
31.86
29.48
29.42
29.34
29.26
28.99
27.64
22.66
14.09
14.01

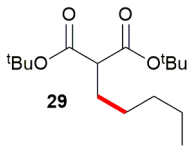


di-tert-butyl 2-pentylmalonate





di-tert-butyl 2-pentylmalonate



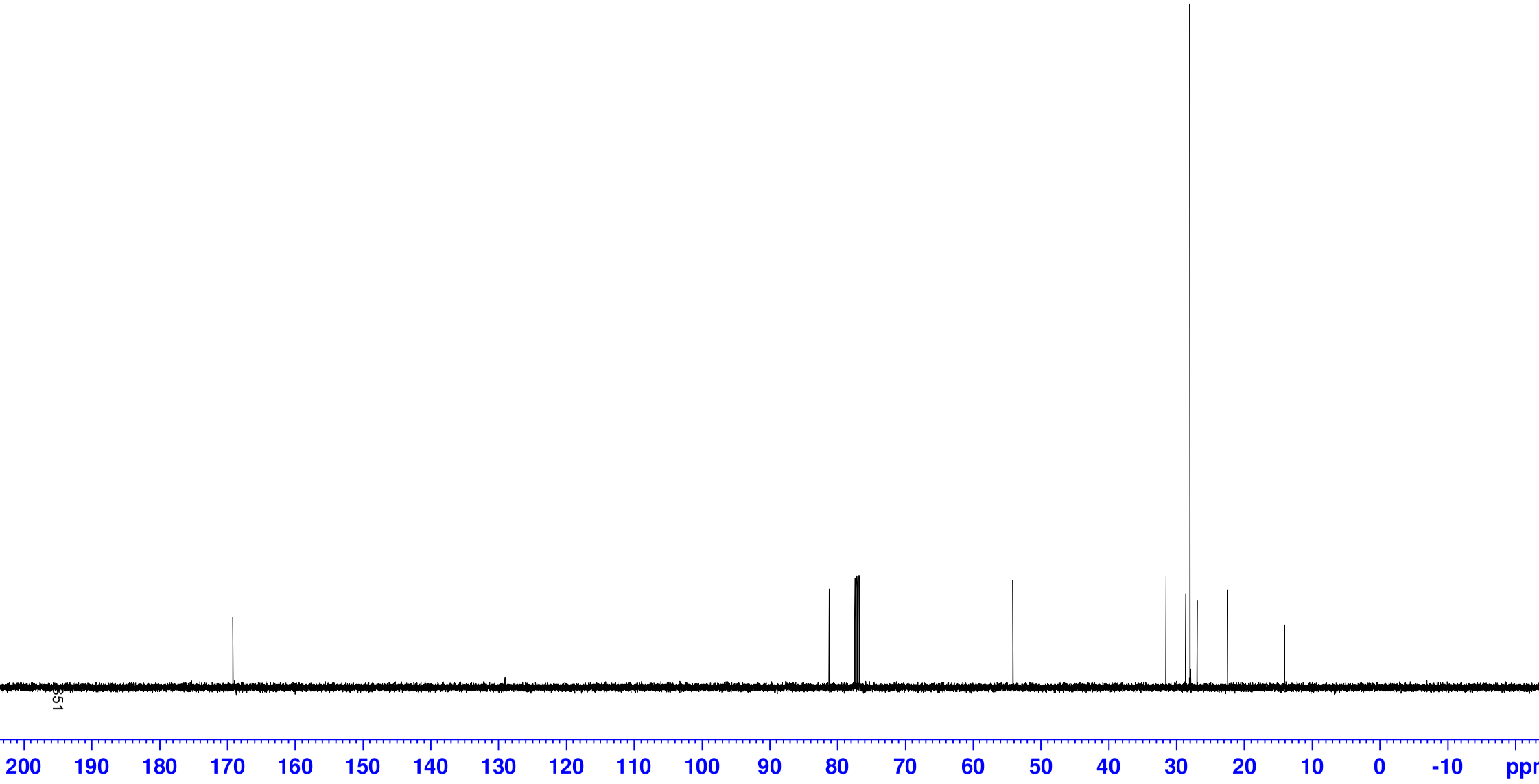
169.12

81.19

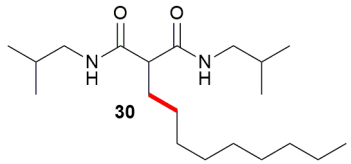
54.10

31.53
28.62
28.02
28.01
26.94
22.45

14.02

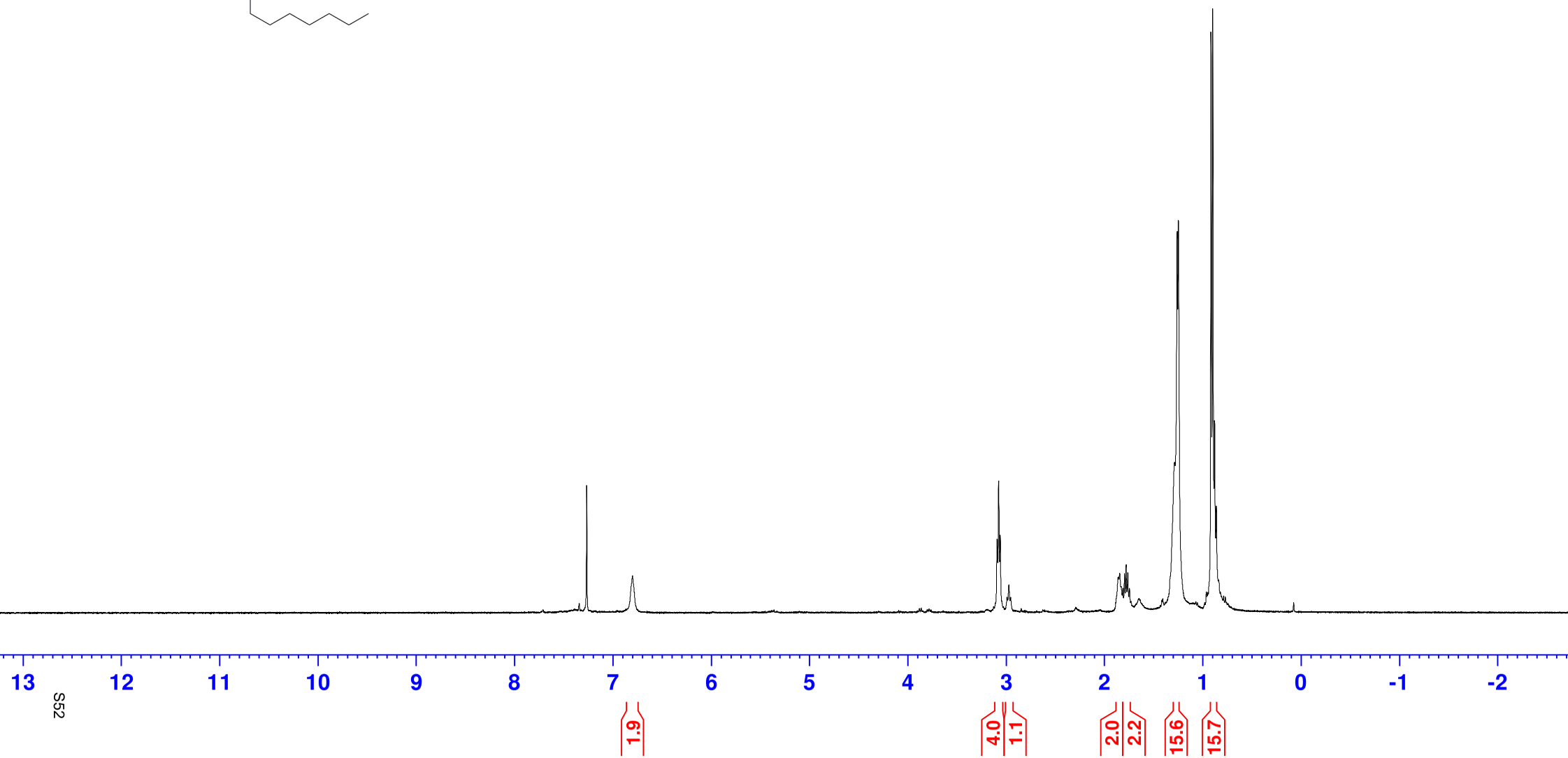


N1,N3-diisobutyl-2-nonylmalonamide

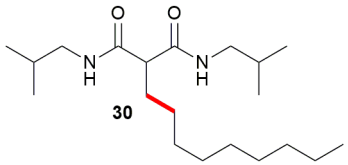


6.800

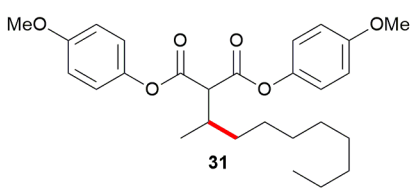
3.090
3.074
3.059
2.989
2.970
2.953
1.859
1.843
1.828
1.810
1.794
1.777
1.761
1.743
1.285
1.258
1.246
0.914
0.897
0.877
0.859

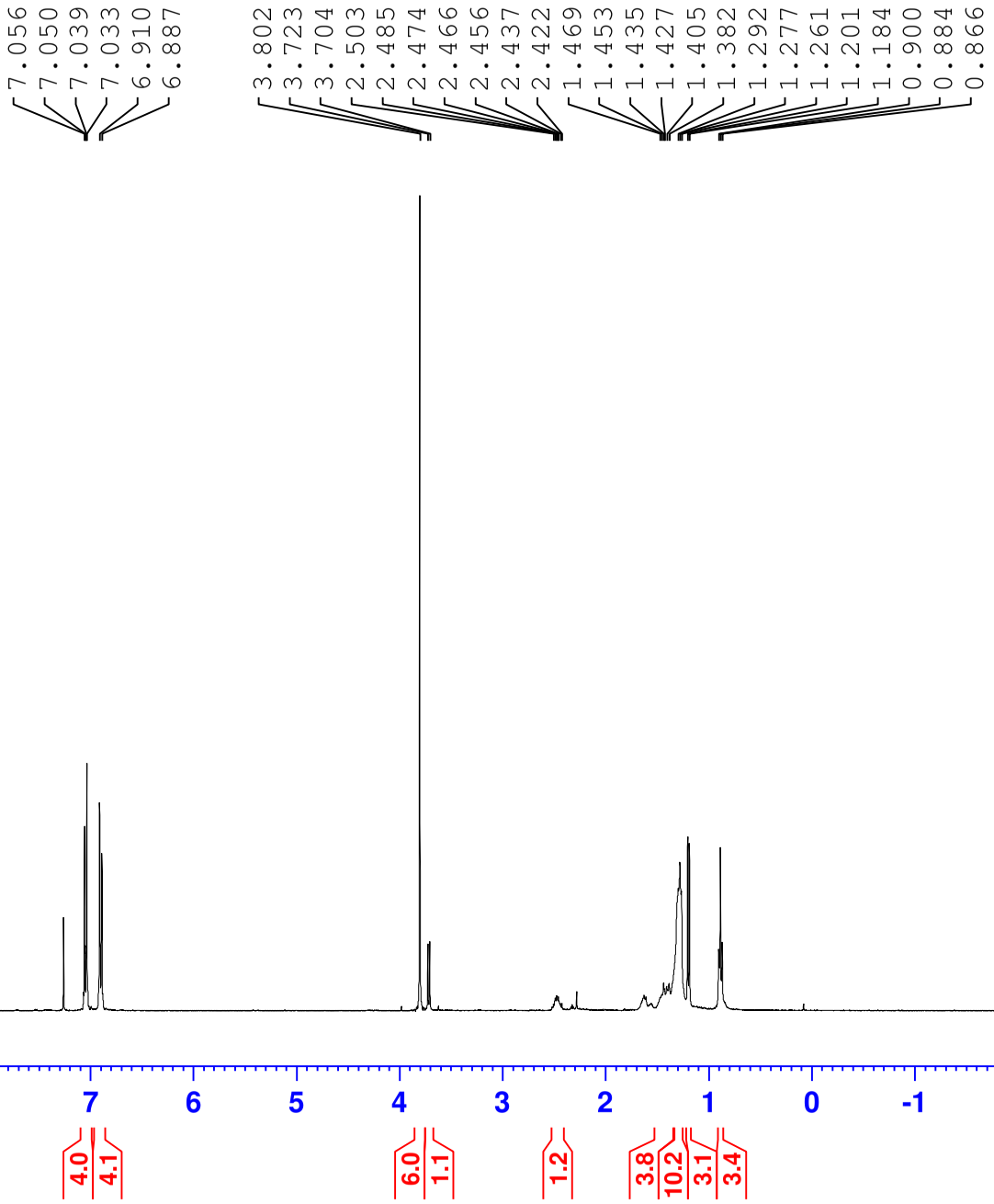


N1,N3-diisobutyl-2-nonylmalonamide

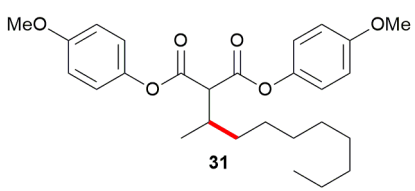


bis(4-methoxyphenyl) 2-(decan-2-yl)malonate

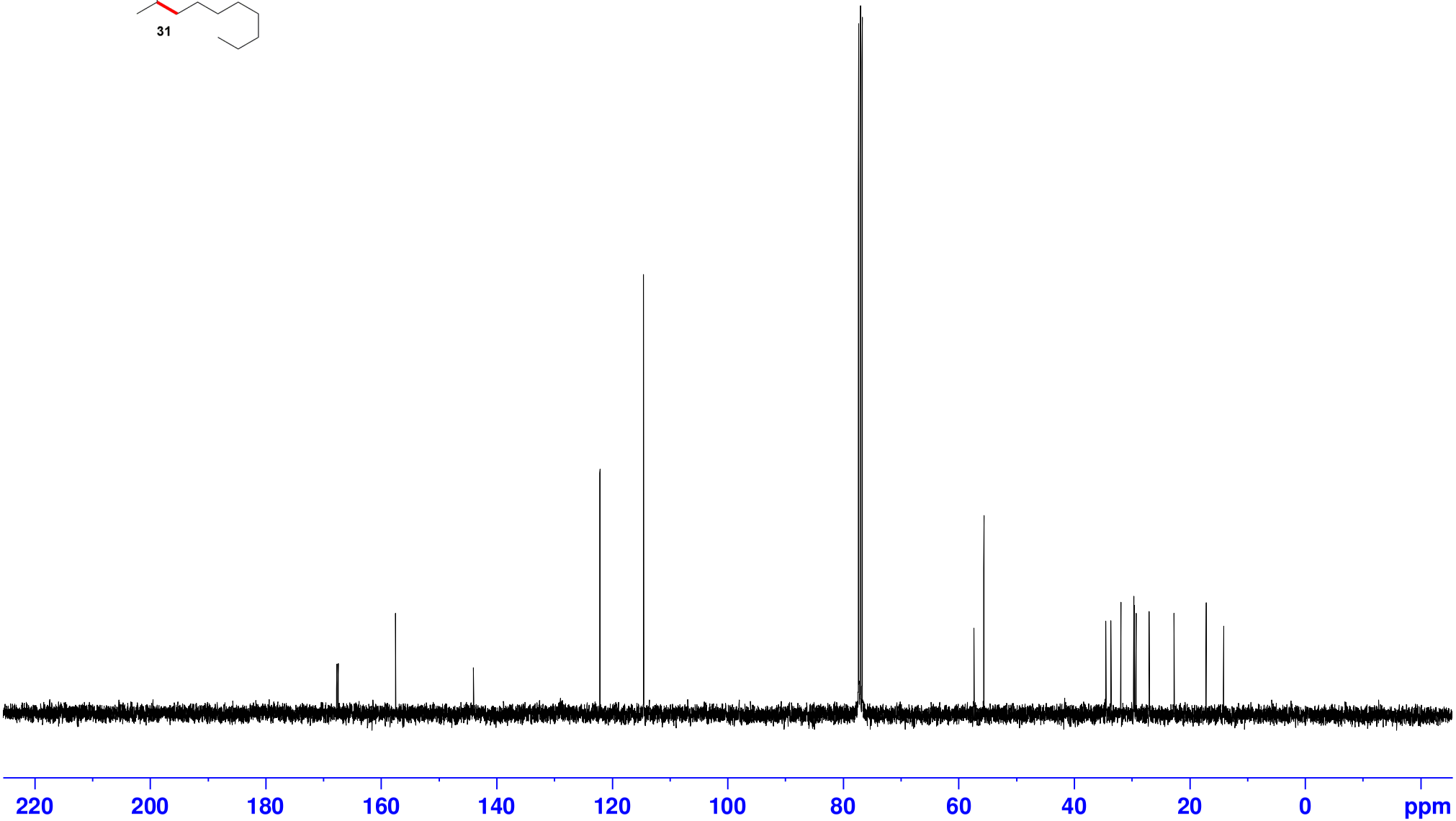




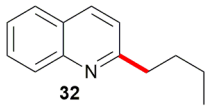
bis(4-methoxyphenyl) 2-(decan-2-yl)malonate



167.69
167.45
157.52
144.03
122.13
122.11
114.55
57.32
55.61
34.48
33.60
31.87
29.64
29.53
29.26
26.97
22.66
17.10
14.09

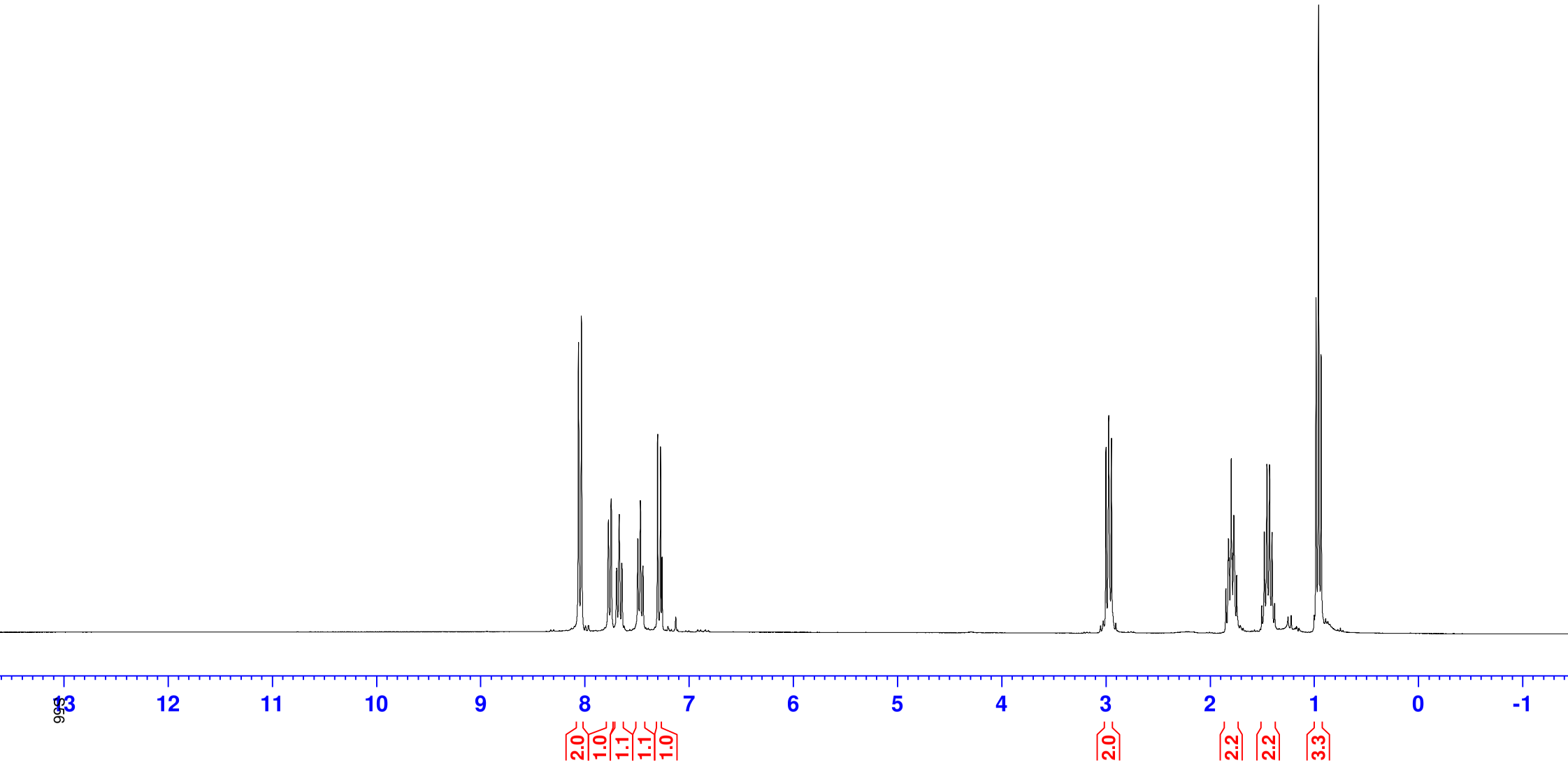


2-butylquinoline

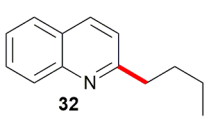


8.062
8.034
7.774
7.747
7.695
7.671
7.647
7.644
7.493
7.468
7.443
7.303
7.275

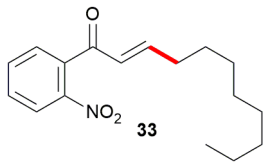
3.000
2.974
2.947
1.849
1.824
1.818
1.798
1.789
1.773
1.767
1.746
1.505
1.480
1.456
1.430
1.406
1.382
0.984
0.960
0.935



2-butylquinoline

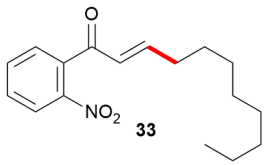


(E)-1-(2-nitrophenyl)undec-2-en-1-one



33

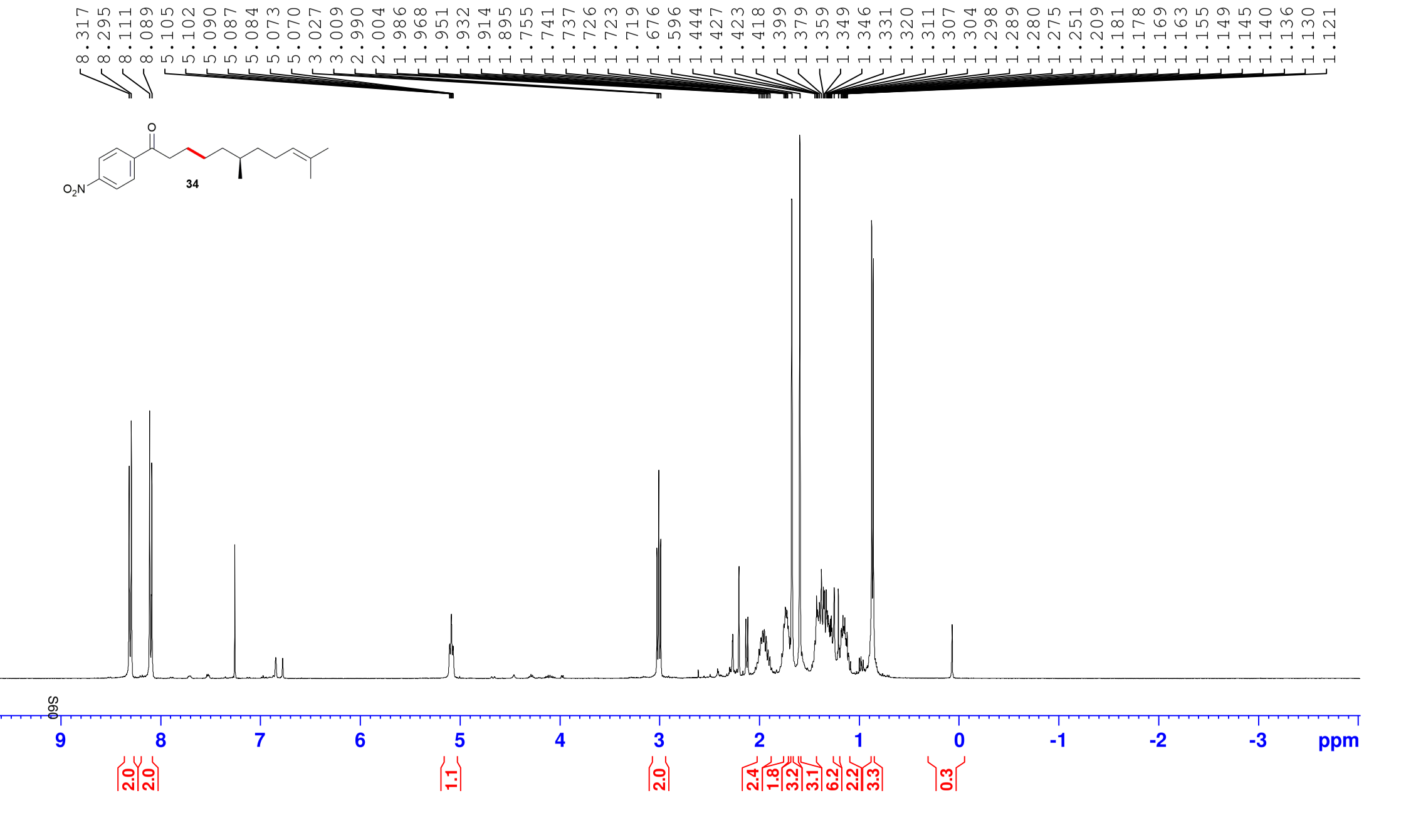
(E)-1-(2-nitrophenyl)undec-2-en-1-one



33

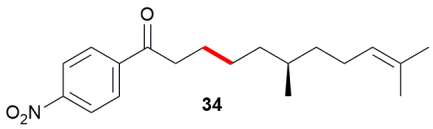
190.90
150.53
137.25
132.86
129.45
127.17
124.68
123.26
30.83
28.83
28.68
28.34
28.32
28.19
28.05
21.64
13.07

ET-(S)-citronellol-nitro-vinyl-ketone



(S)-6,10-dimethyl-1-(4-nitrophenyl)undec-9-en-1-one

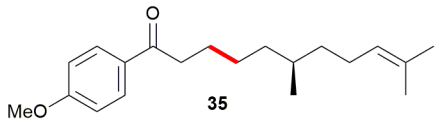
198.88
150.34
141.58
131.18
129.11
124.97
123.92
39.28
37.15
36.80
32.36
26.75
25.79
25.62
24.42
19.59
17.71



34

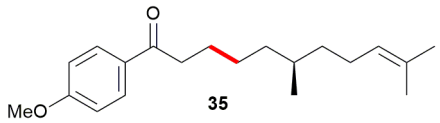
(s)-6,10-dimethyl-1-(4-methoxyphenyl)undec-9-en-1-one

7.947 7.925 6.933 6.911 5.092 5.089 5.086 3.857 2.919 2.901 2.882 1.964 1.961 1.948 1.928 1.724 1.721 1.709 1.706 1.702 1.694 1.690 1.687 1.674 1.672 1.657 1.593 1.427 1.423 1.402 1.390 1.382 1.376 1.369 1.364 1.357 1.348 1.339 1.333 1.325 1.322 1.309 1.301 1.296 1.287 1.285 1.281 1.272 1.253 1.169 1.162 1.155 1.151 1.147 1.137 1.132 1.128 1.113 0.867 0.851



35

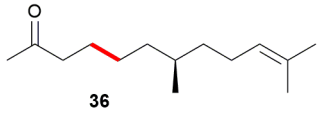
(s)-6,10-dimethyl-1-(4-methoxyphenyl)undec-9-en-1-one



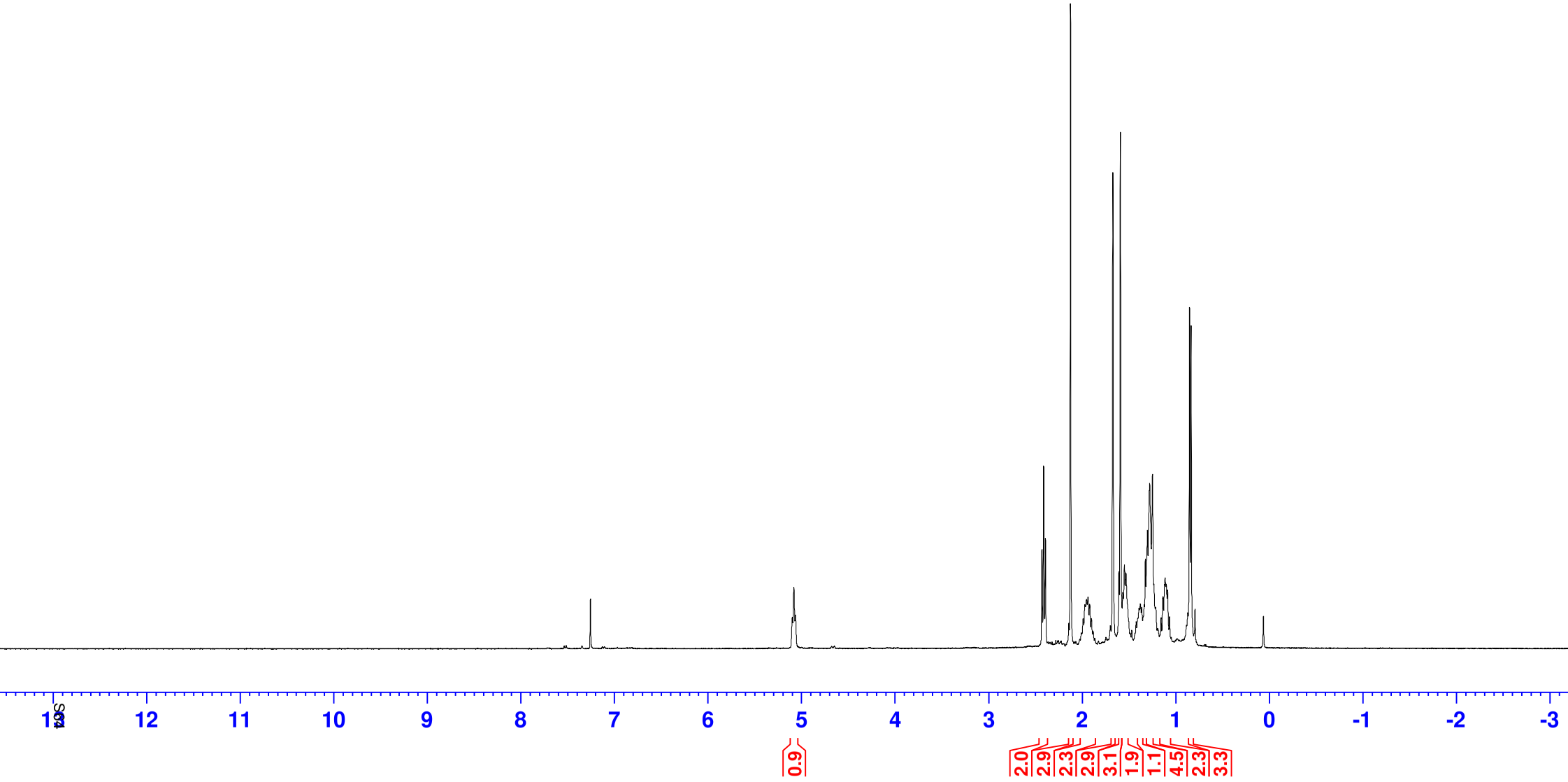
199.22
163.37
131.04
130.32
125.07
113.73
55.48
38.41
37.18
36.86
32.38
26.94
25.76
25.63
25.03
19.60
17.68

220 200 180 160 140 120 100 80 60 40 20 0 ppm

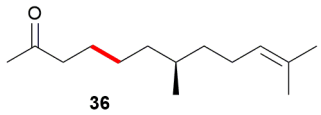
(S)-7,11-dimethyldodec-10-en-2-one



5.099
5.082
5.066
2.431
2.412
2.393
2.125
1.990
1.972
1.954
1.939
1.920
1.902
1.673
1.592
1.549
1.532
1.393
1.380
1.364
1.325
1.303
1.279
1.249
1.234
1.136
1.121
1.113
1.103
1.088
1.067
0.852
0.836



(S)-7,11-dimethyldodec-10-en-2-one



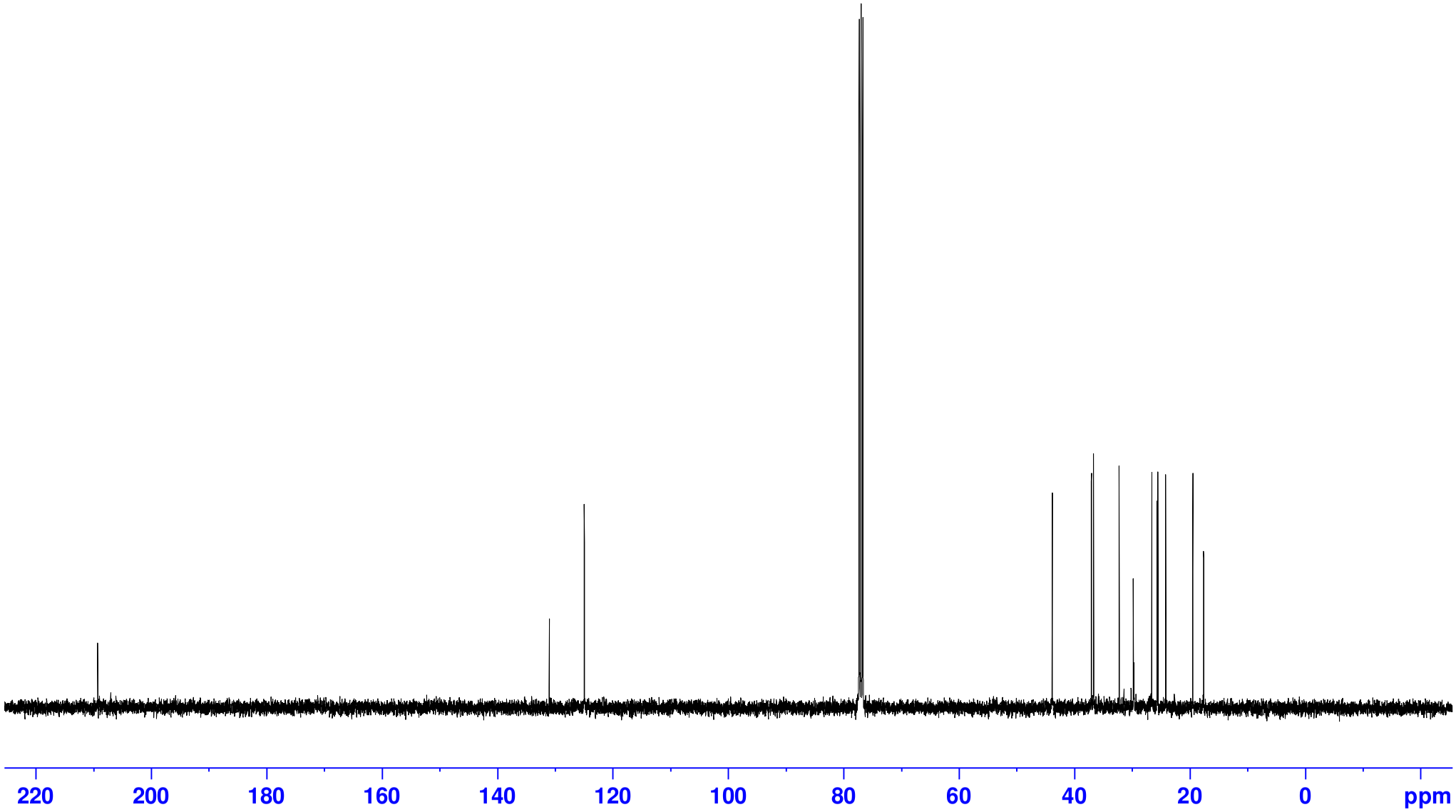
36

209.29

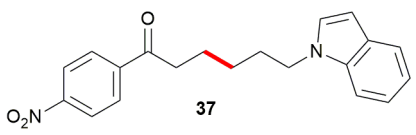
131.03

124.96

43.85
37.08
36.70
32.26
29.83
26.61
25.70
25.54
24.19
19.51
17.62

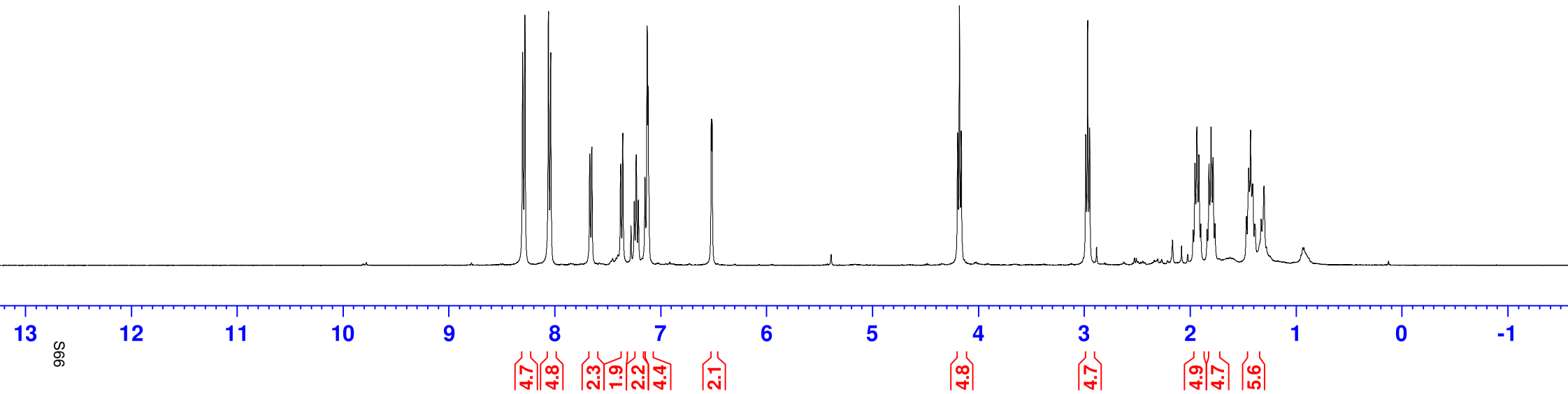


ET-4-nitrophenyl-vinylketone-indoleiodide

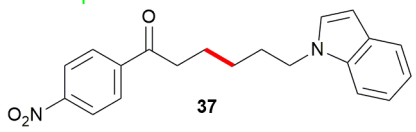


8.279
8.258
8.036
8.014
7.645
7.626
7.353
7.333
7.224
7.206
7.186
7.124
7.103
7.094
6.497
6.490

4.171
4.154
4.137
2.961
2.943
2.925
1.948
1.930
1.911
1.892
1.875
1.815
1.797
1.778
1.759
1.740
1.443
1.423
1.404
1.384
1.365
1.305
1.278

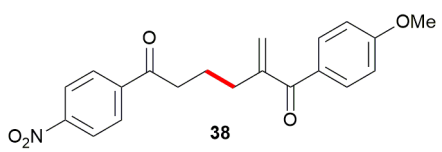


6-(1 H-indol-1-yl)-1-(4-nitrophenyl)hexan-1-one

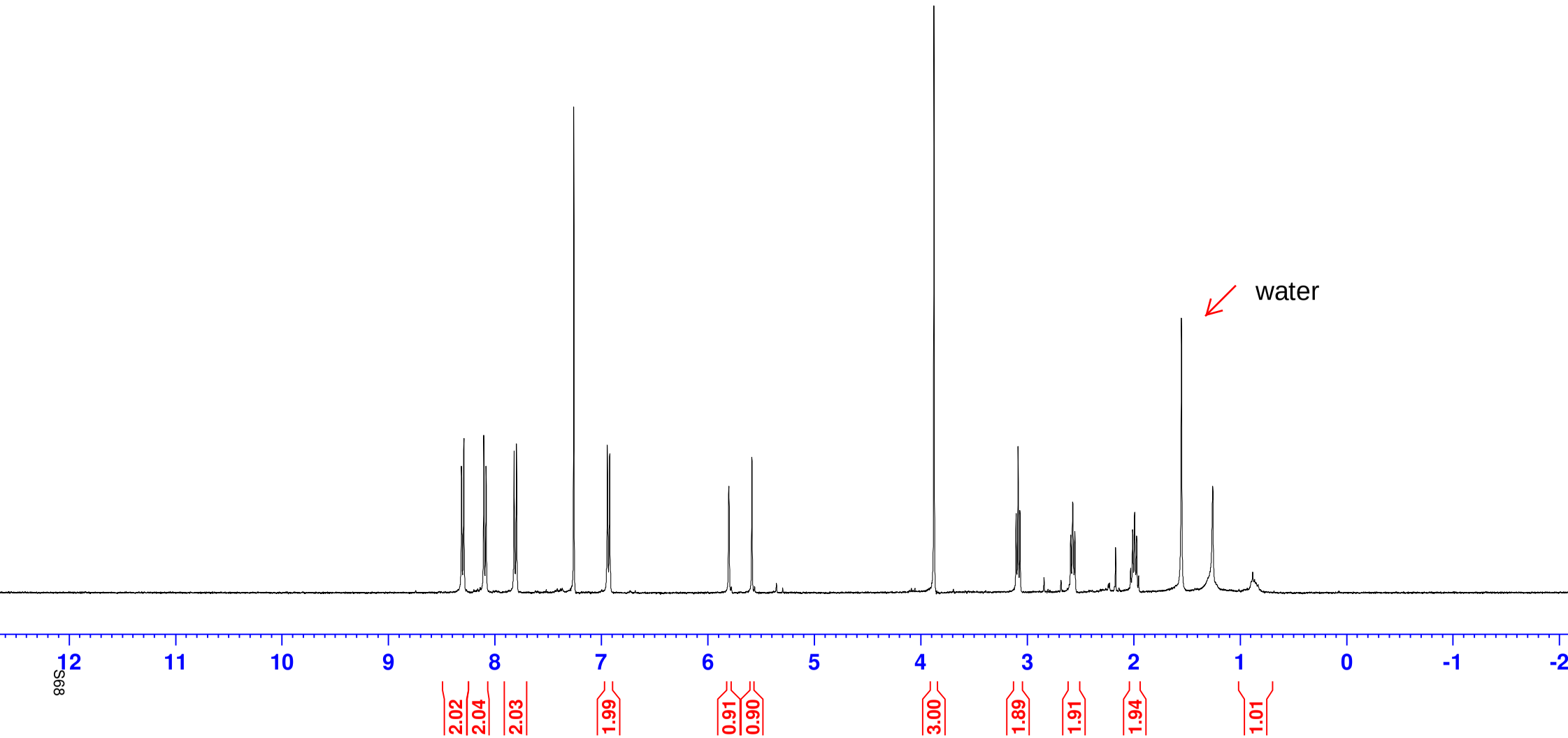


198.34
150.26
141.27
135.93
128.98
128.61
127.79
123.84
121.41
121.01
119.26
109.32
101.06
46.11
38.84
30.10
26.54
23.47

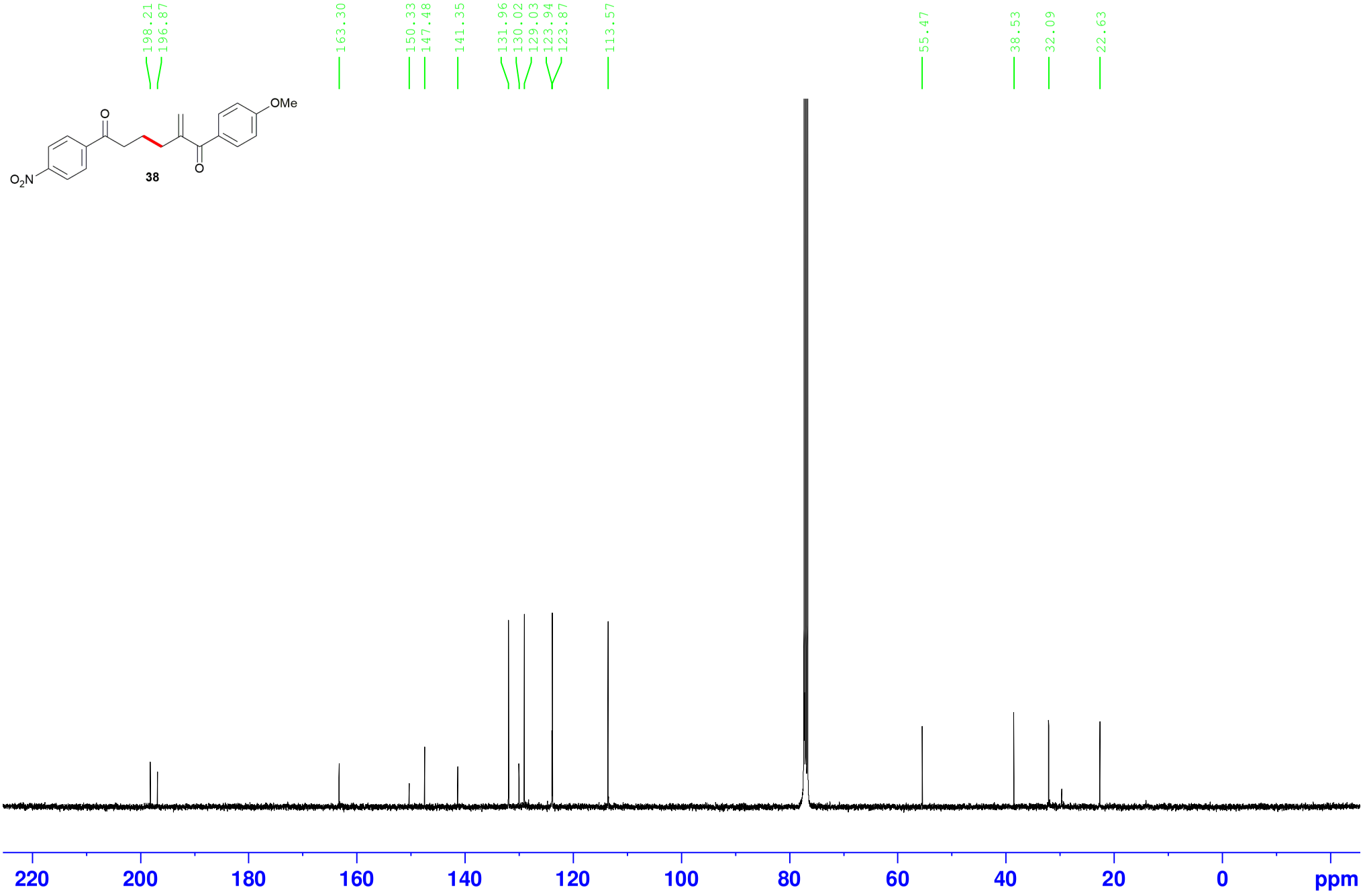
1-(4-methoxyphenyl)-2-methylene-6-(4-nitrophenyl)hexane-1,6-dione



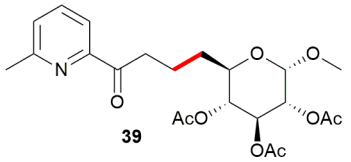
8.314
8.292
8.104
8.082
7.819
7.797
6.944
6.922
5.802
5.585
3.874
3.102
3.084
3.066
2.589
2.570
2.551
2.026
2.007
1.988
1.970
1.951



1-(4-methoxyphenyl)-2-methylene-6-(4-nitrophenyl)hexane-1,6-dione

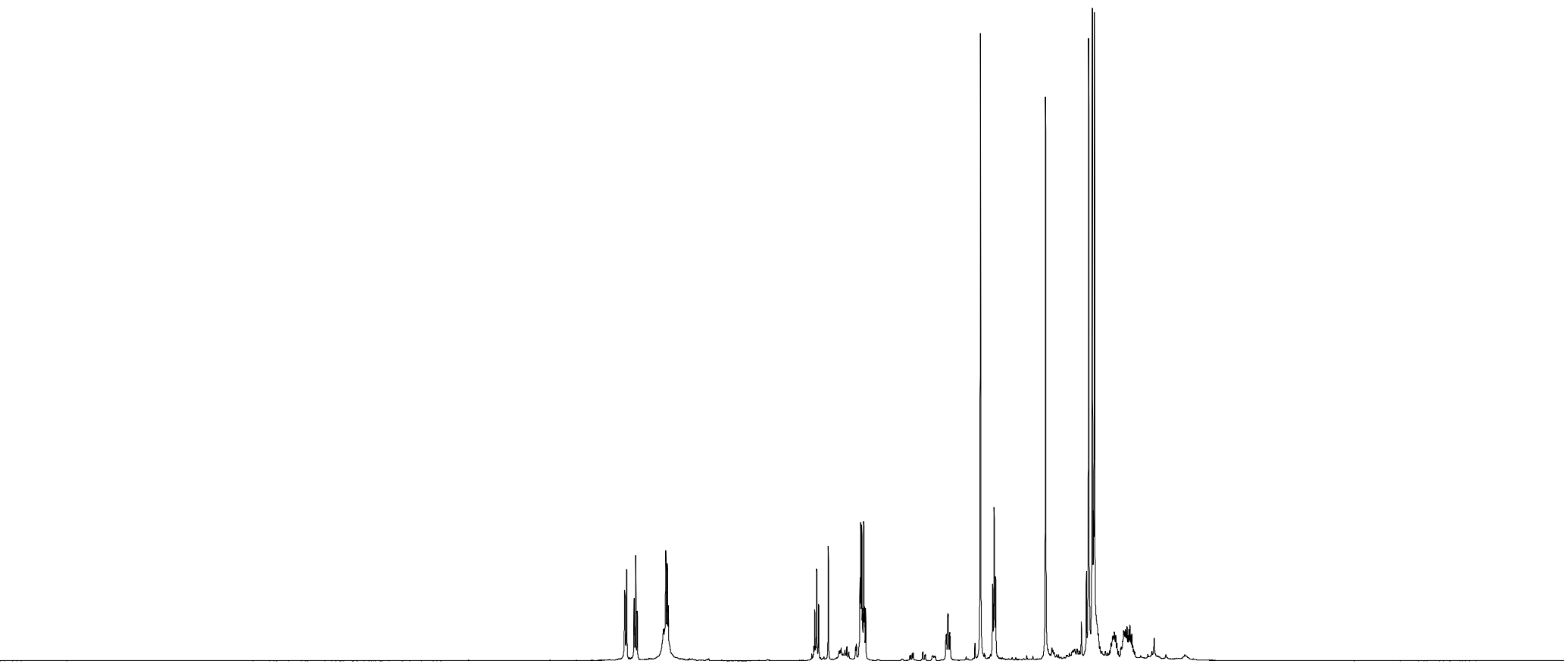


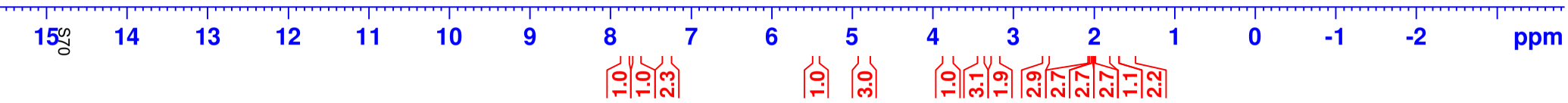
2-methoxy-6-(4-(6-methylpyridin-2-yl)-4-oxobutyl)tetrahydro-2H-pyran-3,4,5-triyl



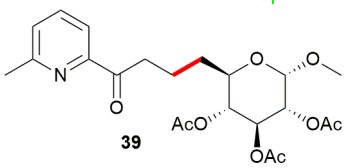
39

7.819
7.800
7.707
7.688
7.668
7.315
7.296
7.284
5.466
5.442
5.417
4.904
4.895
4.882
4.873
4.858
4.848
4.835
3.838
3.832
3.815
3.809
3.791
3.785
3.409
3.258
3.240
3.221
2.601
2.066
2.020
2.007
1.995
1.773
1.765
1.756
1.748
1.731
1.715
1.643
1.634
1.628
1.620
1.610
1.602
1.591
1.577
1.567
1.555





2-methoxy-6-(4-(6-methylpyridin-2-yl)-4-oxobutyl)tetrahydro-2H-pyran-3,4,5-triyl

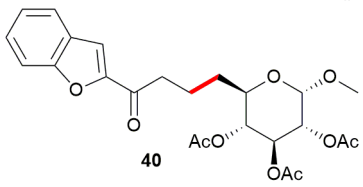


39

201.85
170.58
170.15
170.06
169.82
169.54
157.92
152.96
136.85
128.60
128.38
128.07
127.67
126.65
118.71
96.78
96.49
77.38
77.06
76.74
72.43
71.21
70.78
70.34
70.11
68.56
68.34
67.14
61.92
55.43
55.25
53.41
37.05
30.65
24.39
20.71
20.69
20.67
20.57
19.74

2-(4-(benzofuran-2-yl)-4-oxobutyl)-6-methoxytetrahydro-2H-pyran-3,4,5-triyl tri

7.732 7.713 7.599 7.578 7.514 7.491 7.471 7.345 7.326 7.308 7.284 5.485 5.461 5.436 4.920 4.910 4.894 4.881 4.869 4.858 3.845 3.839 3.821 3.815 3.798 3.791 3.415 3.033 3.015 3.002 2.984 2.084 2.041 2.022 2.013 1.872 1.848 1.837 1.828 1.811 1.796 1.708 1.701 1.695 1.681 1.674 1.667 1.655 1.648 1.640 1.634 1.611 1.598 1.587 1.575 1.563 1.551 1.540



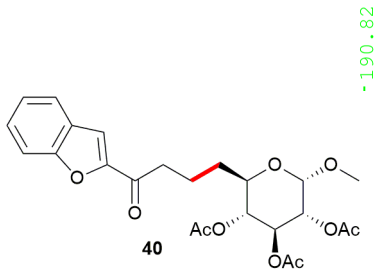
40

S72

10 9 8 7 6 5 4 3 2 1 0 -1 -2

1.0 1.0 2.2 1.2 1.1 3.1 1.1 2.8 1.9 3.4 3.6 4.0 1.2 1.6 1.3

2-(4-(benzofuran-2-yl)-4-oxobutyl)-6-methoxytetrahydro-2H-pyran-3,4,5-triyl tri

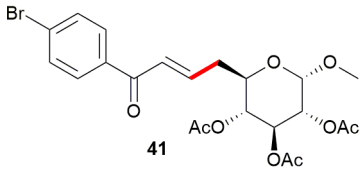


40

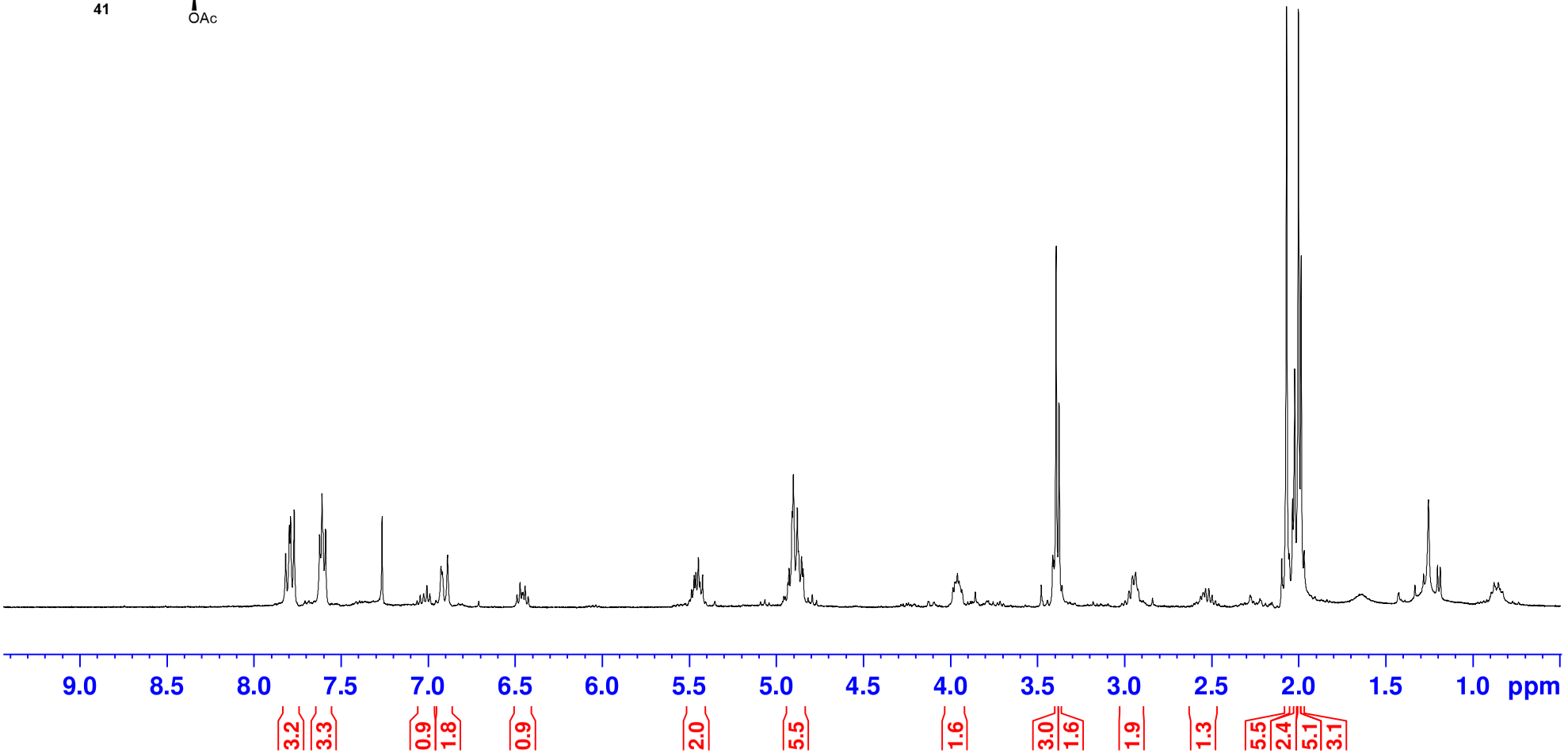
-190.82
170.19
170.06
169.89
155.59
152.55
128.20
127.04
123.91
123.27
112.56
112.43
96.54
72.31
71.18
70.26
68.33
55.32
38.52
30.57
20.71
20.68
19.79

2-(4-(4-bromophenyl)-4-oxobut-2-en-1-yl)-6-methoxytetrahydro-2H-pyran-3,4,5-tri

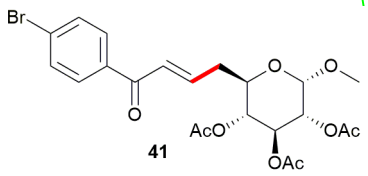
7.818 7.796 7.790 7.768 7.623 7.609 7.588 7.045 7.025 7.006 6.989 6.925 6.917 6.887 6.489 6.471 6.459 6.454 6.442 6.424 5.485 5.471 5.461 5.447 5.437 5.423 4.933 4.926 4.909 4.901 4.878 4.871 4.854 4.845 3.984 3.969 3.959 3.950 3.933 3.855 3.411 3.406 3.392 3.375 2.068 2.021 1.999 1.985



41



2-(4-(4-bromophenyl)-4-oxobut-2-en-1-yl)-6-methoxytetrahydro-2H-pyran-3,4,5-tri

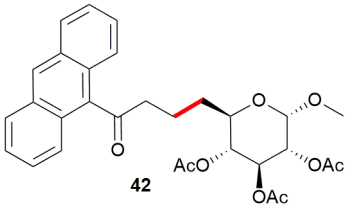


41

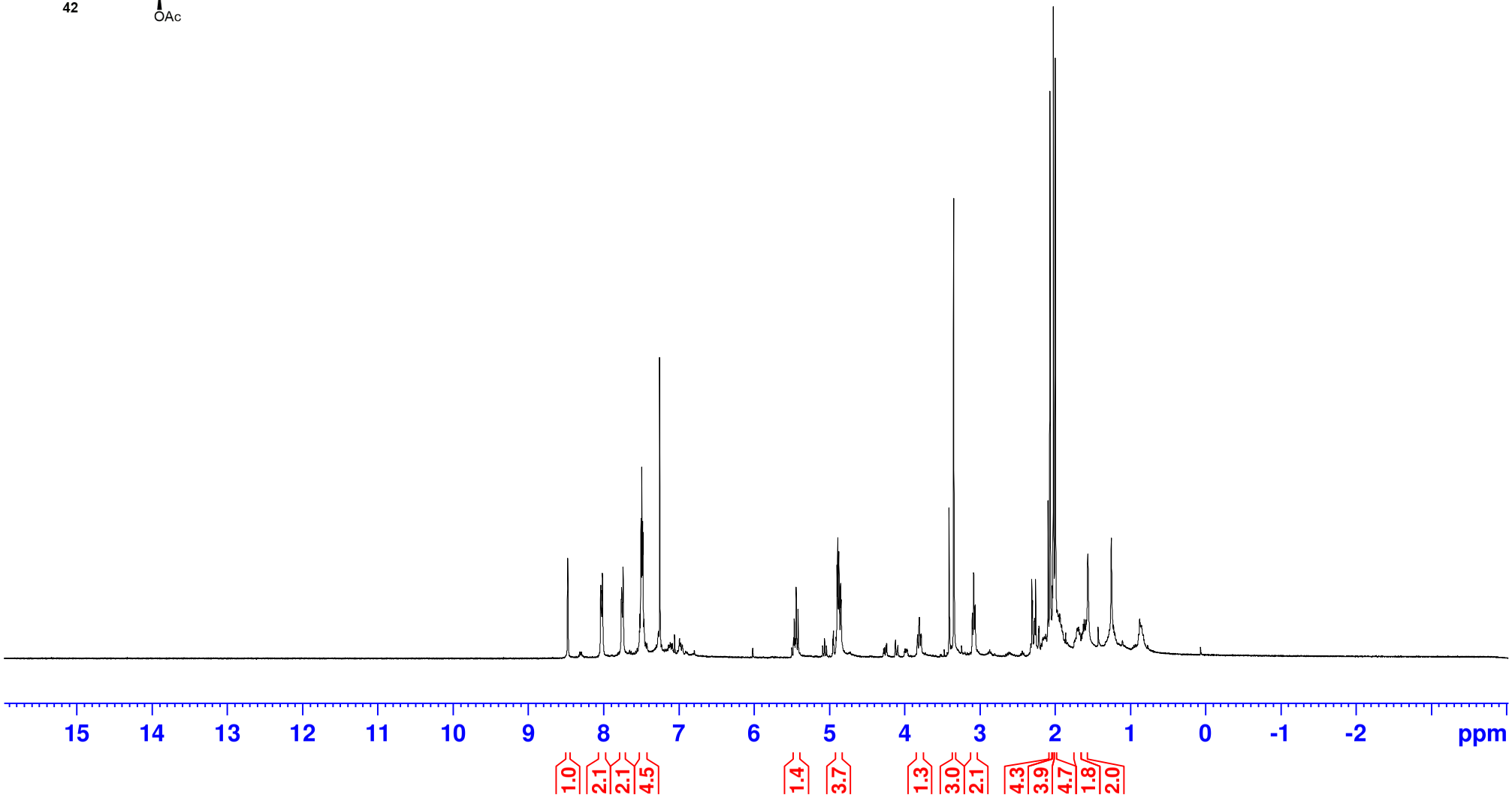
190.31
188.91
170.19
170.13
170.05
170.00
169.79
169.71
143.82
143.76
137.07
136.33
131.92
131.88
130.07
129.84
128.06
128.04
127.98
125.78
96.68
72.15
71.54
71.09
70.97
70.19
70.05
68.07
67.57
55.53
55.32
34.74
30.72
29.68
20.71
20.69
20.64

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

2-(4-(anthracen-9-yl)-4-oxobutyl)-6-methoxytetrahydro
-2H-pyran-3,4,5-triyl triacetate

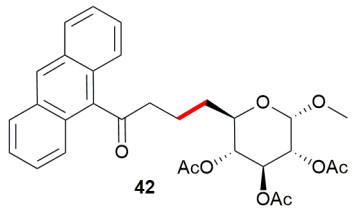


8.478 8.038 8.021 8.016 7.767 7.760 7.754 7.751 7.742 7.509 7.502 7.493 7.483 7.478 7.476 5.466 5.452 5.441 5.417 4.898 4.889 4.877 4.873 4.868 4.852 4.850 4.843 3.350 3.099 3.082 3.063 2.070 2.025 2.006 2.000 1.713 1.707 1.698 1.689 1.682 1.673 1.650 1.636 1.626 1.613 1.602 1.589

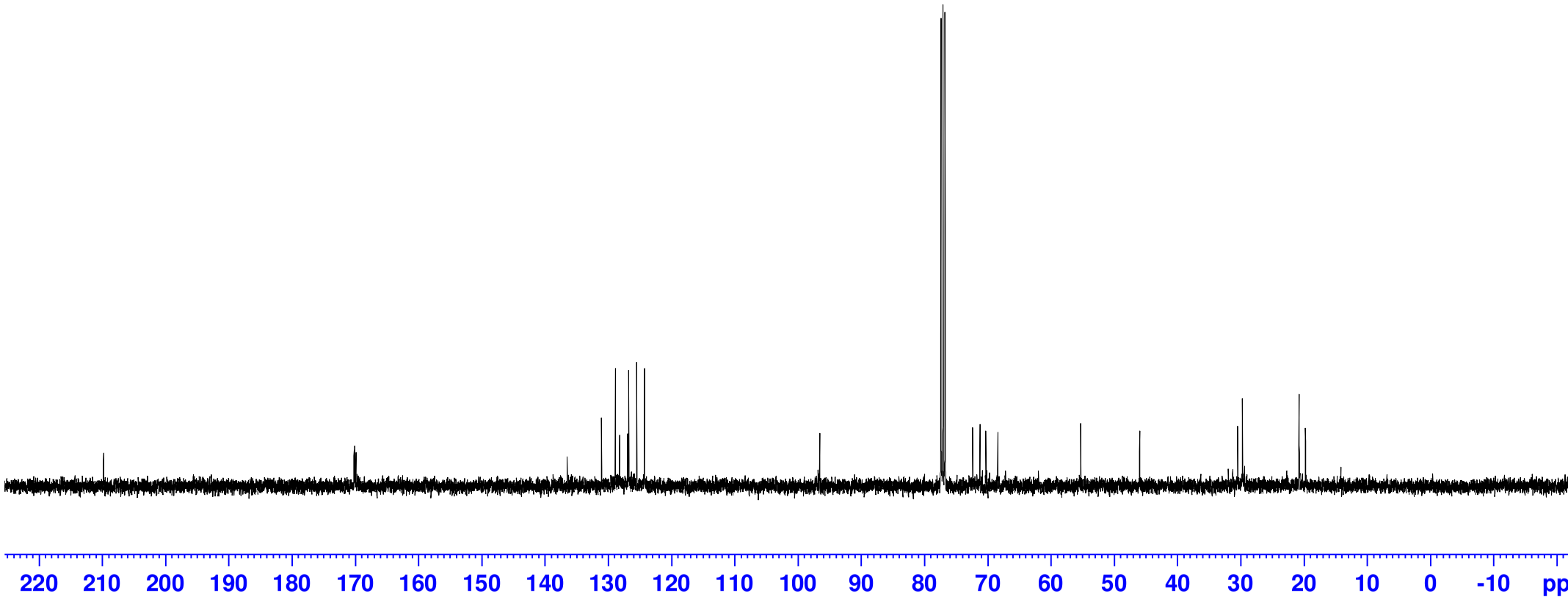


2-(4-(anthracen-9-yl)-4-oxobutyl)-6-methoxytetrahydro-2H-pyran-3,4,5-triyl triac

209.81
170.20
170.09
169.88
136.48
131.08
128.85
128.18
126.93
126.75
125.50
124.25
96.54
72.36
71.19
70.28
68.38
55.27
45.94
30.45
29.70
20.73
19.72

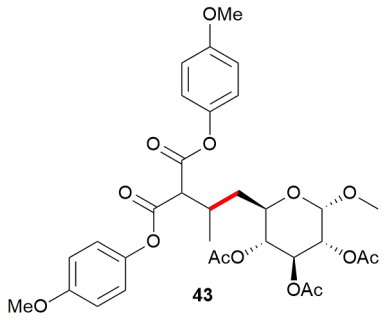


42

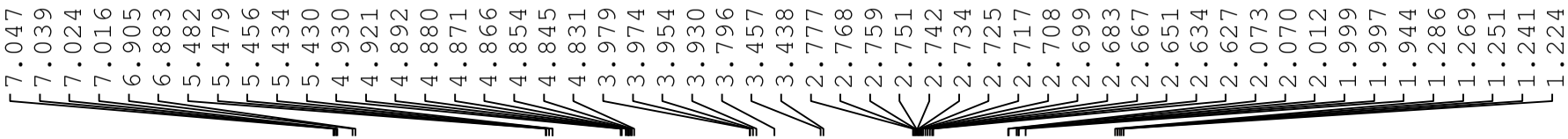


S77

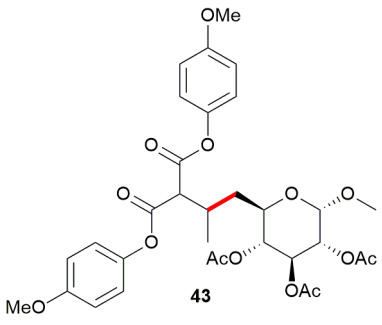
Bis(4-methoxyphenyl) 2-(1-(3,4,5-triacetoxy-6-methoxytetrahydro-2H-pyran-2-yl)propan-2-yl)malonate



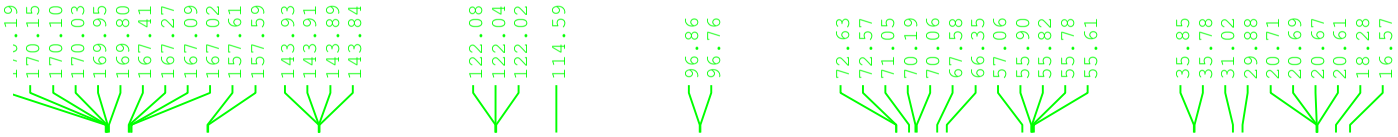
43



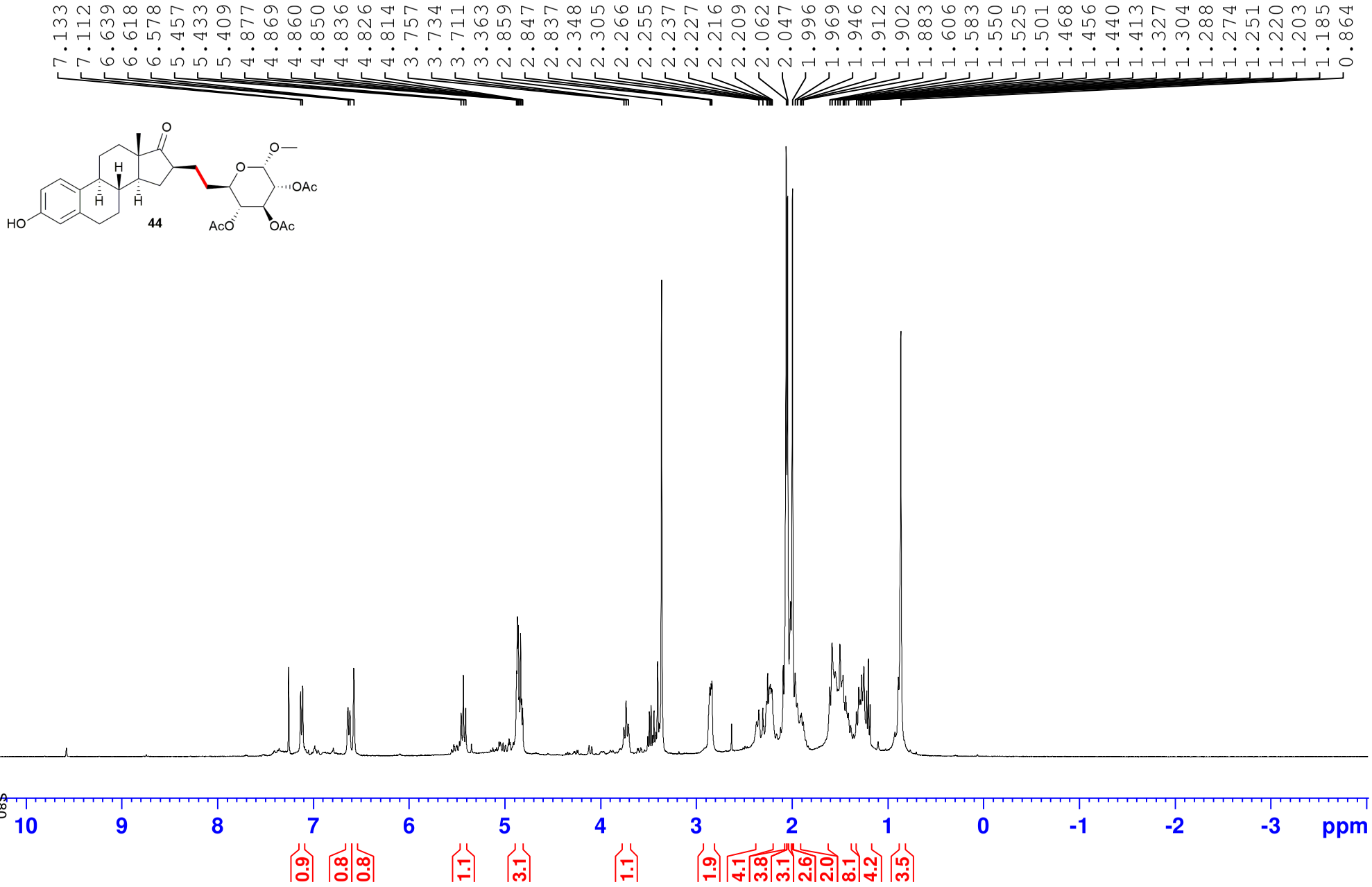
Bis(4-methoxyphenyl) 2-(1-(3,4,5-triacetoxy-6-methoxytetrahydro-2H-pyran-2-yl)propan-2-yl)malonate



43

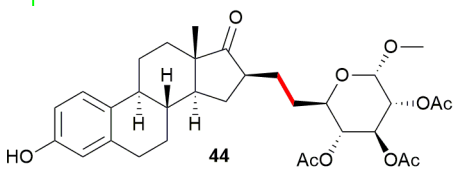


2-(2-(3-hydroxy-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-16-yl)ethyl)-6-methoxytetrahydro-2H-pyran-3,4,5-triyl triacetate



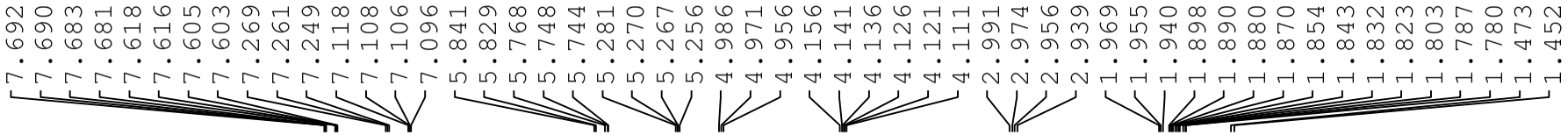
2-(2-(3-hydroxy-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclope
phenanthren-16-yl)ethyl)-6-methoxytetrahydro-2H-pyran-3,4,5-triyl triacetate

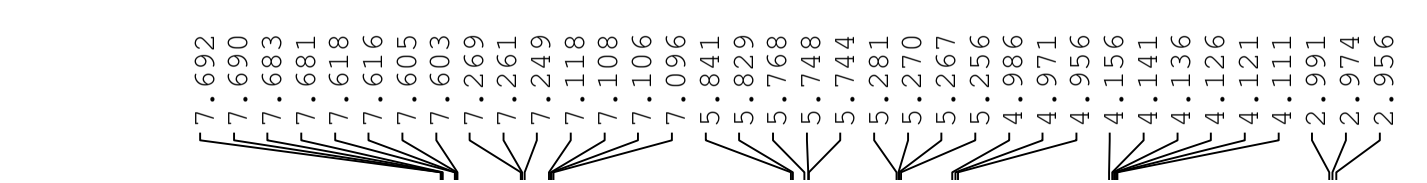
222.31
170.27
170.12
170.03
153.78
137.95
131.87
126.40
115.32
112.87
96.47
72.44
71.19
70.25
68.09
55.30
49.02
48.95
48.51
44.07
37.95
31.89
29.88
29.47
28.59
28.26
26.68
25.85
20.75
20.73
20.68
14.10

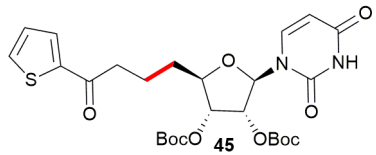


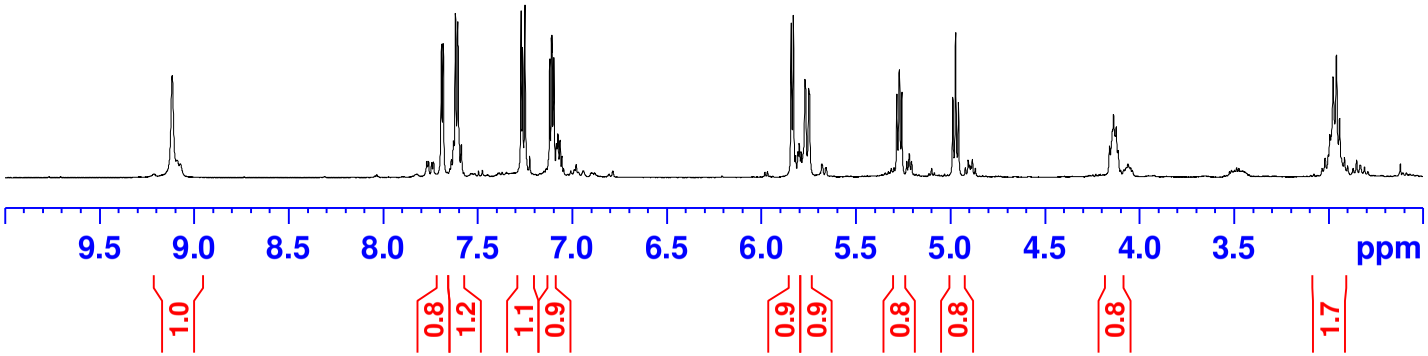
44

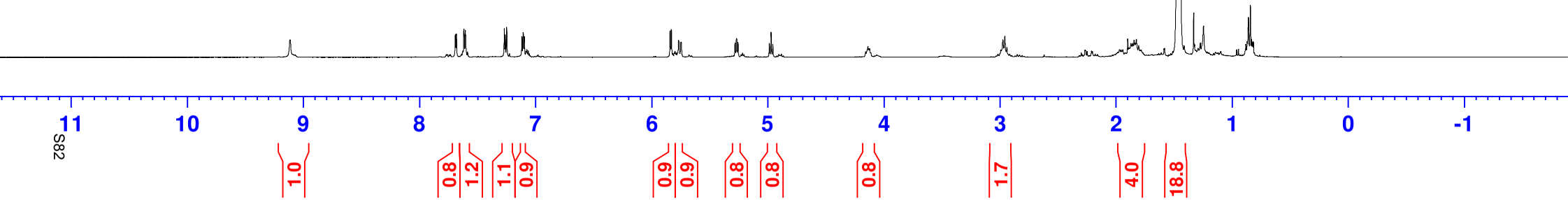
di-tert-butyl (2-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-5-(4-oxo-4-(thiophen-2-yl)butyl) tetrahydrofuran-3,4-diyl) bis(carbonate)





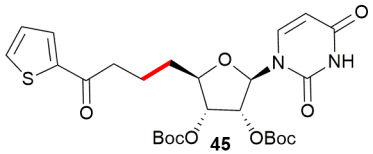




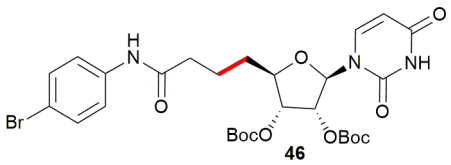


di-tert-butyl (2-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-5-(4-oxo-4-(thiophen-
tetrahydrofuran-3,4-diyl) bis(carbonate)

192.42
162.96
152.22
152.15
149.83
144.19
140.54
133.53
131.78
128.11
103.07
89.15
83.57
83.36
80.78
75.17
75.06
38.40
32.29
27.63
27.60
20.42

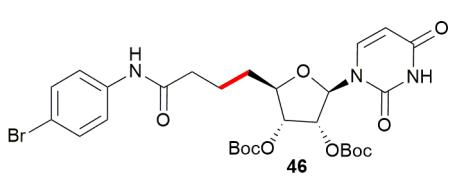


2-(4-((4-bromophenyl)amino)-4-oxobutyl)-5-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)
tetrahydrofuran-3,4-diyl di-tert-butyl bis(carbonate)



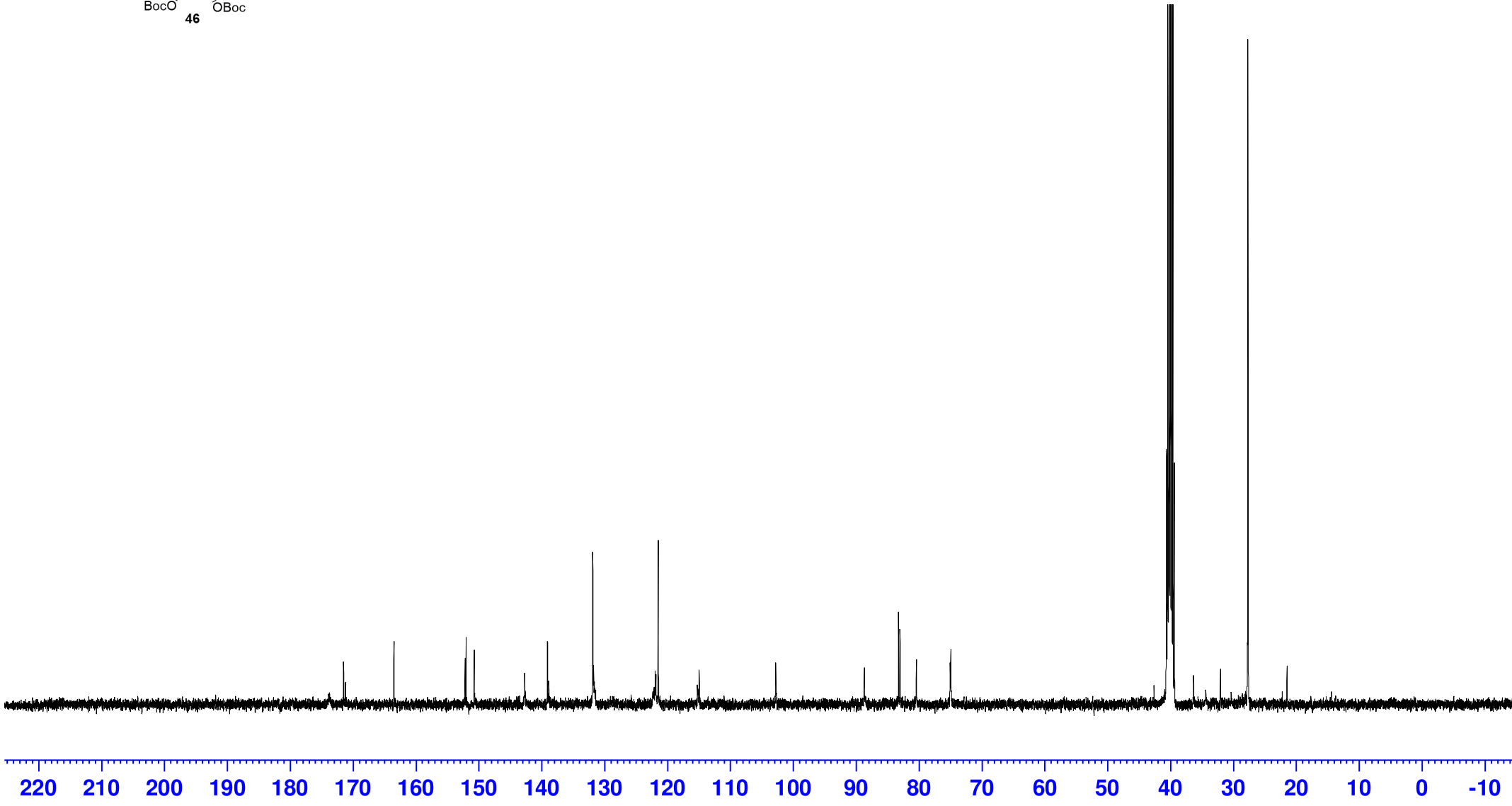
46

2-(4-((4-bromophenyl)amino)-4-oxobutyl)-5-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)
tetrahydrofuran-3,4-diyl di-tert-butyl bis(carbonate)

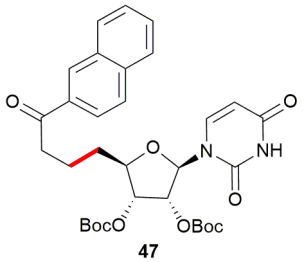


46

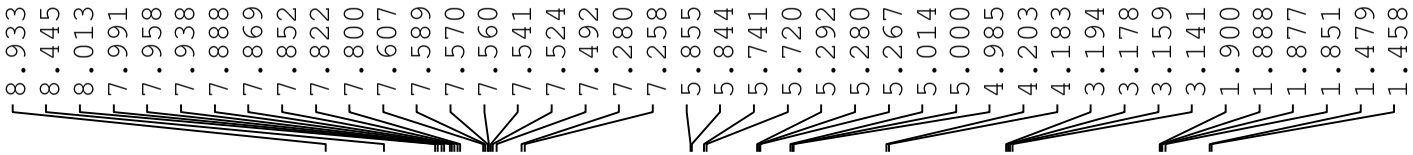
171.51
171.22
163.50
152.18
152.03
150.74
142.71
139.08
138.88
131.90
131.86
121.45
115.26
114.96
102.77
88.67
83.25
83.00
82.97
80.39
75.03
74.91
42.60
36.30
32.01
27.66
21.43

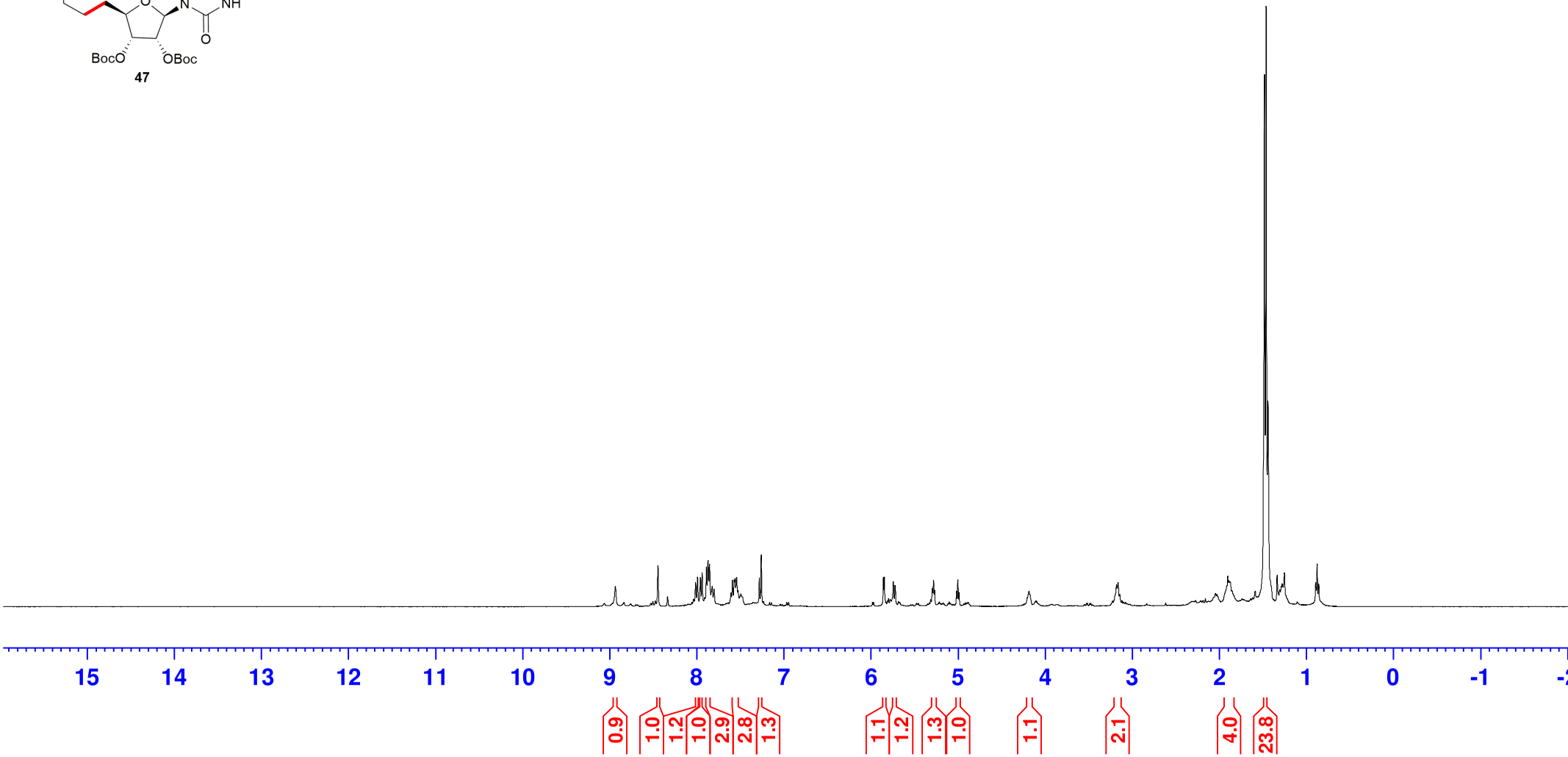


di-tert-butyl (2-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-5-(4-(naphthalen-2-y
tetrahydrofuran-3,4-diyl) bis(carbonate)



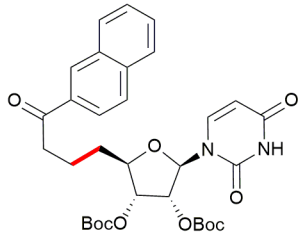
47





di-tert-butyl (2-(2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)-5-(4-(naphthalen-2-y
tetrahydrofuran-3,4-diyl) bis(carbonate)

199.41
162.82
152.26
152.18
149.77
140.49
135.59
134.22
132.53
129.62
129.54
128.48
127.78
127.69
126.81
123.78
103.04
89.11
83.58
83.37
80.85
75.21
75.11
37.69
32.43
27.64
27.61
20.22



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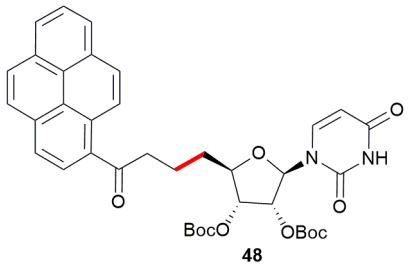
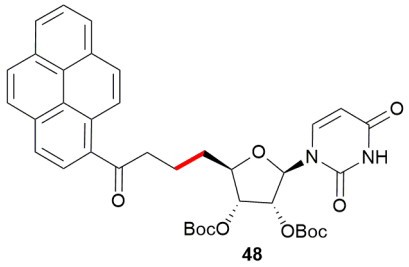
di-tert-butyl (2-(4-(4,5a1-dihydropyren-1-yl)-4-oxobutyl)-5-(2,4-dioxo-3,4-dihy
tetrahydrofuran-3,4-diyl) bis(carbonate)

8.931
8.908
8.811
8.324
8.304
8.274
8.267
8.255
8.248
8.234
8.211
8.185
8.178
8.163
8.088
8.069
8.051
7.284
7.234
7.214
5.850
5.839
5.680
5.660
5.334
5.321
5.309
5.071
5.056
5.041
4.254
4.234
4.211
3.321
3.305
3.293
3.278
2.024
1.998
1.987
1.967
1.953
1.931
1.516
1.493



48

di-tert-butyl (2-(4-(4,5a1-dihydropyren-1-yl)-4-oxobutyl)-5-(2,4-dioxo-3,4-dihy
tetrahydrofuran-3,4-diyl) bis(carbonate)



48