Synthesis of Uniformly ^{13}C-Labeled Polycyclic Aromatic Hydrocarbons

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Experimental methods

General. All reactions were first carried out with natural abundance (NA) starting material in order to optimize the reaction conditions. The NMR spectra of NA-intermediates either matched those of authentic compounds or were consistent with assigned structures. The preparations of the final U-^{13}C-PAHs and key intermediates have been presented in the Experimental Section. All the other procedures and characterization data are included in the supporting information.

All labeled chemicals were purchased from Cambridge Isotope Laboratories Inc. All the other reagents and solvents were used as received or treated by standard methods to ensure purity. NMR spectra were recorded on INOVA-300, 400 or 500 NMR spectrometers in chloroform-*d* unless otherwise noted. Signal assignments in the ^{1}H and ^{13}C NMR spectra of U-^{13}C-labeled compounds are based on published spectra of the NA compounds, where available, and resolved splittings. GCMS was performed on an HP 5972 mass detector coupled with an HP GC 5890. UV/Vis spectra were recorded on Cary 300 UV spectrophotometer.

U-^{13}C-4-Oxo-4-phenylbutanoic acid. U-^{13}C-Succinic (0.726 g 5.95 mmol) acid was refluxed in SO_2Cl_2 (30 mL) for 3 h, excess SO_2Cl_2 was removed under vacuum and the residue suspended in 1,1,2,2-tetrachloroethane (25 mL) cooled in an ice-water bath. Addition of U-^{13}C-bezene (0.5 g 5.95 mmol) was followed by portion-wise addition of fresh $A1C1_3$ (3.5 g, 28 mmol) with stirring**.** The resulting mixture, gradually deepening in color, was stirred overnight at RT with careful exclusion of moisture. The reaction was poured into the mixture of ice (50 g) and conc. HCl (5 mL) and rinsed into a 250 mL flask with water and ether. The solvent and most of the water were removed on Rotavpor™ and the remaining content extracted with ether (5 x 25 mL). The combined organic layers were washed with water (2 x 10 mL), brine (15 mL), and dried with $MgSO_4$. Removal of solvent afforded a yellowish solid (1.115 g, 91 % yield): Mp: 117 – 119.0 °C. The crude product was of sufficient purity to continue to the next step.

U-^{13}C-4-Phenylbutanoic acid. U-^{13}C-4-Oxo-4-phenylbutanoic acid from the previous step (1.115 g, 5.93 mmol) was dissolved in diethylene glycol (18 mL) and hydrazine hydrate (64% aqueous, 1.5 mL) was added. After stirring at RT for 30 min, potassium hydroxide (1.52 g) was added and the mixture was brought to reflux (~120 °C) for 1.5 h. The temperature was gradually increased to distill low boiling material until the bath temperature reached 215 °C and then the reaction was refluxed for 3 h. After cooling, the reaction mixture was poured into ice (50 g), acidified with HC1 to pH 2 and extracted with diethyl ether (5 x 25 mL**).** The combined organic extracts were backwashed with water (2 x 10 mL) and brine (15 mL), dried over $MgSO_4$ and the solvent evaporated to give a white solid (0.98 g. 95% yield): Mp 50 – 52 °C. ^{1}H NMR, Figure S1, (300 MHz) δ 7.18 (bd, J = 158.9 Hz, 5H, Ar), 2.67 (bd, J = 125.7 Hz, 2H, C4H_2), 2.37 (t, J = 123.1 Hz, 2H, C2H_2), 1.97 (p, J = 125.7 Hz, 2H, C3H_2). ^{13}C NMR, Figure S2, (75 MHz) δ 178.5 (d, J = 56.2 Hz, C1), 140.7 – 142.5 (m, C1′), 127.9 – 129.7 (m, C2′, C3′, C5′, C6′), 125.7 – 127.2 (m, C4′), 35.5 (ddd, J = 44.2, 34.3, 3.5 Hz, C4H_2), 33.5 (ddd, J = 55.5, 34.1, 3.0 Hz, C2H_2), 26.6 (t, J = 34.6 Hz, C3H_2). ^{1}H and ^{13}C chemical shifts are identical to those reported.[1,2] This material was of sufficient purity to proceed to the next step.

U-^{13}C-Tetralone (1). U-^{13}C-4-Phenylbutanoic acid (0.984 g, 5.6 mmol) was added to methanesulfonic acid (15 mL) and the mixture was heated to 90 °C for 30 min under nitrogen. The reaction mixture was then poured into ice-water and extracted with ether (3 x 40 mL). The combined organic extracts were washed with dilute sodium bicarbonate (20 mL), water (2 x 15 mL) and brine (15 mL) dried over $MgSO_4$ and filtered. Evaporation of solvent yielded a yellowish liquid (0.835 g, 95 % yield). ^{1}H NMR, Figure S3,, (300 MHz) 8.00 (bd, J = 157.8 Hz, 1H, H8) , 7.48 (db, J = 166.2 Hz, 1H, H7), 7.26 (m, J = 160.2 Hz, 2H, H5, H6), 2.95(bd, J = 123.3 Hz, 2H, C4H_2), 2.64 (bd, J = 126.4 Hz, 2H, C2H_2), 2.12 (bd, J = 132.7 Hz, 2H, C3H_2); ^{13}C NMR, Figure S4, (75 MHz) δ 198.8 (dd J = 48.7, 46.0 Hz, C1), 143.3 – 145.6 (m, C4a), 132.4 – 134.8 (m, C4a, C8a), 129.9 (t, J = 55.2 Hz, C8), 126.8 – 127.8 (m, C5, C7), 39.62 (ddd, J = 41.2, 32.8, 11.1 Hz, C2H_2), 30.1 (dd, J = 42.4, 32.5 Hz, C4H_2),

23.71 (t, J = 32.6 Hz, C3H$_2$). ^{13}C and ^{1}H NMR assignments are based on published NMR spectra of NA-tetralone.[3]

U-^{13}C-Tetralin (2). Wolff-Kishner reduction of U-^{13}C-tetralone (830 mg, 5.32 mmol) by the procedure described above for 4-oxo-4phenylbutanoic acid afforded a brownish residue which was further purified by chromatography (SiO_2, hexane) to give a colorless oil (620 mg. 80% yield). Due to the volatility of tetralin under the reaction conditions, precautions are necessary to prevent loss due evaporation. ^{1}H NMR. Figure S5, (300 MHz) δ 7.07 (bd, J = 150.4 Hz, 4H, Ar), 2.77 (bd, , J = 126.1 Hz, 4H, C1H_2, C4H_2), 1.80 (bd, J = 123.3 Hz, 4H, C2H_2, C3H_2). ^{13}C NMR, Figure S6, (75 MHz) δ 136.86 – 137.59 (m, C4a, C8a), 128.83 – 129.76 (m, C5, C8), 125.40 – 126.18 (m, C6, C7), 29.36 – 30.30 (m, C1H$_2$, C4H$_2$), 23.13 – 24.16 (m, C2H$_2$, C3H$_2$). ^{1}H NMR shifts corresponded to those of NA-tetralin.[4] The ^{13}C shifts are based on the ^{13}C NMR spectrum of NA-tetralin.[5]

U-^{13}C-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid. U-^{13}C-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid was prepared by the procedure described above for the preparation of 4-oxo-4-phenylbutanoic acid. U-^{13}C-tetralin (600 mg 4.2 mmol) with U-^{13}C-succinic acid (0.439 g, 4.2 mmol) afforded a yellowish solid (0.991 g 90 % yield): Mp 114 – 116 °C. ^{1}H NMR, Figure S7, (300 MHz) δ 7.68 (bd, J = 158.7 Hz, 2H, H1′, H3′ or H4′), 7.13 (bd, J = 161.9 Hz, 1H, H3′ or H4′), 3.27 (bd, J = 139.4 Hz, 2H, C3H_2), 2.79 (bd, J = 131.2 Hz, 6H, C2H_2, C5′H_2, C8′H_2), 1.81 ((bd, J = 123.7 Hz, 4H, C6′H_2, C7′H_2).^{13}C NMR, Figure S9, (75 MHz) δ 198.2 (dd, J = 54.2, 40.2 Hz, C4), 178.7 (d, J = 56.4 Hz, C1), 143.9 (ddt, J = 55.6, 42.5, 7.3 Hz, C4′a), 137.9 (dt, J = 55.2, 42.5 Hz, C8′a), 134.4 (q, J = 56.5 Hz, C2′), 129.8 (t, J = 55.2 Hz, C1′ or C3′), 129.4 (t, J = 55.1 Hz, C1′ or C3′), 125.4 (t, J = 56.3 Hz, C4′), 33.5 (dt, J = 40.4, 13.4 Hz, C3H$_2$), 29.3 – 30.5 (m, C5′H$_2$, C8′H$_2$), 8.4 (dd, J = 55.0, 39.5 Hz, C2H$_2$), 22.7 – 23.8 (m, C6′H$_2$, C7′H$_2$).

For NA-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid: ^{1}H NMR, Figure S7 inset, (500 MHz) δ 7.68 (br, 2H, H1′ ,H3′ or H4′), 7.13 (d, J = 8 Hz, 1H, H3′ or H4′), 3.27 (t, J = 6 Hz, 2H, C3H_2), 2.79 (m, 6H, C2H_2, C5′H_2, C8′H_2), 1.81 (br, 4H, C6′H_2,C7′H_2). ^{13}C NMR Figure S8, (100 MHz) δ 198.0(C4), 179.2(C1), 143.7(C4a′), 137.7(C2′), 134.1(C8a′), 129.6(C1′), 129.2(C3′), 125.3(C4′), 33.2(C3H$_2$), 29.8 & 29.7(C5H$_2$,C8H$_2$), 28.4(C2H$_2$), 23.2 & 23.0(C6H$_2$,C7H$_2$).

NA-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid. Procedure was the same as described above for the reduction of U-^{13}C-4-oxo-4-phenylbutanoic acid. ^{1}H NMR Figure S10, (400 MHz) δ 6.97 (d, J = 7.7 Hz, 1H), 6.89 (d, J = 8.7 Hz, 1H), 2.73 (br 4H, C5′H_2,C8′H_2), 2.59 (t, 2H, J = 7.5 Hz, C4H_2), 2.36 (2H, t, J = 7.5 Hz, C2H_2), 1.92 (2H, q, J = 7.3Hz, C3H_2), 1.76 (br 4H, C6′H_2, C7′H_2). Chemical shifts are in agreement with the published spectrum[6]; signal assignments are tentative. ^{13}C NMR, Figure S11, (100 MHz) δ 180.3 (C1), 138.4(C2′), 137.3(C8a′), 135.0(C4a′), 129.3 (C1′,C4′), 125.8(C3′), 34.8(C4H$_2$), 33.7(C2H$_2$), 29.6 & 29.2(C5H$_2$,C8H$_2$), 26.5(C3H$_2$), 23.5 & 23.4(C6H$_2$,C7H$_2$). ^{1}H NMR shifts are from the published spectrum.[6] ^{13}C shift assignments are based on 6-butanyltetralin.[7]

U-^{13}C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (**3**). Compound **3** was prepared in 92% yield by reduction of U-^{13}C-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid. ^{1}H NMR, Figure S12, (300 MHz) δ 6.97 (bd, J = 150.5 Hz, 1H, H3′ or H4′), 6.89 (bd, J =150.5 Hz, 2H, H1′, H3′ or H4′). 2.74 (bd, J = 125.2 Hz, 4H, C5′H_2, C8′H_2), 2.60 (bd, J = 131.2 Hz, 2H, C4H_2), 2.37 (bd, J = 127.2 Hz, 2H, C2H_2), 1.95 (bd, J = 128.7 Hz, 2H, C2H2), 1.82 (bd, J = 125.2 Hz, 4H, C6′H_2, C7′H_2).^{13}C NMR, Figure S13, (75 MHz) δ 179.4 (dd, J = 55.2, 3.1 Hz, C1), 134.1 – 139.7 (m, C2′, C4a′, C8a′), 129.5 (t, J = 57.2 Hz, C1′), 125.9 (t, C4′), 35.0 (ddq, J = 41.2, 34, 3.3 Hz, C4H$_2$), 33.7 (ddd, J = 55.5, 34.2, 3.6 Hz, C2H$_2$), 29.0 – 30.2 (m, C5′H$_2$, C8′H$_2$) 26.7 (t, J = 34.5 Hz, C3H$_2$), 23.1 – 24.2 (m, C6′H$_2$, C7′H$_2$).

NA-3,4,5,6,7,8-hexahydroanthracen-1(2H)-one. Procedure was the same as that described in the Experimental Section for the preparation of **4**. The ^{1}H NMR (Figure S14) and ^{13}C NMR (Figure S15) spectra are identical to the published spectrum.[8] For compound **4,** ^{1}H NMR, Figure S16; ^{13}C NMR, Figure S17.

U-^{13}C-1,2,3,4,5,6,7,8-octahydroanthracene. U-^{13}C-1,2,3,4,5,6,7,8-Octahydroanthracene was prepared from **4** in 80 % yield by the procedure described above for tetralin: Mp 70 – 72 °C. ^{1}H NMR, Figure S18, (300 MHz) δ 6.78 (bd, J = 149.3 Hz, 2H, H5, H10), 2.70 (bd, J = 125.9 Hz, 8H, C1H_2, C4H_2, C5H_2, C8H_2), 1.76 (bd, J = 121.9 Hz, 8H, C2H_2, C3H_2, C6H_2, C7H_2). ^{13}C NMR, Figure S19, (75 MHz) δ 134.0 – 135.4 (m, C4a, C8a, C9a, C10a), 129.8 – 130.6 (m, C9, C10), 29.0 – 29.9 (m, C1H$_2$, C4H$_2$, C5H$_2$, C8H$_2$), 23.6 – 24.1 (m, C2H$_2$, C3H$_2$, C6H$_2$, C7H$_2$). For NA-1,2,3,4,5,6,7,8-octahydroanthracene, the ^{1}H NMR, Figure S18 inset, is identical to the published spectrum.[9]

Methyl U-^{13}C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate. To the solution of **3** (1.764 g, 7.17 mmol) in methanol (30 mL) was added a few drops of sulfuric acid and the mixture was refluxed over night. After cooling, the mixture was diluted with ether (100 mL) and washed with dilute aqueous $NaHCO_3$, water and brine, then dried over $MgSO_4$. After evaporation of solvent, a yellowish oil was obtained and purified by silica gel chromatography (hexane/ethyl acetate, 15:1) to afford ester **3**. (1.494 g, 85 %). ^{1}H NMR, Figure S23, (300 MHz) δ 6.98 (bd, J = 153.3 Hz, 2H, H3′, H4′), 6.87 (bd, J = 152.4 Hz, 1H, H1′), 3.66 (s, 3H, CH_3), 2.73 (bd, J = 127.4 Hz, 4H, C5′H_2, C8′H_2), 2.56 (bd, J = 126.2 Hz, 2H, C4H_2), 1.98 (bd, J = 119.0 Hz, 2H, C2H_2), 1.90 (bd, J =

125.2 Hz, 2H, C3H_2), 1.76 (bd, J = 123.9 Hz, 4H, C6′H_2, C7′H_2). ^{13}C NMR, Figure S24, (75 MHz) δ 174.1 (d, J = 58.2 Hz, C1), 133.6 – 139.5 (m, C2′, C4′a, C8′a), 129.2 (t, J = 55.7 Hz, C1′, C4′), 125.7 (t, J = 59.6 Hz, C3′), 34.9 (ddq, J = 44.2, 34.4, 3.6 Hz, C4H_2), 33.6 (ddd, J = 57.5, 34.6, 3.4 Hz, C2H_2), 28.3 – 30.3 (m, C5′H_2, C8′H_2) 26.7 (t, J = 32.5 Hz, C3H_2), 22.9 – 23.9 (m, C6′H_2, C7′H_2).

NA Methyl 4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate. NA-Methyl 4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate was synthesized by the procedure described above for the U-^{13}C compound. ^{1}H NMR, Figure S22, (300 MHz) δ 6.98 (d, J = 7.6 Hz, 1H, H3′or H4′), 6.88 – 6.91 (m, 2H, H1′, H3′or H4′), 3.66 (s, 3H, CH_3), 2.73 (br, 4H, C5′H_2 , C8′H_2), 2.57 (t, J = 7.5 Hz, C4H_2), 1.98 (t, J = 7.5 Hz, C2H_2), 1.90 (p, J = 7.5 Hz, C3H_2), 1.72 – 1.80 (m, 4H, C6′H_2 , C7′H_2).

NA-Methyl 4-(naphthalen-2-yl)butanoate: Procedure was the same as that described in the Experimental Section for the synthesis of **12.** ^{1}H NMR, Figure S25, (300 MHz) δ 7.76 – 7.82 (m, 3H, H4′, H5′, H8′), 7.62 (s, 1H, H1′), 7.40-7.45 (m, 2H, H6′, H7′), 7.25 (d, J = 8 Hz, 1H, H3′), 3.67 (s, 3H, CH_3), 2.82 (t, J = 7.7 Hz, 2H, C4H_2), 2.37 (t, J = 7.7 Hz, 2H, C2H_2), 2.08 (p, J = 7.7 Hz, 2H, C3H_2). ^{13}C NMR, Figure S26, (100 MHz) δ 174.1(C1), 139.1(C2′), 133.8(C4a′), 132.3(C8a′), 128.2, 127.8, 127.4, 126.8, 126.2, 125.4, 51.7(CH$_3$), 35.5(C4H$_2$), 33.6(C2H$_2$), 26.6(C3H$_2$). ^{1}H NMR and ^{13}C NMR assignments are based on 2-methyl naphthalene.[10]

Methyl U-^{13}C- 4-(naphthalen-2-yl)butanoate (9). Compound **9** was prepared by the procedure described in the Experimental Section for synthesis of **12.** Compound **11** was isolated as an oil in 85% yield. ^{1}H NMR, Figure S27, (300 MHz) δ 7.76 (bd, J = 150.1 Hz, 3H, H4′, H5′, H8′), 7.59 (bd, J = 157.1 Hz, 1H, H1′), 7.40 (bd, J = 152.8 Hz, 2H, H6′, H7′), 7.30 (bd, J = 153.8 Hz, 1H, H3′), 3.65 (d, J = 3.6 Hz, 3H, CH_3), 2.82 (bd, J = 127.8 Hz, 2H, C4H_2), 2.37 (bd, J = 126.8 Hz, 2H, C2H_2), 2.08 (bd, J = 128.6 Hz, 2H, C3H_2). ^{13}C NMR, Figure S28, (75 MHz) δ 173.8 (d, J = 58.2 Hz, C1), 138.6 – 139.4 (m, C2′), 131.1 – 134.7 (m, C4a′, C8a′), 124.5 – 128.9 (m, C1′, C3′, C4′). 34.8 (ddq, J = 44.6, 34.2, 3.4 Hz, C4H$_2$), 33.4 (ddd, J = 57.3, 34.5, 3.8 Hz, C2H$_2$), 26.4 (t, J = 34.6 Hz, C3H$_2$).

NA-2,3-dihydrophenanthren-4(1*H*)-one. Procedure was the same as that described in the Experimental Section for the synthesis of **4.** ^{1}H NMR, Figure S29, (400 Hz) δ 9.41 (d, J = 8.8 Hz, 1H, H5), 7.92 (d, J = 8.4 Hz, 1H, H9), 7.80 (d, J = 8.1 Hz, 1H, H8), 7.62 (ddd, J = 8.1, 6.8, 1.5 Hz, 1H, H6), 7.49 (ddd, J = 8.6, 6.8, 1.2 Hz, 1H, H7), 7.32 (d, J = 8.4 Hz, 1H, H10), 3.12 (t, J = 6.2 Hz, 2H, C1H_2), 2.79 (t, J = 7.2 Hz, 2H, C3H_2), 2.20 (q, J = 6.4 Hz, 2H, C2H_2). ^{13}C NMR, Figure S30, ^{1}H NMR and ^{13}C NMR shift assignments are from published data.[11]

U-^{13}C-2,3-dihydrophenanthren-4(1*H*)-one (7). Compound **7** was prepared in 98% yield as described above for **4**. ^{1}H NMR, Figure S31, (500 MHz) δ 9.41 (bd, J = 163.8 Hz, 1H, H5), 7.92 (bd, J = 160.4 Hz, 1H, H9), 7.80 (bd, J = 163.7 Hz, 1H, H8), 7.62 (bd, J = 162.0 Hz, 1H, H6), 7.49 (bd, J = 164.2 Hz, 1H, H7), 7.32 (bd, J = 160.1 Hz, 1H, H10), 3.12 (bd, J = 130.2 Hz, 2H, C1H_2), 2.79 (bd, J = 127.7 Hz, 2H, C3H_2), 2.20 (bd, J = 130.1 Hz, 2H, C2H_2). ^{13}C NMR, Figure S32, (100 MHz) δ 200.7 (dd, J = 51.1, 40.8 Hz, C4), 143.9 (dt, J = 54.7, 40.8 Hz, C10a), 131.5 (t, J = 54.3 Hz, C9), 130.0 (q, J = 50.8 Hz, C8a), 128.5 (q, J = 53.8 Hz, C4b), 122.3 – 126.7 (m, Ar), 38.3(ddd, J = 40.2, 31.8, 11.6 Hz, C3H$_2$), 28.8 (dd, J = 40.9, 35.6 Hz, C1H$_2$), 20.2 (t, J = 32.4 Hz, C2H$_2$).

NA-1,2,3,4-tetrahydrophenanthrene. Procedure was the same as that described in the Experimental Section for the synthesis of tetralin**.** ^{1}H NMR, Figures S33, (400 MHz) δ 7.95 (d, J = 8.4 Hz, 1H, H5), 7.78 (d, J = 8.3 Hz, 1H, H8), 7.60 (d, J = 8.4 Hz, 1H, H9), 7.48 (t, J = 6.9 Hz, 1H, H6), 7.41 (t, J = 6.89 Hz, 1H, H7), 7.19 (d, J = 8.4 Hz, 1H, H10). 3.11 (t, J = 6.5 Hz, 2H, C4H_2), 2.91 (t, J = 6.2 Hz, 2H, C1H_2), 1.95 (m, 2H, C3H_2), 1.87 (m, 2H, C2H_2). ^{13}C NMR, Figures S34, (100 MHz) δ 134.52, 132.74, 132.25, 131.7, 128.6, 128.3, 126.2, 125.8, 125.9, 123.0, 30.7, 25.9, 23.5, 23.2. The ^{1}H and ^{13}C NMR spectra are identical to published spectra with assignments.[11]

U-^{13}C-1,2,3,4-tetrahydrophenanthrene. U-^{13}C-1,2,3,4-Tetrahydrophenanthrene was prepared from **9** by the procedure described above for the reduction of U-^{13}C-tetralone to U-^{13}C-tetralin and isolated as an oil in 88 % yield. ^{1}H NMR, Figure S35, (400 MHz) δ 7.95 (bd, J = 150.4 Hz, 1H, H5), 7.78 (bd, J = 153.3 Hz, 1H, H8), 7.60 (bd, J = 155.4 Hz, 1H, H9), 7.48 (md, J = 166.9 Hz, 2H, H6, H7), 7.19 (bd, J = 160.4 Hz, 1H, H10). 3.11 (bd, J = 126.5 Hz, 2H, C4H_2), 2.91 (bd, J = 12 8.2 Hz, 2H, C1H_2), 1.95 (bd, J = 126.7 Hz, 2H, C3H_2), 1.87 (bd, J = 124.7 Hz, 2H, C2H_2). ^{13}C NMR, Figure S36, (100 MHz) δ 122.7 – 134.5 (m, Ar), 30.2 (m, C1H$_2$), 25.4 – 26.2 (m, C4H$_2$), 22.7 – 23.8 (m, C2H$_2$, C3H$_2$).

NA-Phenanthrene. For procedure see the Experimental Section. ^{1}H NMR, Figure S37 inset, (300 MHz) δ 8.70 (d, J = 8.2 Hz, 2H, H4, H5), 7.87 – 7.91 (m, 2H, H1, H8), 7.74 (s, 2H, H9, H10), 7.57 – 7.68 (m, 4H, H2, H3, H6, H7). The ^{1}H and ^{13}C NMR spectra of NA-phenanthrene are identical to published spectra with assignments.[12] For U-^{13}C-phenanthrene, ^{1}H NMR, Figure S37; ^{13}C NMR, Figure S38.

NA-Ethyl 2-(phenanthren-4-yl)acetate. The procedure is identical to that described in the Experimental Section for synthesis of ethyl U-^{13}C-2-(phenanthren-4-yl)acetate. ^{1}H NMR spectrum, Figure S39, (300 MHz) δ 8.63 (m, 1H, H5′), 7.92 – 7.94 (m, 1H, H8′), 7.85-7.88 (m, 1H, H1′), 7.73 (br, 2H, Ar), 7.57 – 7.66 (m, 4H, Ar), 4.48 (s, 2H, ArCH_2), 4.28 (q, J = 7.2 Hz, 2H, OCH_2), 1.26 (t, J = 7.1 Hz, 3H, CH_3). Assignments are based on 4-Me-phenanthrene.[13] For ethyl U-^{13}C-2-(phenanthren-4-yl)acetate (**12**). ^{1}H NMR, Figure S40; ^{13}C NMR, Figure S41.

U-^{13}C-Pyrene (15). The procedure is described in the Experimental Section. ^{1}H NMR, Figure S42; ^{13}C NMR, Figure S43. The ^{1}H (Figure S42 inset) and ^{13}C NMR spectra of NA-Pyrene are identical to published spectra. [12]

U-^{13}C-Phthalic acid (14). The procedure is described in the Experimental Section. ^{1}H NMR, Figures S44. For NA-Phthalic acid: ^{1}H NMR, Figure S44 inset. (300 MHz, d_6-DMSO) δ 13.26 (br, 2H, COOH), 7.63–7.69 (m, 2H, H3, H6), 7.56 – 7.61 (m, 2H, H4, H5); ^{13}C NMR (75 MHz, d_6-DMSO) δ 169.3, 133.4, 131.4, 128.9.

U-^{13}C-Naphthalene (6). The procedure is described in the Experimental Section. ^{1}H NMR, Figure S45; ^{13}C NMR, Figure S46. For NA-Naphthalene**:** ^{1}H NMR, Figure S45 inset, (300 MHz) δ 7.84 (dd, J = 6.2, 3.6 Hz, 4H, H1, H4, H5, H8), 7.49 (dd, J = 6.2, 3.6 Hz, 4H, H2, H3, H6, H7). The ^{1}H and ^{13}C NMR shift assignments are from published spectra.[12]

NA-Benz[*a*]anthracene. The procedure is identical to that described in the Experimental Section for synthesis of U-^{13}C-benz[*a*]anthracene (**16**). ^{1}H NMR, Figure S47, (500 MHz) δ 9.16 (s, 1H, H12), 8.83 (d, J = 8.2 Hz, 1H, H1), 8.36 (s, 1H, H7), 8.11 – 8.12 (m, 1H, H8), 8.03 – 8.05 (m, 1H, H11), 7.77 – 7.86 (m, 2H, H4, H5), 7.53 – 7.56 (m, 5H, H2, H3, H6, H9, H10). ^{13}C NMR, Figure S48, (100 MHz) δ 132.4, 132.4, 131.1, 131.0, 129.9, 129.1, 128.9, 128.2, 127.8, 127.5, 127.3, 127.2, 126.2, 126.1, 123.4, 122.0. The ^{1}H and ^{13}C NMR shift assignments are from published spectra.[14,15] For compound **16**, ^{1}H NMR, Figure S49; ^{13}C NMR, Figure S50.

NA-4-bromo-1,2-dihydronaphthalene. Triphenyl phosphite (1.3 mL, 5 mmol) was dissolved in anyhdrous dichloromethane (10 mL) cooled to 78 °C under Ar, bromine (0.28 mL, 6 mmol) was added dropwise followed by DIPEA (0.5 mL, 4 mmol) and tetralone (550 mg, 4 mmol). The reaction mixture was stirred for 18 h while warming to ambient temperature, and then refluxed for an additional 2 h. After cooling and concentration, the reaction mixture waspurified by chromatography (SiO_2, hexane) to yield 4-bromo-1,2-dihydronaphthalene (0.78 g, 98 %). ^{1}H NMR (300 MHz) δ 7.54 (d, J = 7.6 Hz, 1H, H5), 7.15 – 7.25 (m, 2H, , H6, H7), 7.07 (d, J = 7.2 Hz, 1H, H8), 6.42 (t, J = 3.2 Hz, 1H, H3), 2.82 (t, J = 7.8 Hz, 2H, C1H_2), 2.34 (AB, J = 7.6 Hz, 2H, C2H_2).^{13}C NMR (75 MHz) δ 136.7, 133.5, 131.2, 128.7, 127.7, 127.2, 126.9, 121.8, 28.1, 25.8. The ^{1}H NMR and ^{13}C NMR spectra are identical to the published spectra.[16]

NA-1-Bromonaphthalene (17). 4-Bromo-1,2-dihydronaphthalene from the previous reaction (477 mg, 2.3 mmol), was dissolved in benzene (10 mL), and following addition of DDQ (0.529 g, 2.6 mmol), the mixture was refluxed for 4 h. After cooling, the reaction mixture was filtered and the filter washed with benzene. The concentrated filtrate was purified by chromatography (Al_2O_3, benzene) to yield (**19**,0.344 g, 72 %). ^{1}H NMR (300 MHz) δ 8.22 (d, J = 8 Hz, 1H, H8), 7.75 – 7.82 (m, 3H, H2, H4, H5), 7.48 – 7.62 (m, 2H, H6, H7), 7.26 – 7.31 (m, 1H, H3). ^{13}C NMR (75 MHz) δ 135.1, 132.4, 130.3, 128.7, 128.4, 127.8, 127.5, 127.2, 126.6, 123.3. The ^{1}H NMR spectrum is identical to the published spectrum. [17]

NA-Naphthalen-1-ylboronic acid (19). A solution of 1-bromonaphthalene (430 mg, 2.1 mmol) in THF (5 mL) under Ar was treated dropwise at -78 ^{o}C with *n*-butyllithium (2.6 ml, 4.1 mmol, 1.6 M in hexane). The reaction was stirred at -78 °C for 45 min, and then triisopropylborate (0.99 mL, 4 mmol) was added in one portion. The resulting mixture was stirred at -78 °C for 30 min and then at room temperature for 1 h. Five mL of 10% HCl and ether (50 mL) were added and the aqueous layer extracted with ether. and The combined organic extracts were washed with brine and dried over $MgSO_4$. Evaporation of the solvent yielded **19** as a solid (245 mg, 72 %). ^{1}H NMR (300 MHz) δ 8.58 (s, 1H), 7.86 – 7.88 (m, 3H), 7.44 – 7.48 (m, 3H). The ^{1}H NMR spectrum is identical to the published spectrum. [18]

U-^{13}C-4-phenyl-1,2-dihydronaphthalene (20). U-^{13}C-bromobenzene (0.5 g, 3.1 mmol) was added to Li (47 mg, 6.7 mmol) in ether at rt under argon. After stirring for 20 min, the solution of **1** (200 mg, 1.28 mmol) in ether (1 mL) was added and stirred for 1 h. The reaction mixture was quenched with ice water and diluted with ether. After work up, the crude product was dissolved in benzene (20 mL) and refluxed for 2 h with toluenesulfonic acid (20 mg). The reaction mixture was washed with dilute aqueous Na_2CO_3, water and brine, dried over Na_2SO_4. After evaporation of solvent, the residue was purified by chromatography (SiO_2, hexane) to give **22**. (266 mg, 90 % yield). ^{1}H NMR, Figure S51, (300 MHz) δ 6.77 – 7.81 (m, 9H, Ar-H), 6.10 (bd, J = 155.7 Hz, 1H, H3), 2.86 (bd, J = 126.7 Hz, 2H, C1H_2), 2.42(bd, J = 126.7 Hz, 2H, C2H_2). ^{13}C NMR, Fifure S53, (75 MHz) δ 134.3 – 142.2 (m, C4, C1′, C4a, C8a), 125.3 – 129.9 (m, Ar), 28.8 (t, J = 38.5 Hz, *C*1H$_2$), 23.9 (t, J = 37.6 Hz, *C*2H$_2$). For NA-4-phenyl-1,2-dihydronaphthalene: ^{1}H NMR Figure 51 inset, (300 MHz) δ 7.34 (br, 5H, Ar), 6.99 – 7.17 (m, 4H, Ar), 6.07 (AB, J = 4.5 Hz, 1H, H3), 2.80 – 2.86 (m, 2H, C1H_2), 2.35 – 2.41 (m, 2H, C2H_2). ^{13}C NMR, Figure S52, (75 MHz) δ 141.3, 140.4, 137.3, 135.6, 129.3, 128.7, 128.1, 126.7, 125.9, 28.8, 24.1. ^{1}H and ^{13}C NMR shift assignments are from published spectra.[19, 20]

U-^{13}C-2-Iodo-1-phenylnaphthalene (21). To **20** (266 mg, 1.2 mmol) and iodine (426 mg, 1.7 mmol) in acetonitrile (5 mL), was added AgOTf (330 mg, 1.3 mmol) in acetonitrile (3 mL) at rt. The mixture was stirred for 1 h, diluted with ether then washed with $Na_2S_2O_3$ and dried over $MgSO_4$. After evaporation of solvent, the residue was dissolved in benzene. DDQ (545 mg, 2.4 mmol) was added and the mixture was refluxed overnight. After cooling, the mixture was filtered, washed with benzene, and the combined filtrate concentrated and purified

by chromatography (neutral Al_2O_3, hexane/ethyl acetate, 25:1) to give **21** (346 mg, 83% yield), which was used directly in the next step. ^{1}H and ^{13}C NMR spectra are identical to published spectra.[21]

U-^{13}C-Fluoranthene (**22**). The procedure is described in the Experimental Section. ^{1}H NMR and ^{13}C NMR spectra, Figures S54, S56. For NA-fluoranthene: ^{1}H NMR, S54 inset, (300 MHz) δ 7.91 – 7.97 (m, 4H, H1, H6, H7*H*, H10*H*), 7.86 (d, 2H, H3, H4), 7.65 (t, J = 8 Hz, 2H, ,H2, H5), 7.38 – 7.41 (m, 2H, H8, H9). ^{13}C NMR, Figure S55, δ 139.7, 137.2, 130.2, 128.2, 127.8, 126.9, 121.7, 120.3. The ^{1}H and ^{13}C NMR shift assignments are from published spectra.[12, 22]

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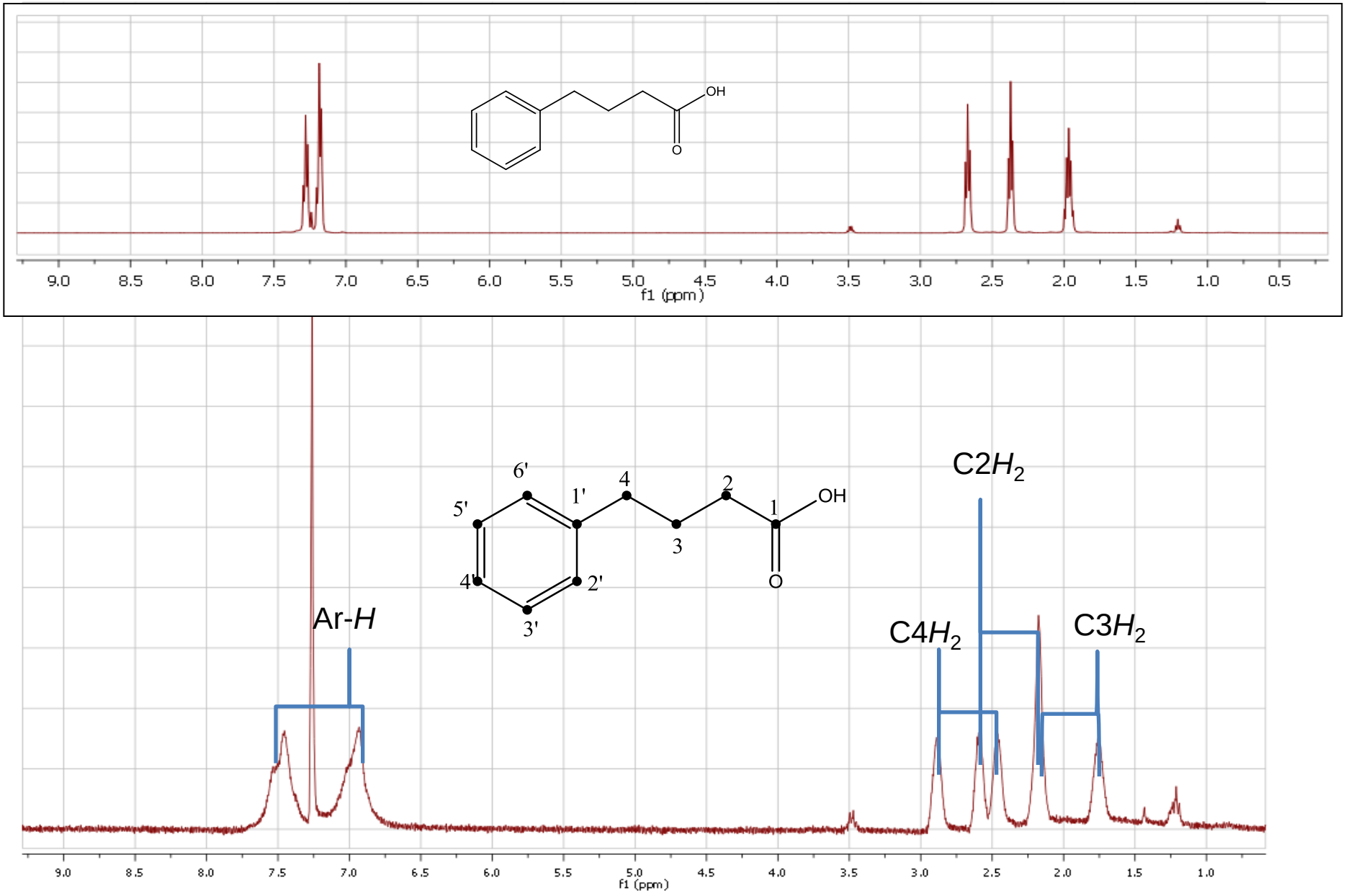


Figure S1. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-4-phenylbutanoic acid. (Inset: ^{1}H NMR NA-4-phenylbutanoic acid. Chemical shifts identical to report: Auger P.; Malaiyandi, M.; Wightman, R. H. *J. Labelled Compounds Radiopharm*. **1993**, *33*, 263.)

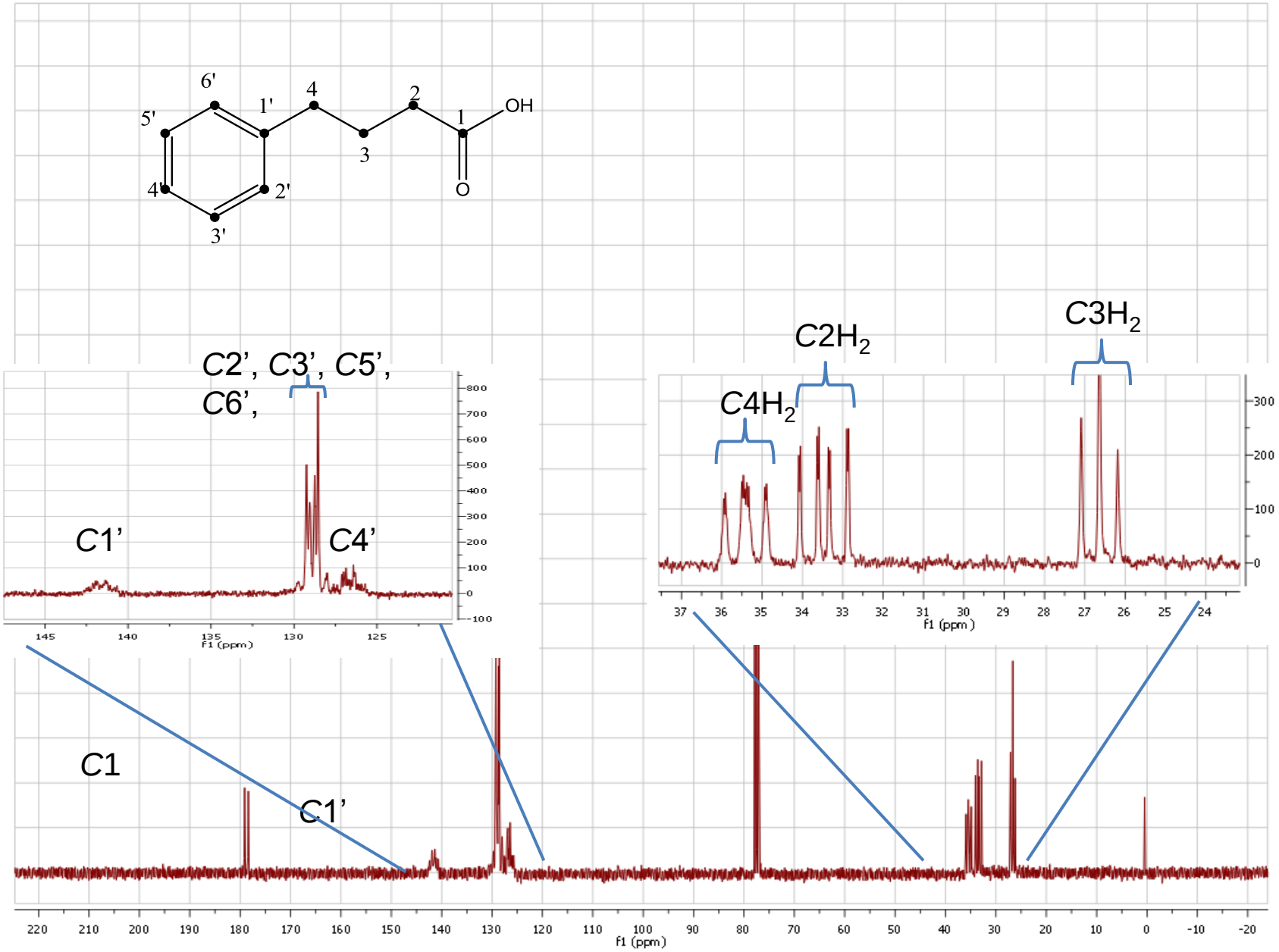


Figure S2. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-4-phenylbutanoic acid. (For ^{13}C NMR assignments of NA-4-phenylbutanoic acid see: Hill, E. A.; Guenther, H. E. *Org. Magn. Reson*. **1981**, *16*, 177.)

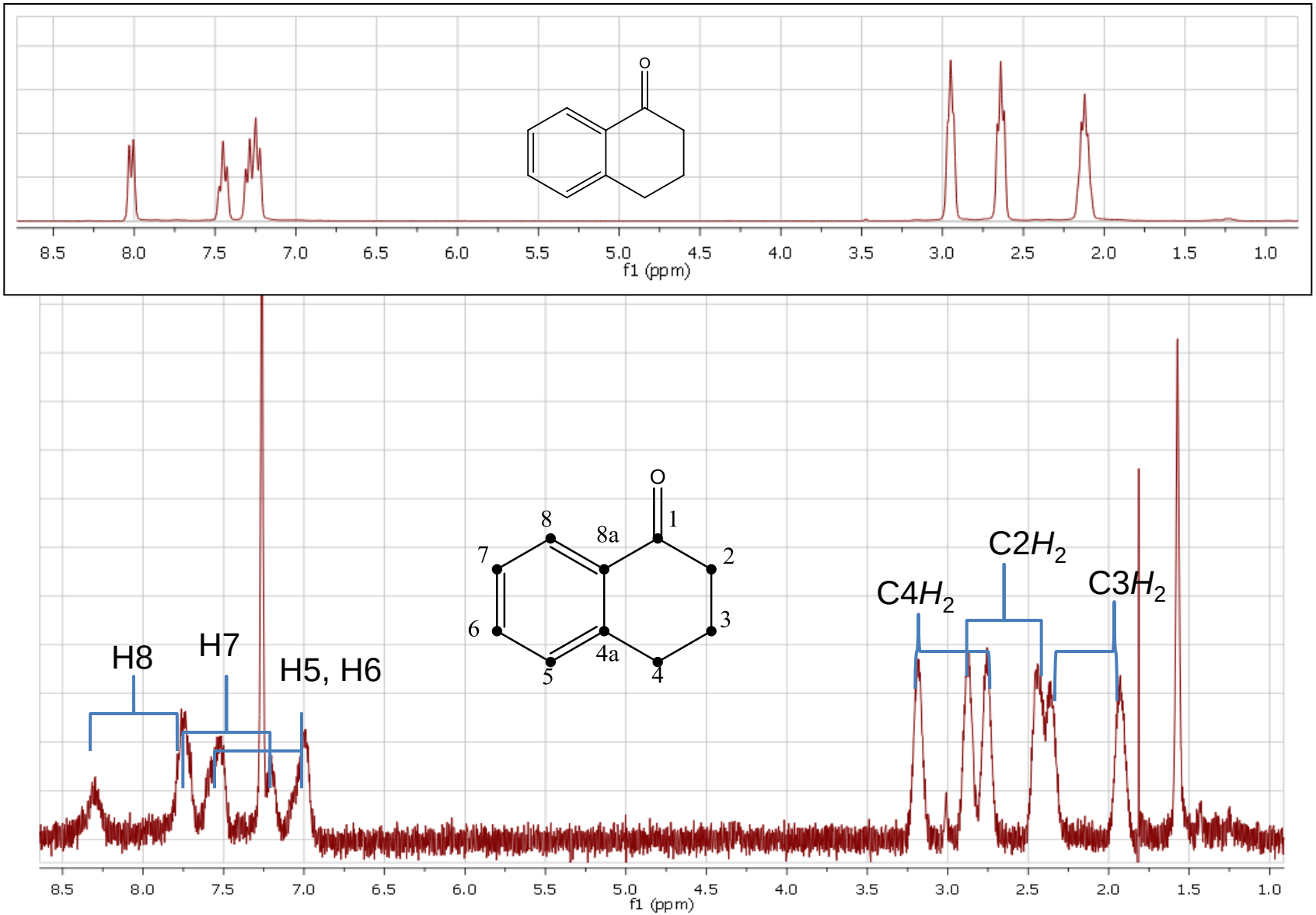


Figure S3. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-tetralone (**1**). (Inset: ^{1}H NMR of NA-tetralone. Chemical shifts identical to report with assignments: Agrawal, P. K.; Thakur, R.S.; Frahm, A. W.; Schneider, M. *Magn. Reson. Chem.* **1990,** *28,* 931.)

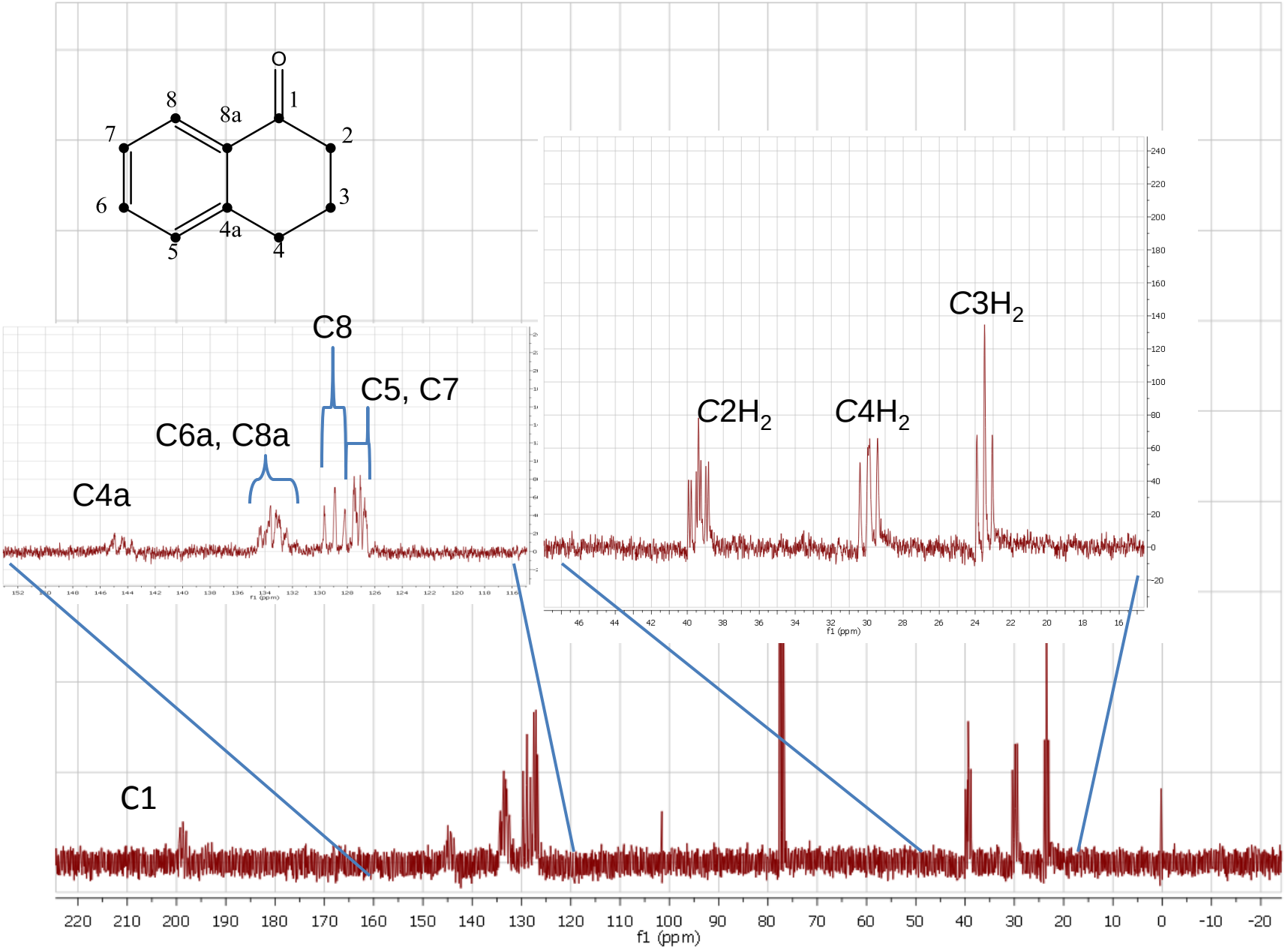


Figure S4. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-tetralone (**1**). (For ^{13}C NMR assignments of NA-tetralone see: Agrawal, P. K.; Thakur, R.S.; Frahm, A. W.; Schneider, M. *Magn. Reson. Chem.* **1990,** *28,* 931.)

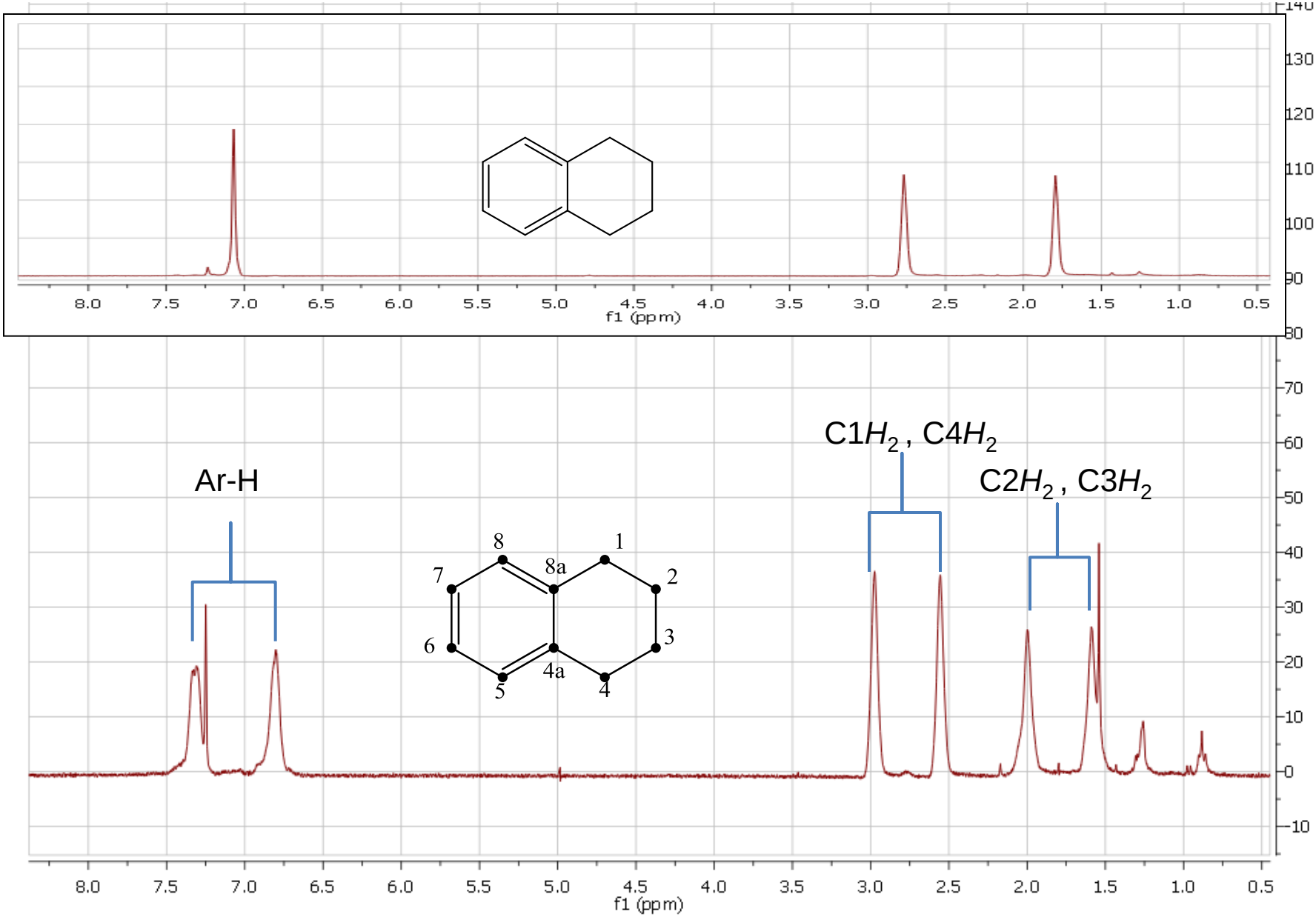


Figure S5. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-tetralin (**2**). (Inset: ^{1}H NMR of NA-tetralin. For ^{1}H NMR of 1-$^{13}C_1$-tetralin see: Pickering, R. E.; Bymaster, D. L.; Eisenbraun, E. J. *J. Labelled Compd. Radiopharm.* **1985**, *22*, 1149.)

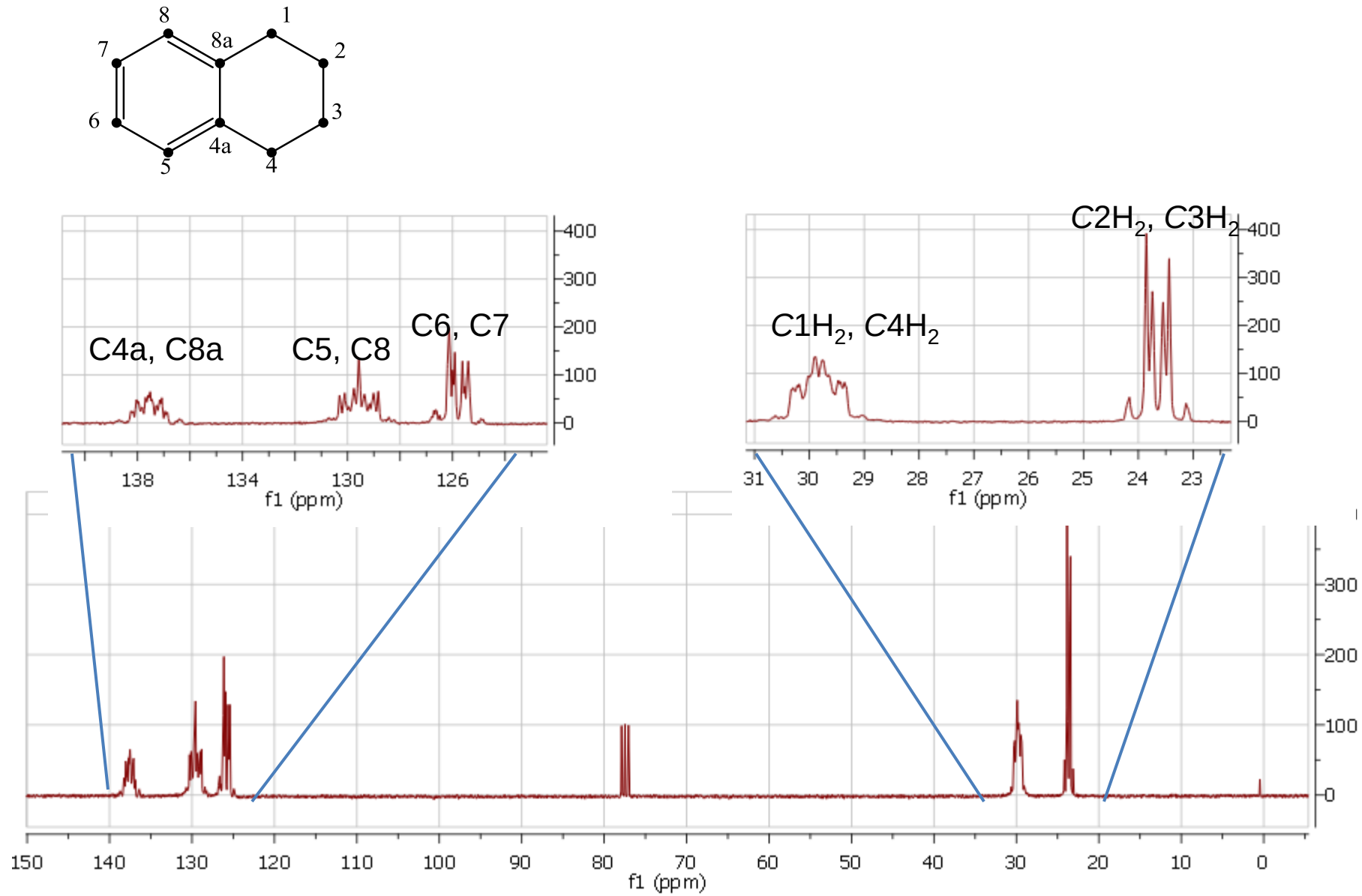


Figure S6. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-tetralin (**2**). (For ^{13}C NMR assignments of NA-tetralin see: Lawens, T.; Schmit-Quih, F.; Nicole, D. *Mag. Res. Chem.* **1995**, *33*, 523.)

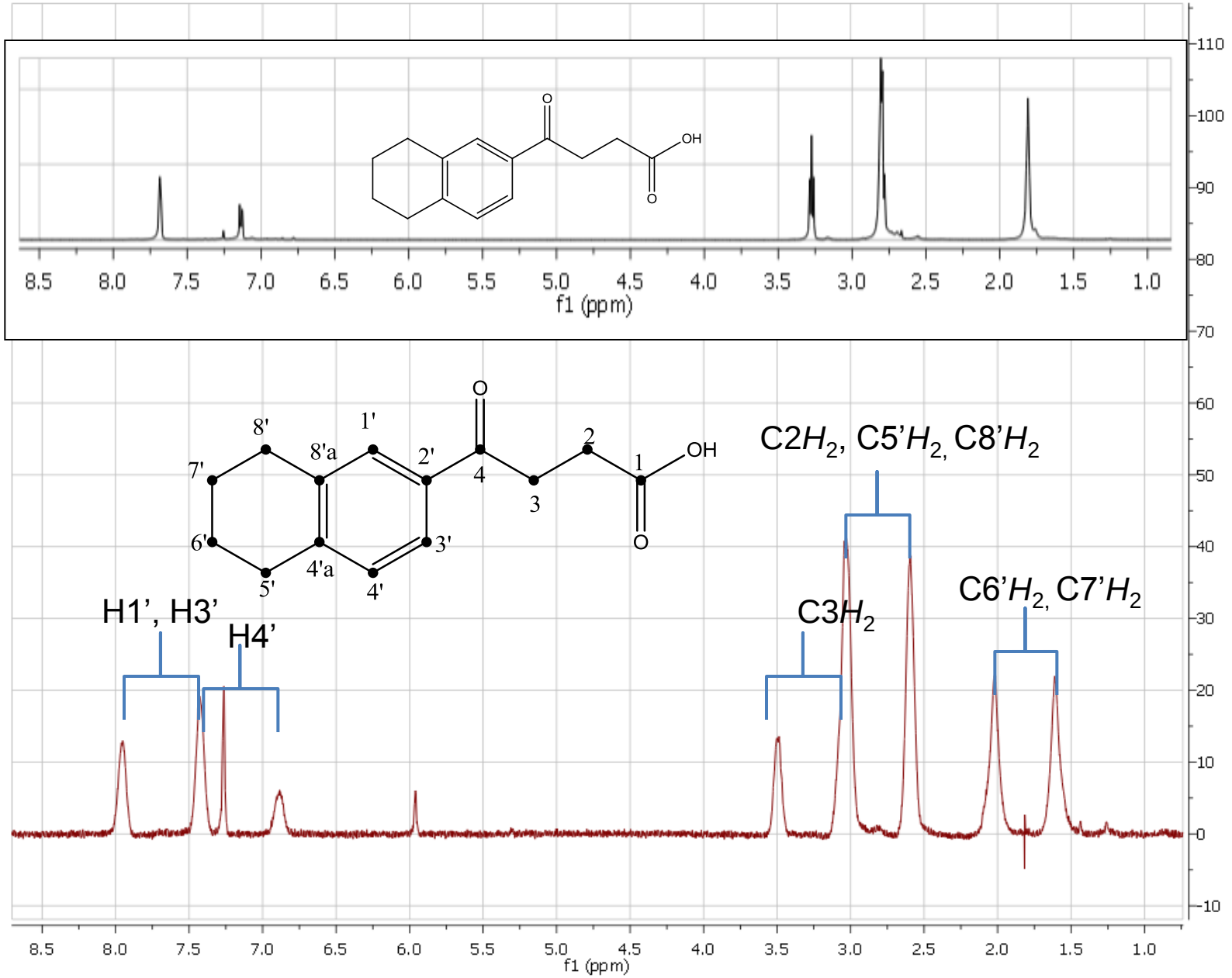


Figure S7. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid. (Inset: ^{1}H NMR of NA-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid)

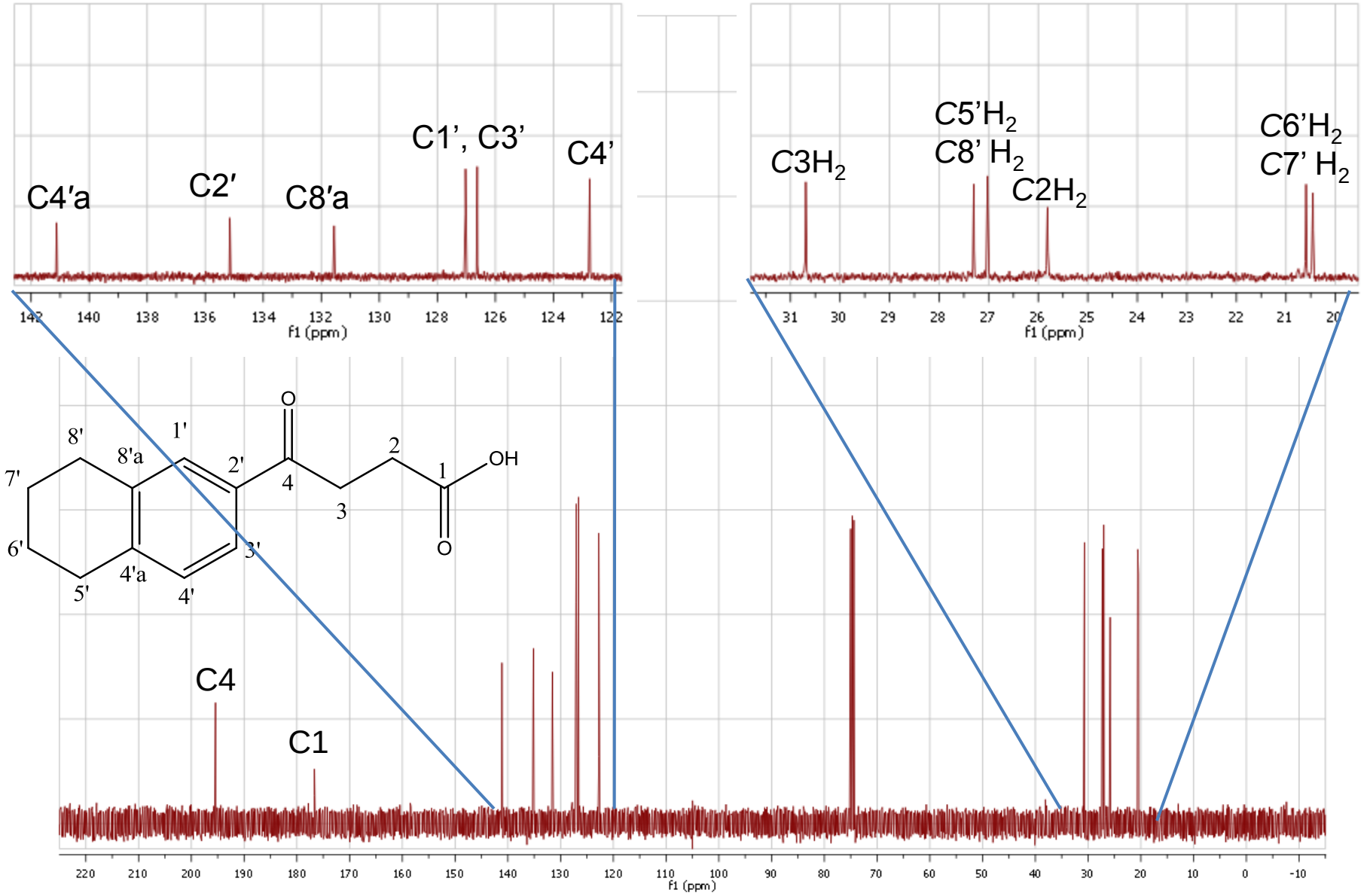


Figure S8. ^{13}C NMR (100 MHz, $CDCl_3$) of NA-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid.

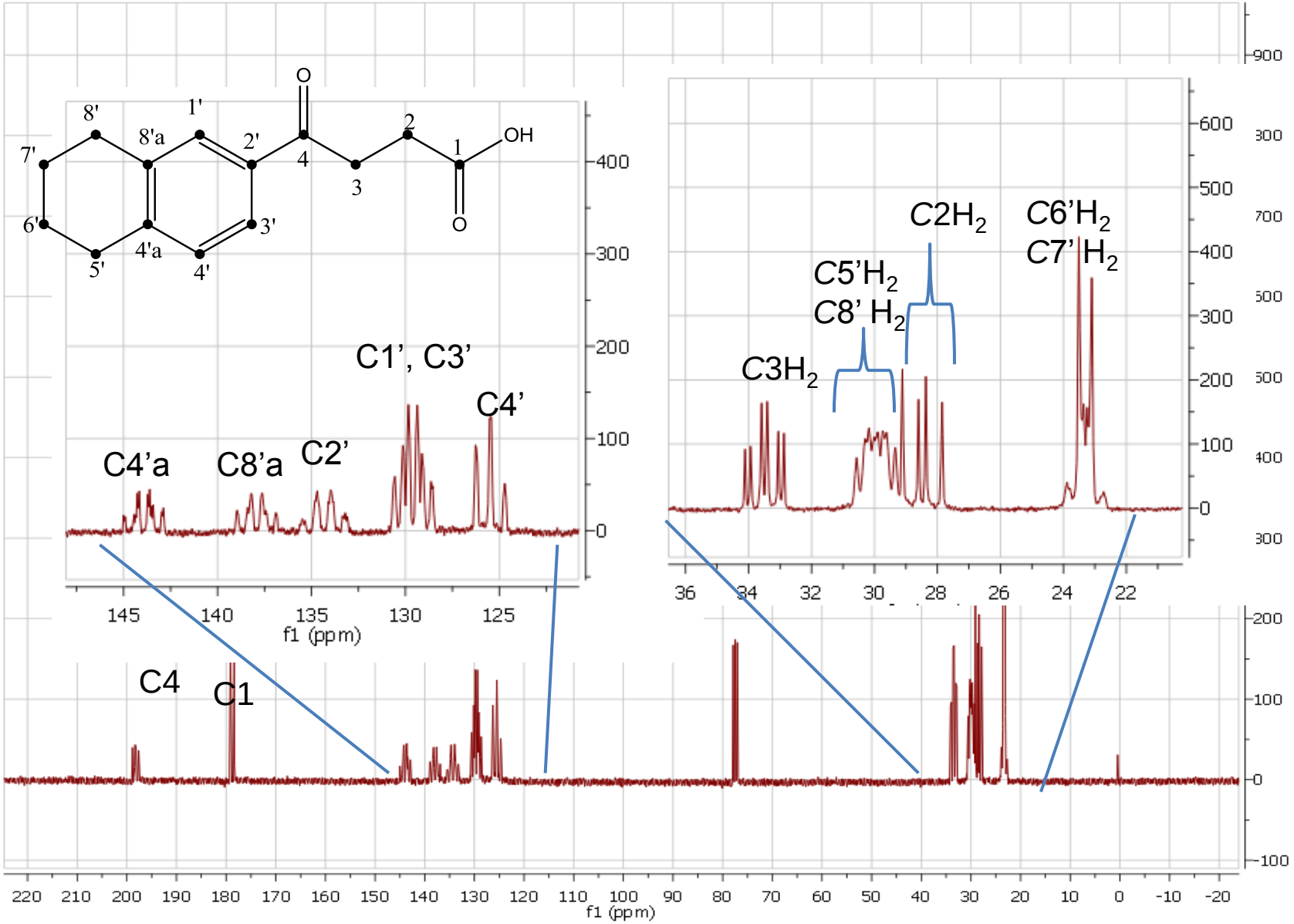


Figure S9. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid.

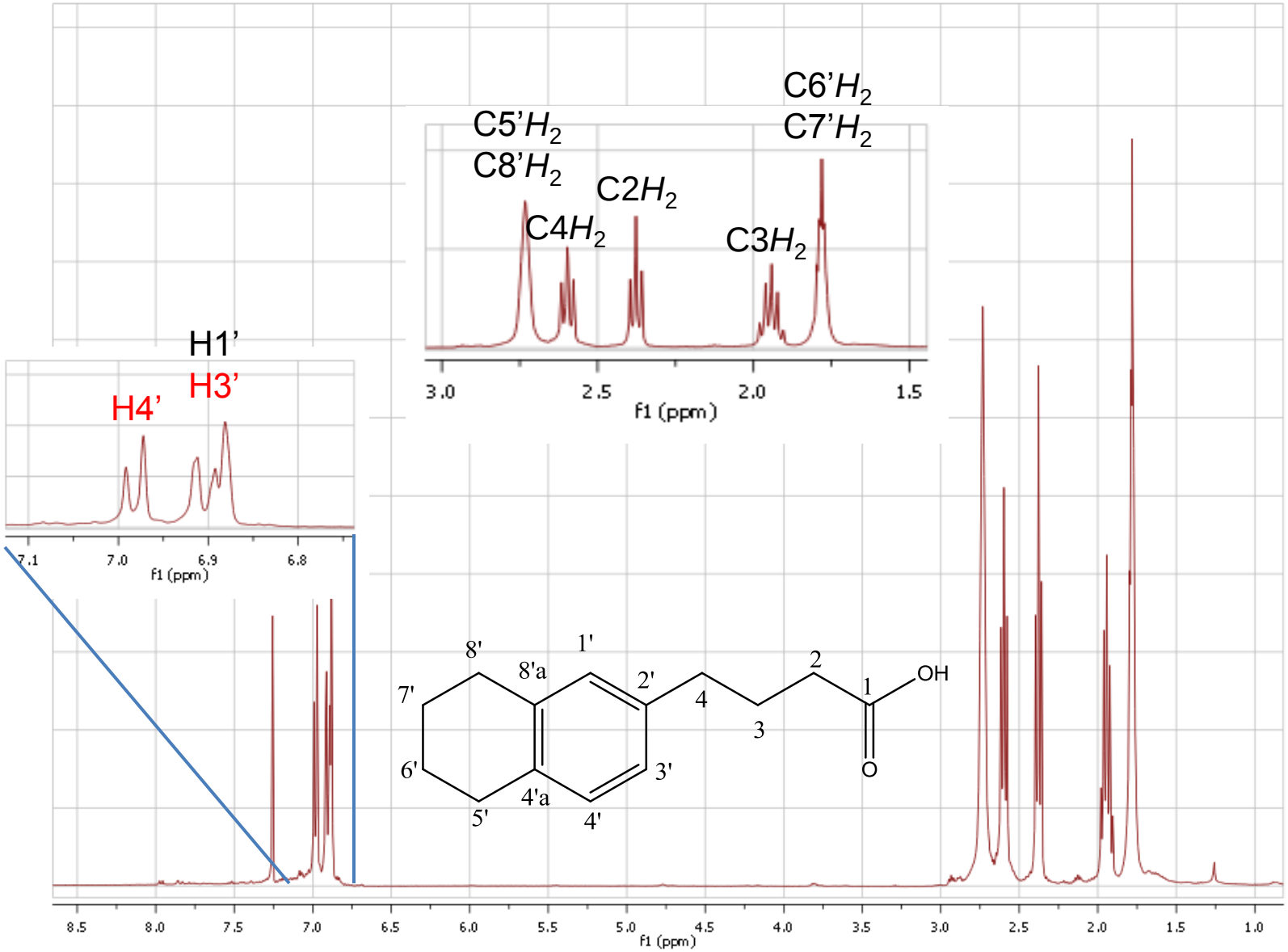


Figure S10. ^{1}H NMR (400 MHz, $CDCl_3$) of NA-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (**3**). (Chemical shifts identical to report: Murphy, P. V.; Hubbard, R. E.; Manallack, D. T.; Wills, R. E.; Montanaby, J. G.; Taylor, R. J. K. *Bioorg. Med. Chem.* **1998,** *6*, 2421.)

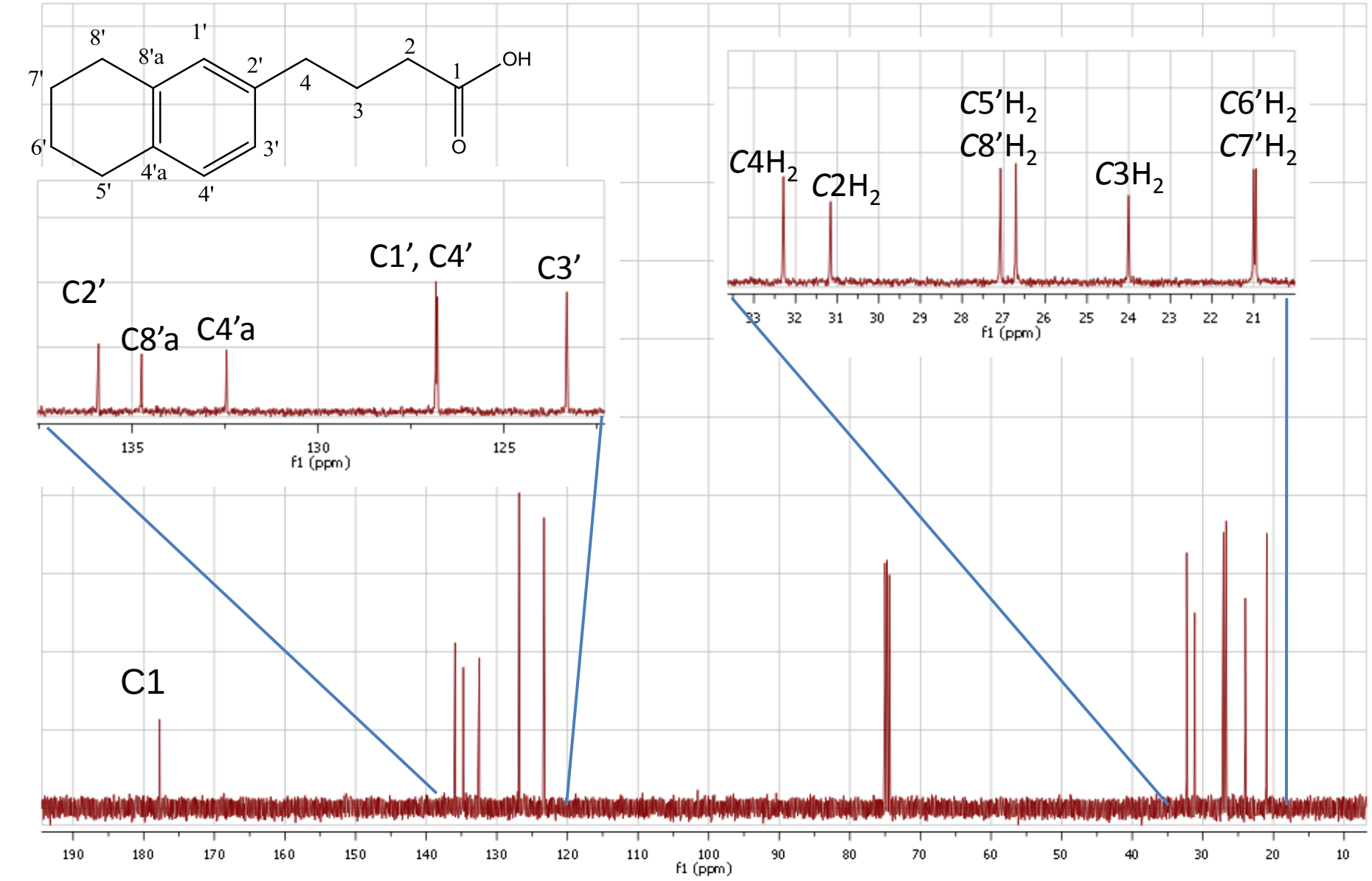


Figure S11. ^{13}C NMR (100 MHz, $CDCl_3$) of NA-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (**3**). (Chemical shifts identical to report: Murphy, P. V.; Hubbard, R. E.; Manallack, D. T.; Wills, R. E.; Montanaby, J. G.; Taylor, R. J. K. *Bioorg. Med. Chem.* **1998,** *6*, 2421. Assignments based on 6-butanyltetralin: Morin, F. G.; Horton, W. J.; Grant, D. M.; Pugmire, R. J.; Dalling, D. K. *J. Org. Chem.* **1985**, *50*, 3380.)

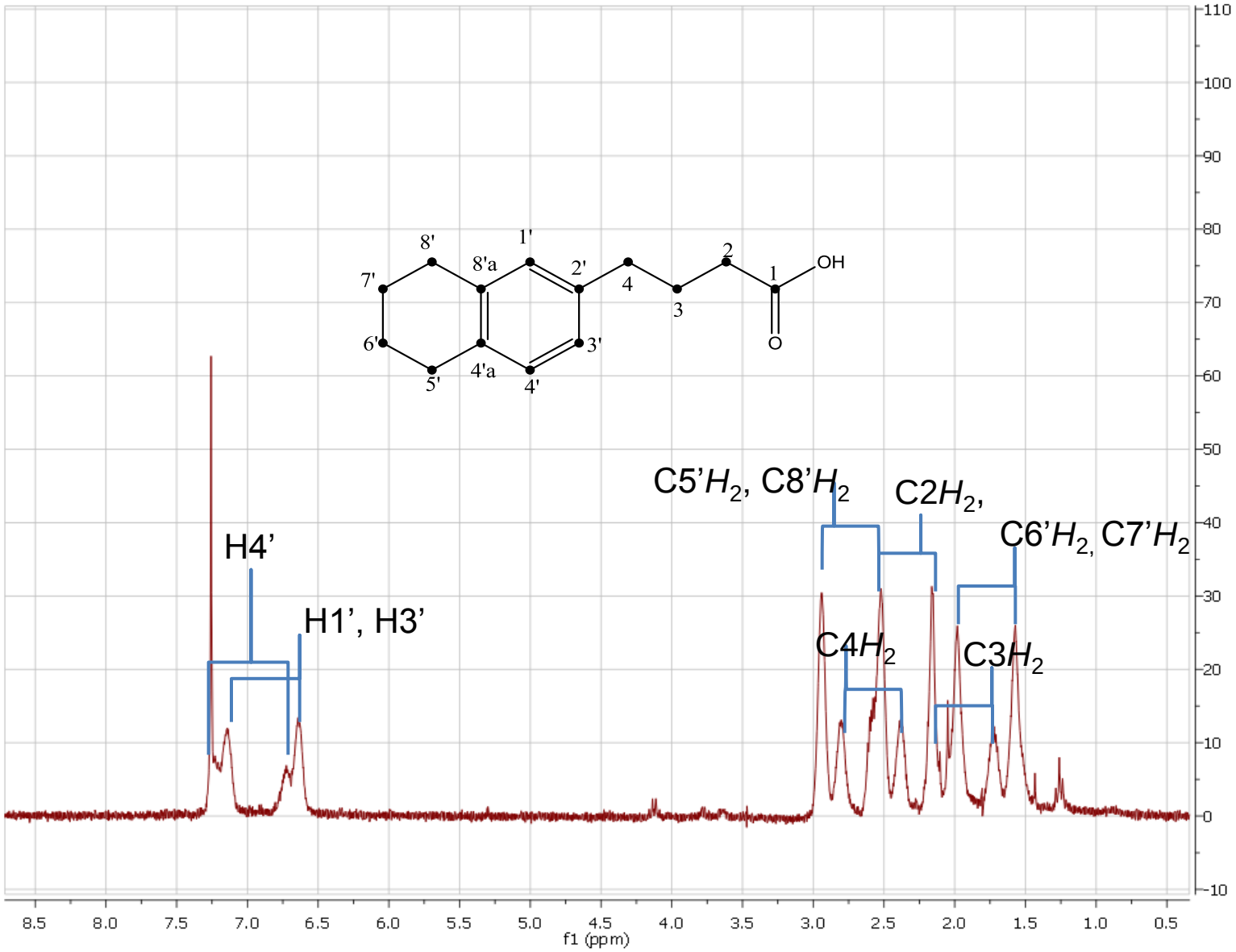


Figure S12. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (**3**).

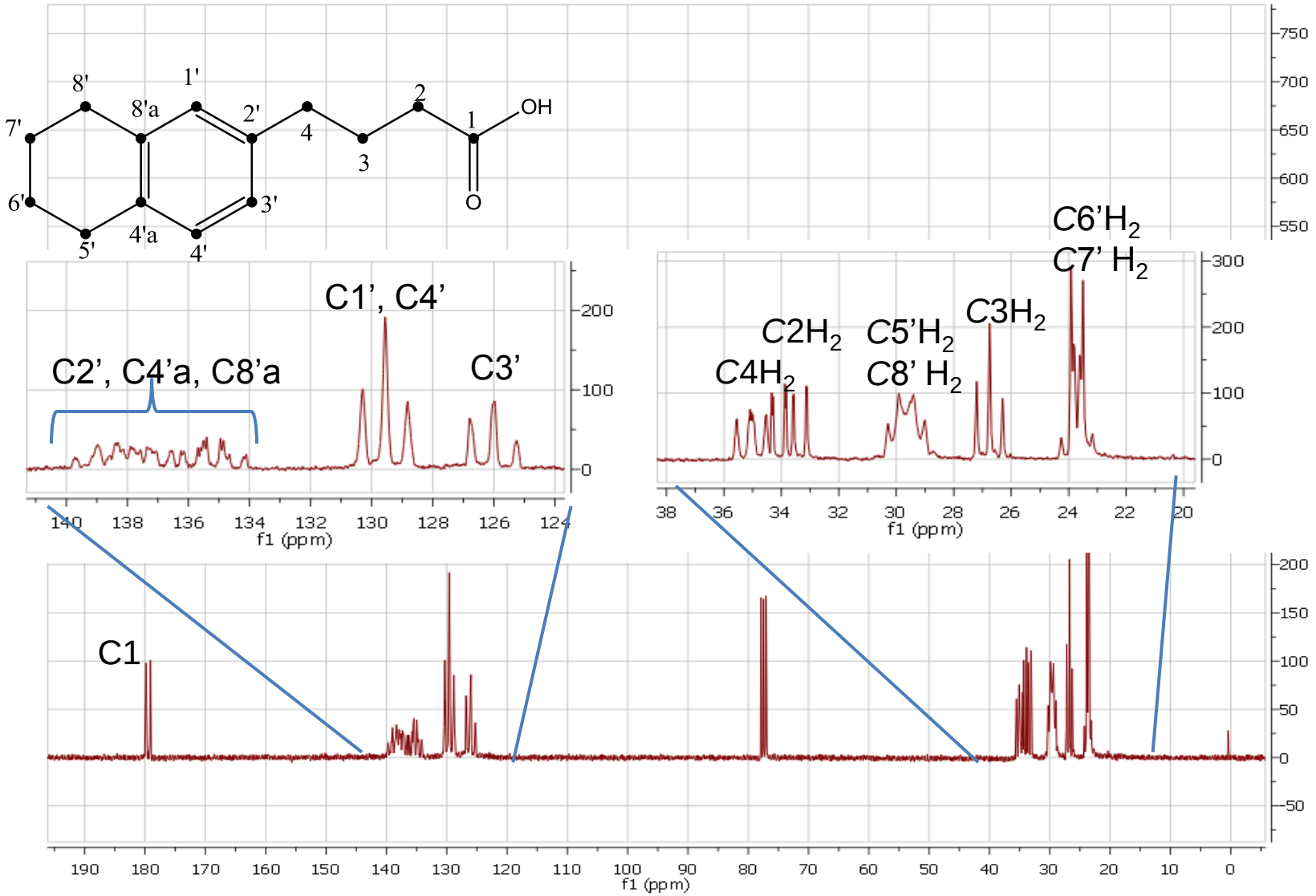


Figure S13. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (**3**).

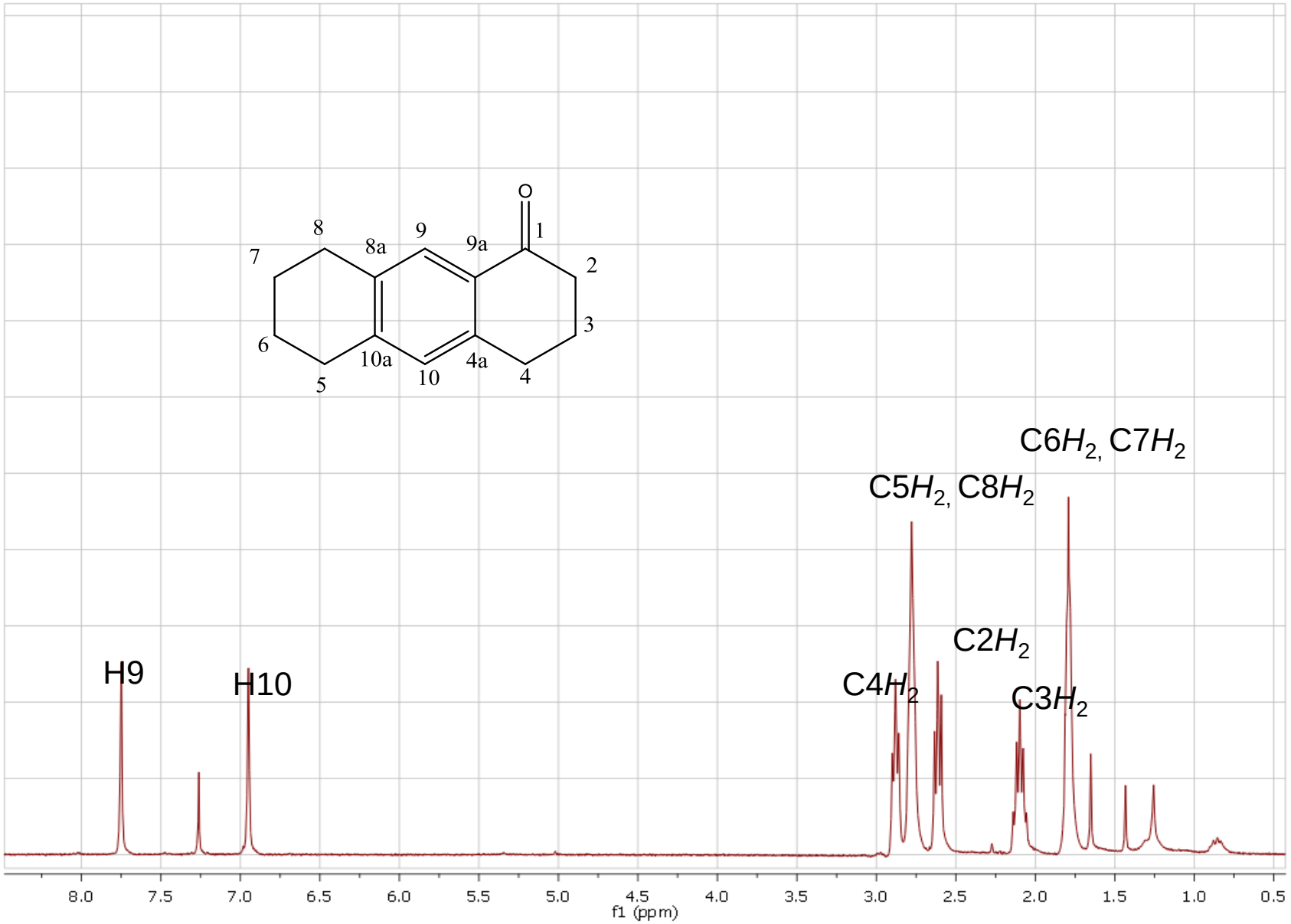


Figure S14. ^{1}H NMR (300 MHz, $CDCl_3$) of NA-3,4,5,6,7,8-hexahydroanthracen-1(2*H*)-one . (Chemical shifts identical to report: Boykin, D. W. *J. Org. Chem.* **1990**, *55*, 425.)

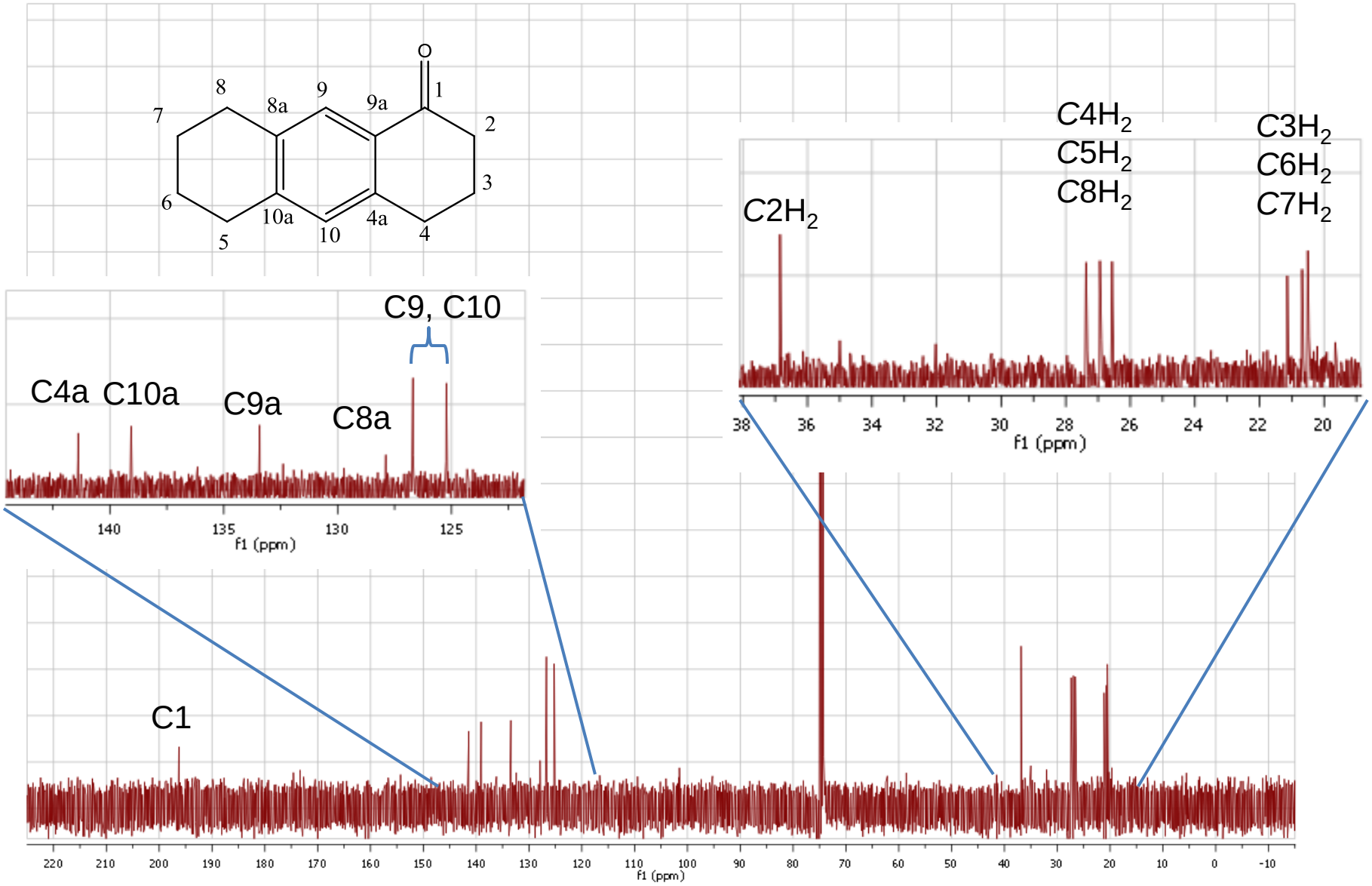


Figure S15. ^{13}C NMR (100 MHz, $CDCl_3$) of NA-3,4,5,6,7,8-hexahydroanthracen-1(2*H*)-one . (Chemical shifts identical to report: Boykin, D. W. *J. Org. Chem.* **1990**, *55*, 425.)

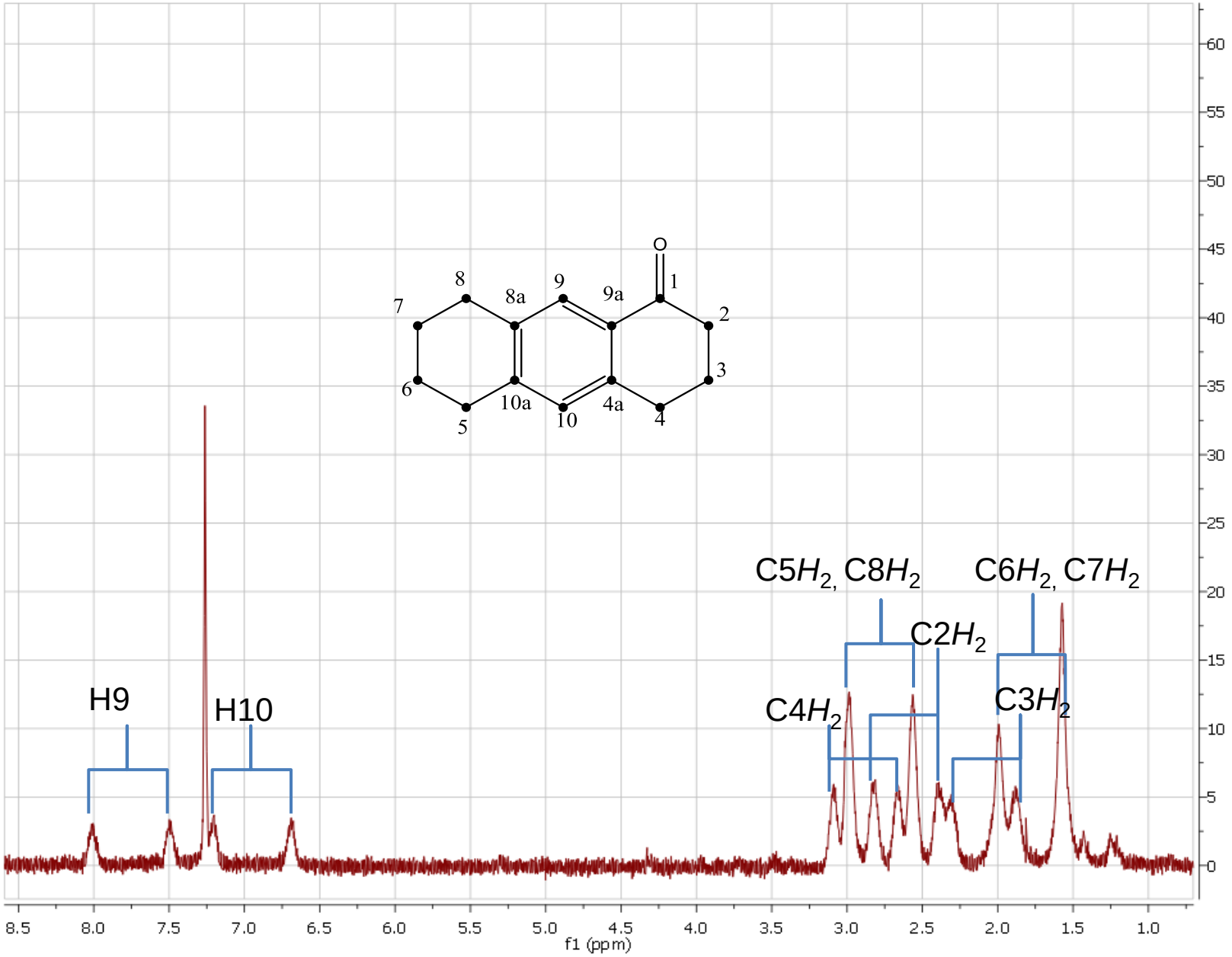


Figure S16. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-3,4,5,6,7,8-hexahydroanthracen-1(2*H*)-one (**4**).

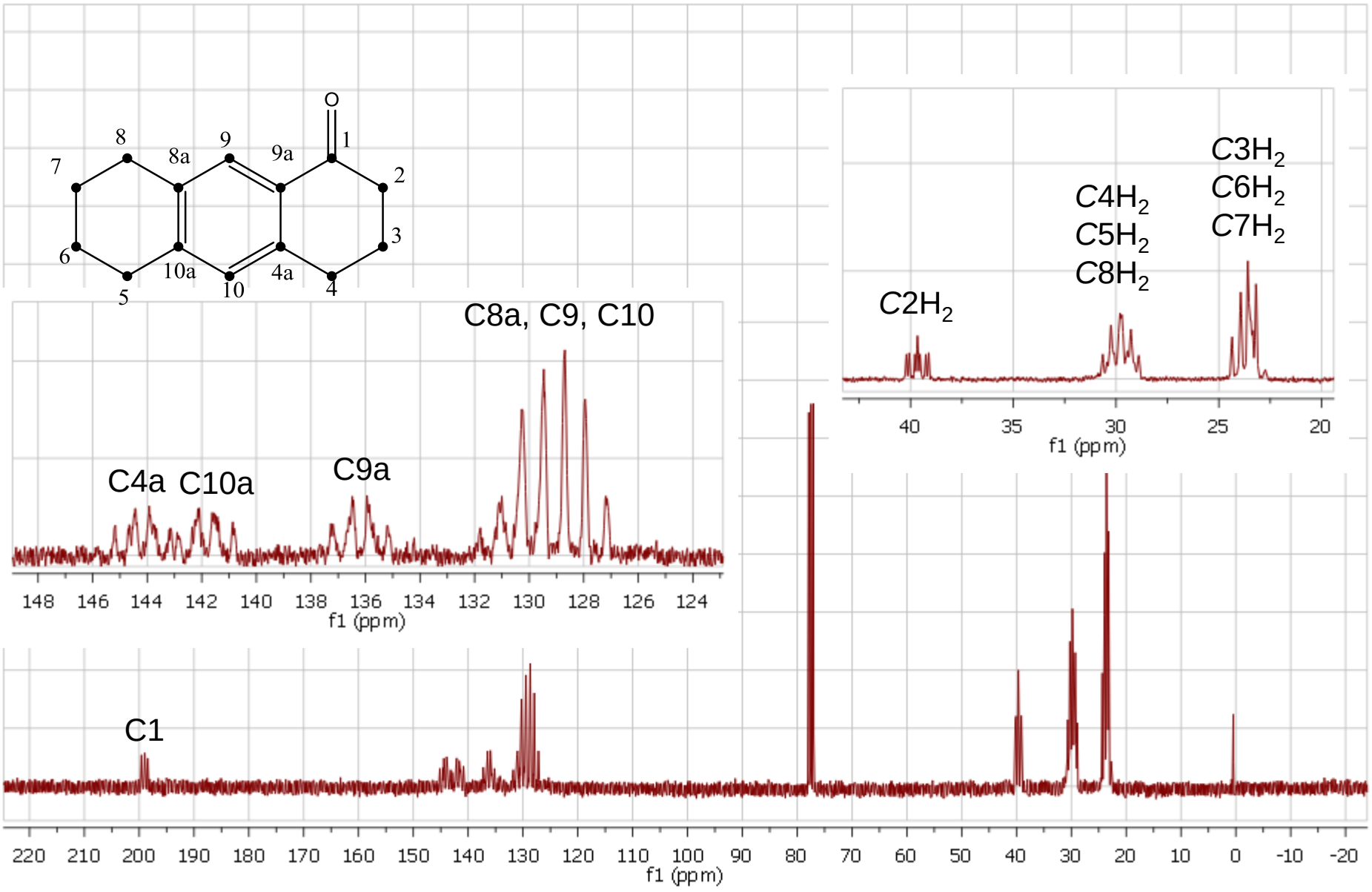


Figure S17. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-3,4,5,6,7,8-hexahydroanthracen-1(2*H*)-one (**4**).

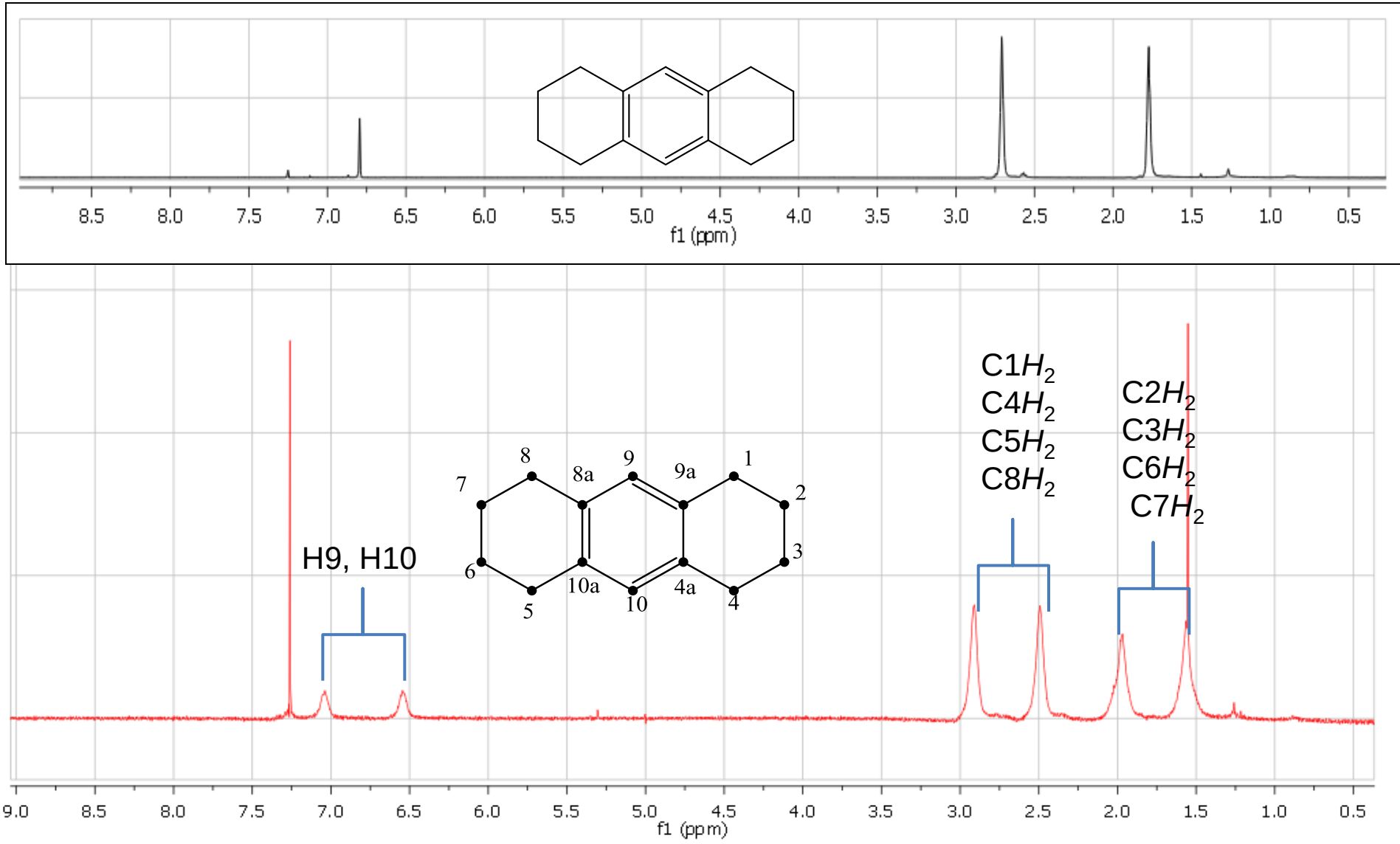


Figure S18. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-1,2,3,4,5,6,7,8-octahydroanthracene: (Inset: ^{1}H NMR of NA-1,2,3,4,5,6,7,8-octahydroanthracene. Chemical shifts identical to report with assignments: Thummel, R. P.; Nutakul, W. *J. Org. Chem.*, **1978**, *43*, 3170.)

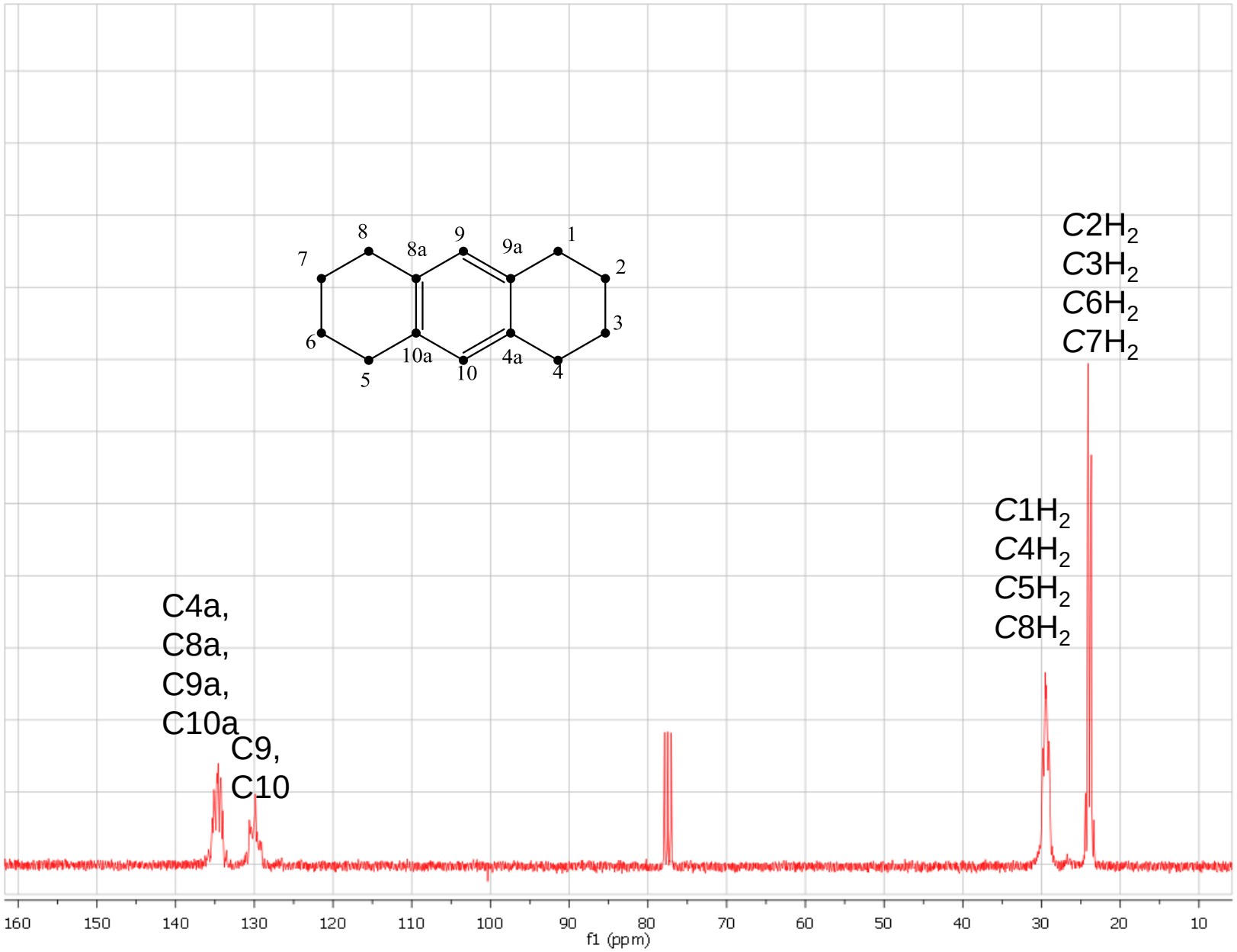


Figure S19. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-1,2,3,4,5,6,7,8-octahydroanthracene. (For ^{13}C NMR assignments of NA-1,2,3,4,5,6,7,8-octahydroanthracene see: Thummel, R. P.; Nutakul, W. *J. Org. Chem.*, **1978**, *43*, 3170.)

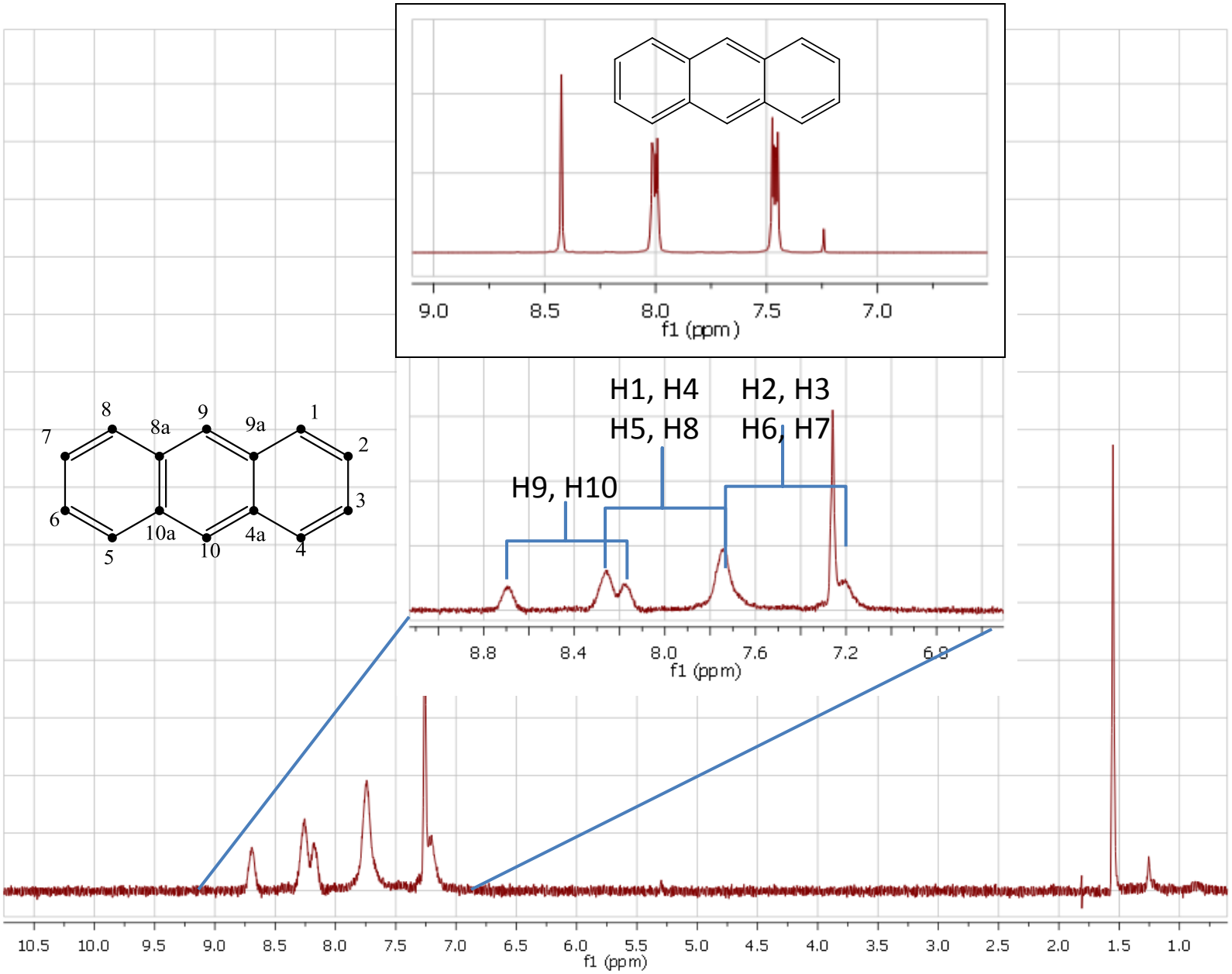
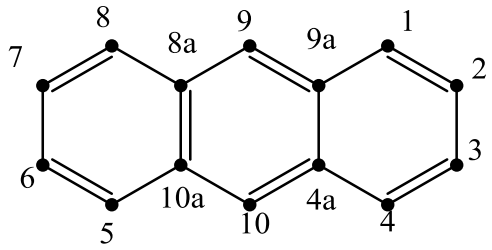


Figure S20. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-anthracene (**5**). (Inset: ^{1}H NMR of NA-anthracene. Chemical shifts identical report with assignments: Gobert, F.; Combrisson, S.; Platzer, N.; Ricard M. *Org. Magn. Reson*. **1976**, *8*, 293.)



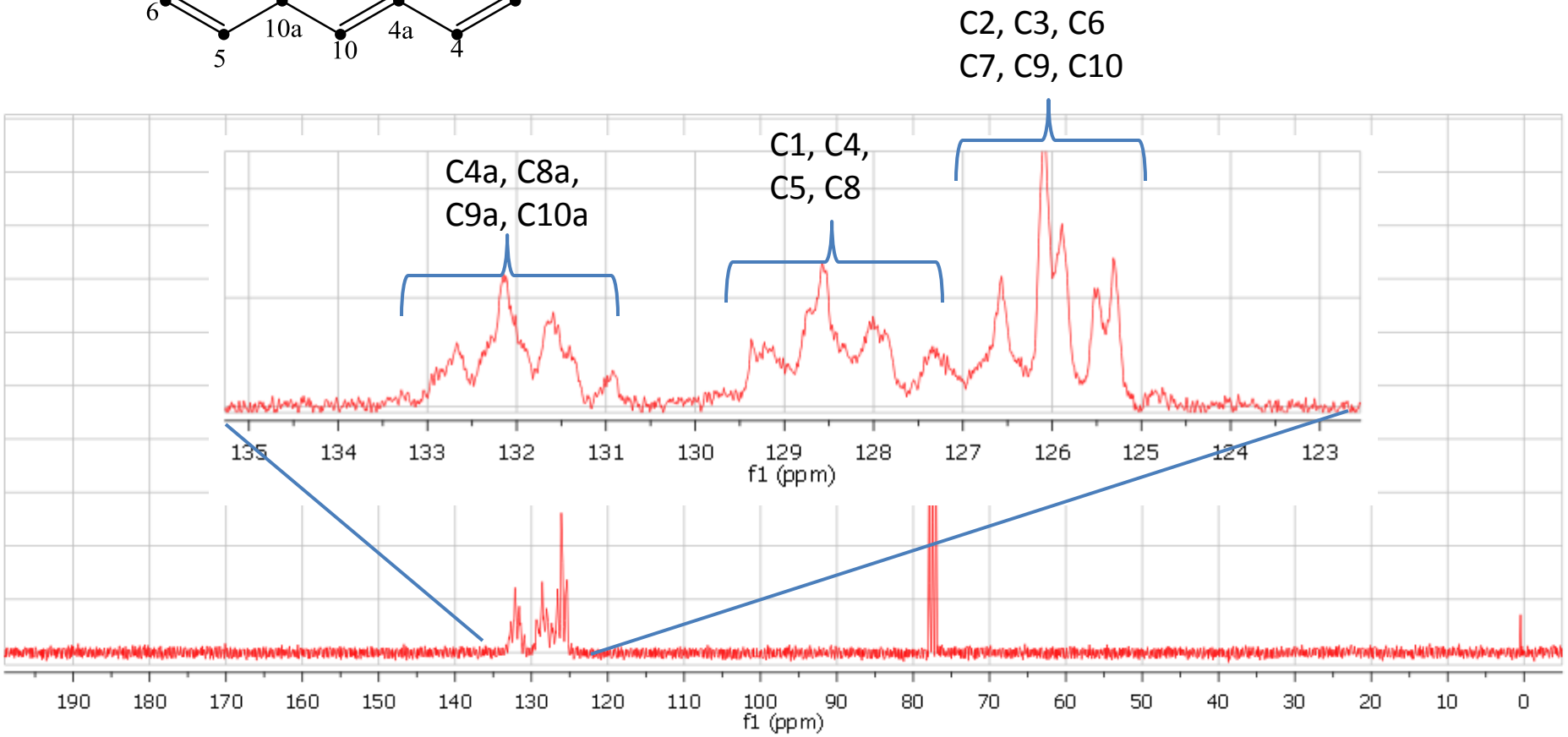


Figure S21. ^{13}C NMR (100 MHz, $CDCl_3$) of U-^{13}C-anthracene (**5**). (For ^{13}C NMR assignments of NA-anthracene see: Gobert, F.; Combrisson, S.; Platzer, N.; Ricard M. *Org. Magn. Reson*. **1976**, *8*, 293.)

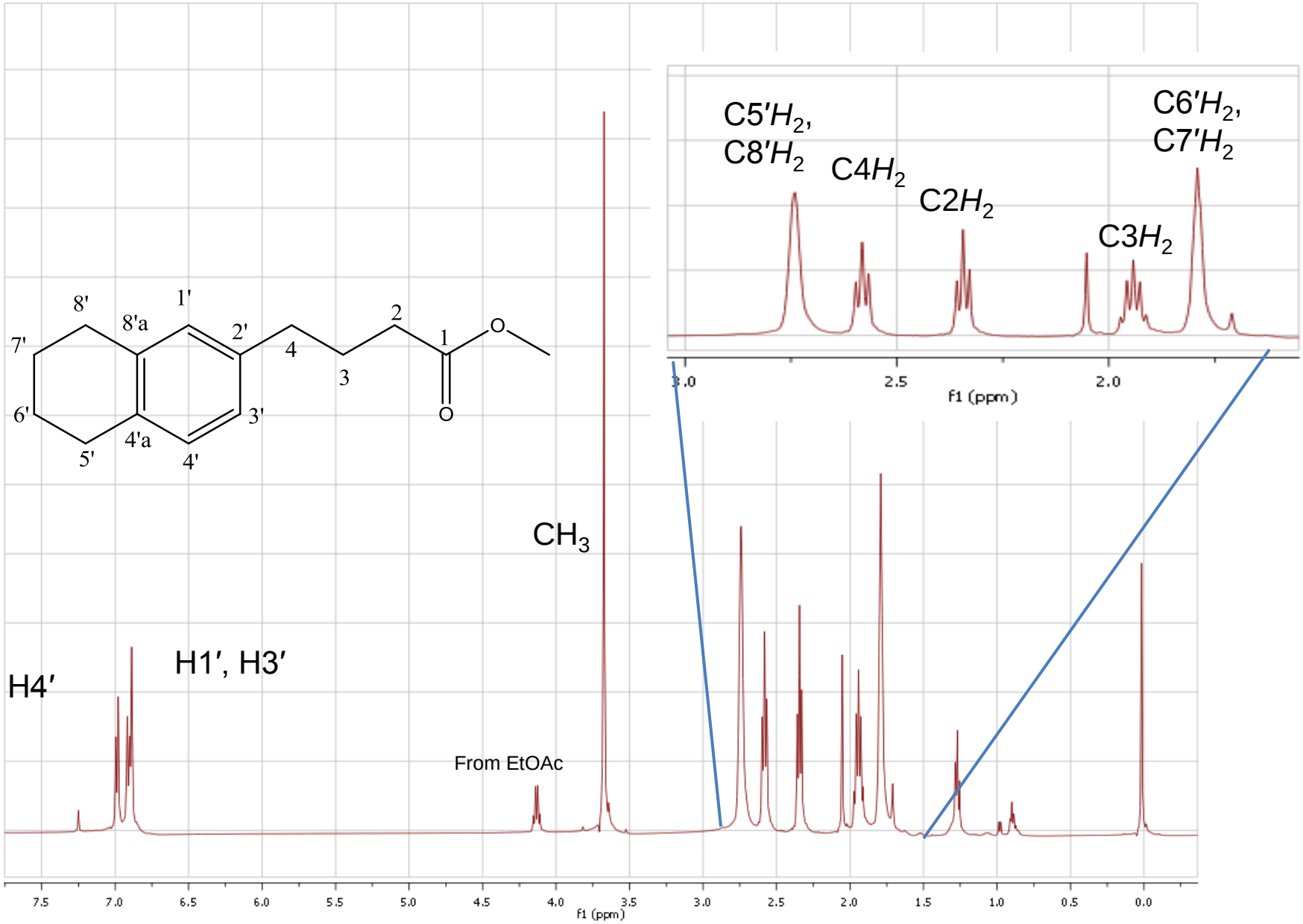


Figure S22. ^{1}H NMR (300 MHz, $CDCl_3$) of NA methyl 4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate.

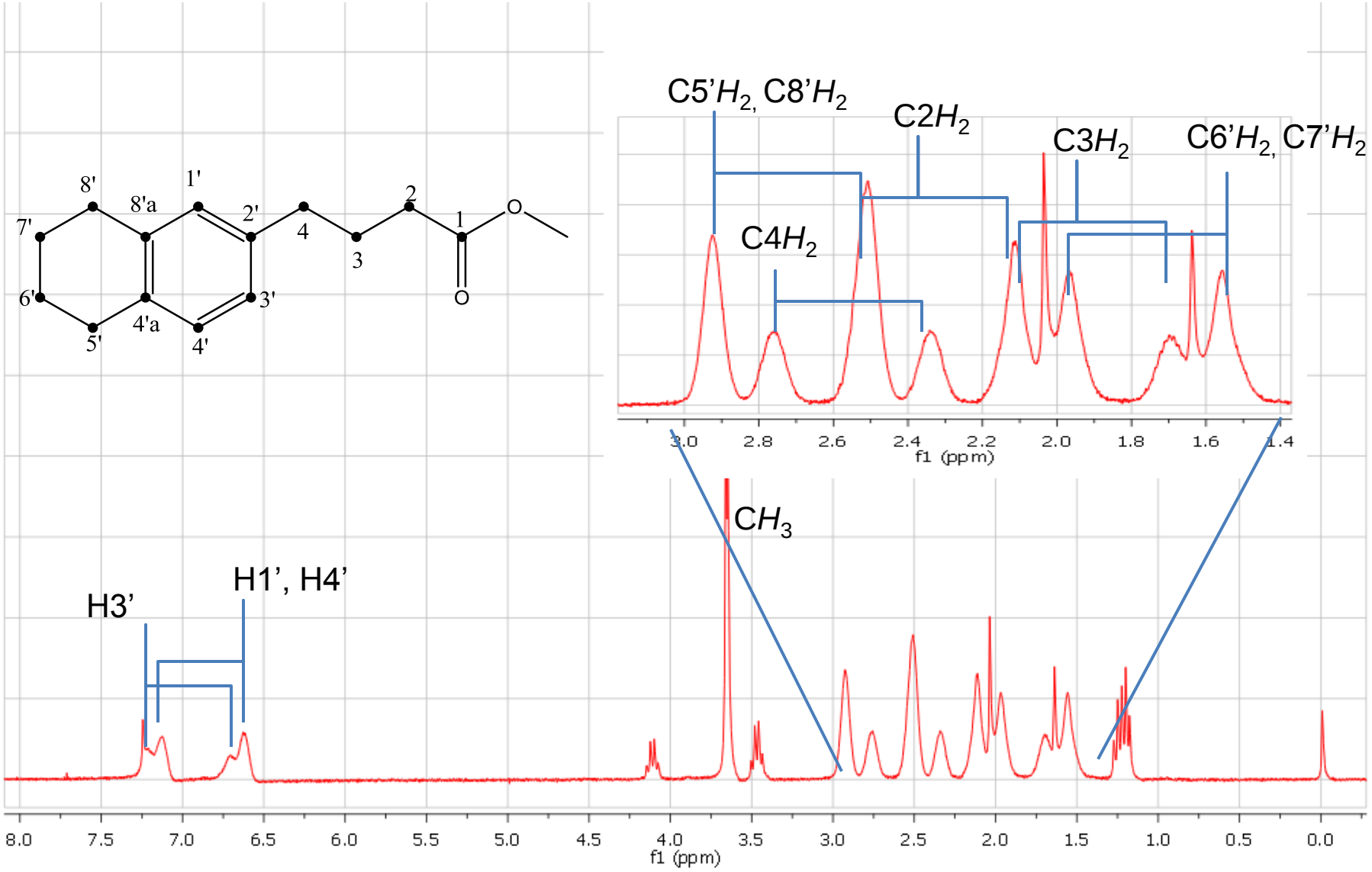


Figure S23. ^{1}H NMR (300 MHz, $CDCl_3$) of methyl U-^{13}C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate.

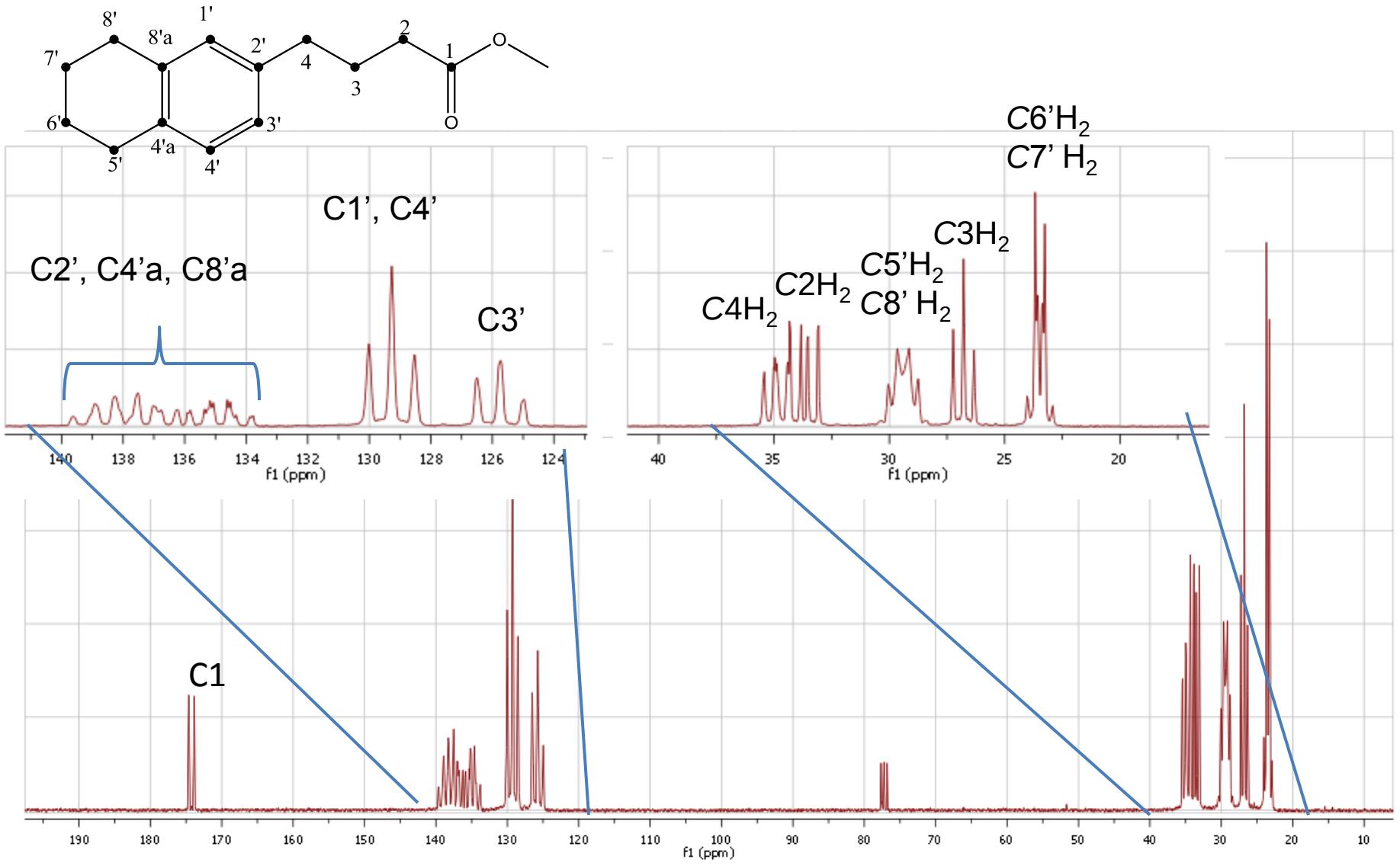


Figure S24. ^{13}C NMR (75 MHz, $CDCl_3$) of methyl U-^{13}C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate. (For the ^{13}C NMR of the corresponding butanoic acid see: Murphy, P. V.; Hubbard, R. E.; Manallack, D. T.; Wills, R. E.; Montanaby, J. G.; Taylor, R. J. K. *Bioorg. Med. Chem.* **1998,** *6*, 2421.)

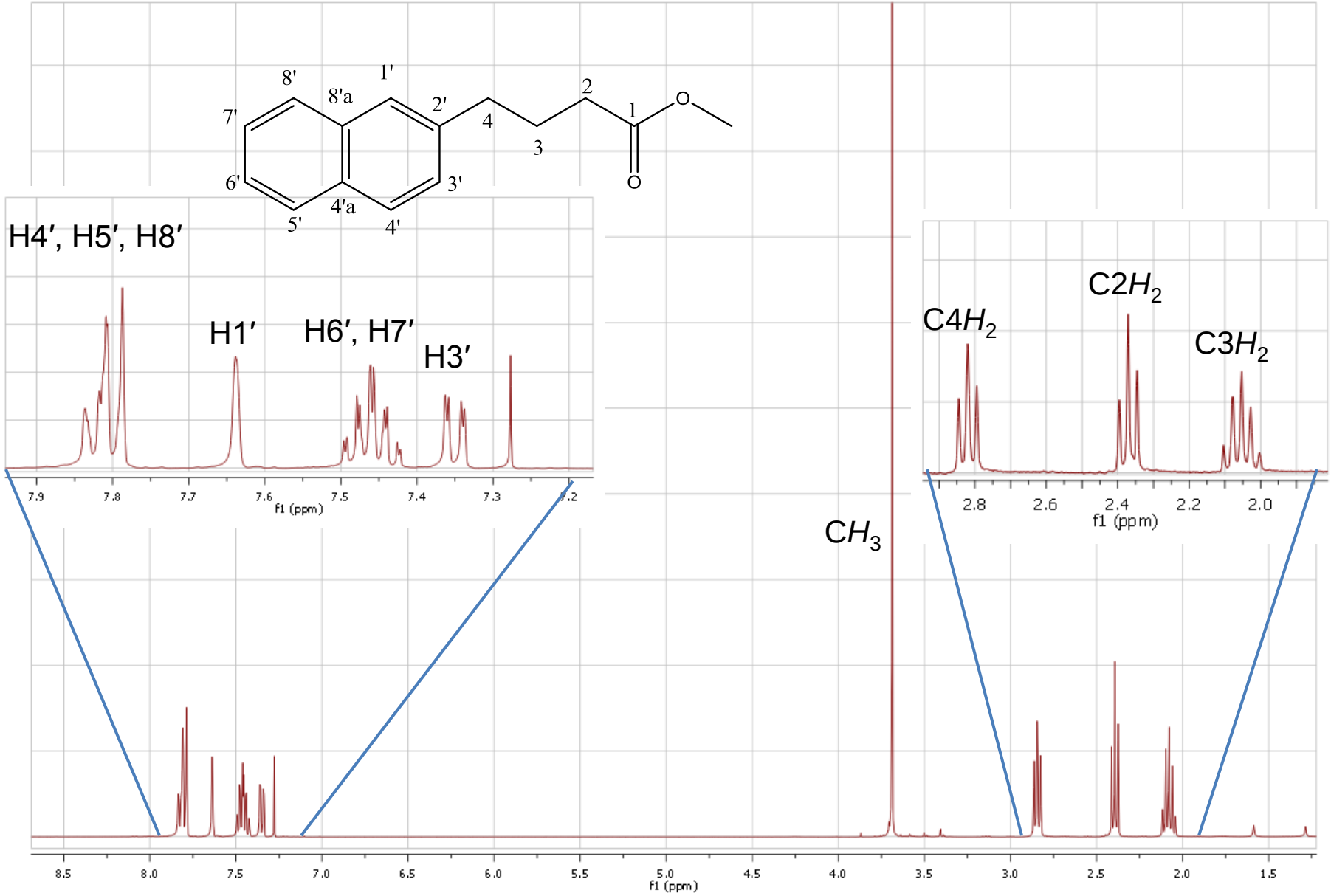


Figure S25. ^{1}H NMR (400 MHz, $CDCl_3$) of methyl NA-4-(naphthalen-2-yl)butanoate. (Assignments based on 2-methyl naphthalene: Salman, S. R. *Org. Magn. Reson*. **1982**, *19*, 181.)

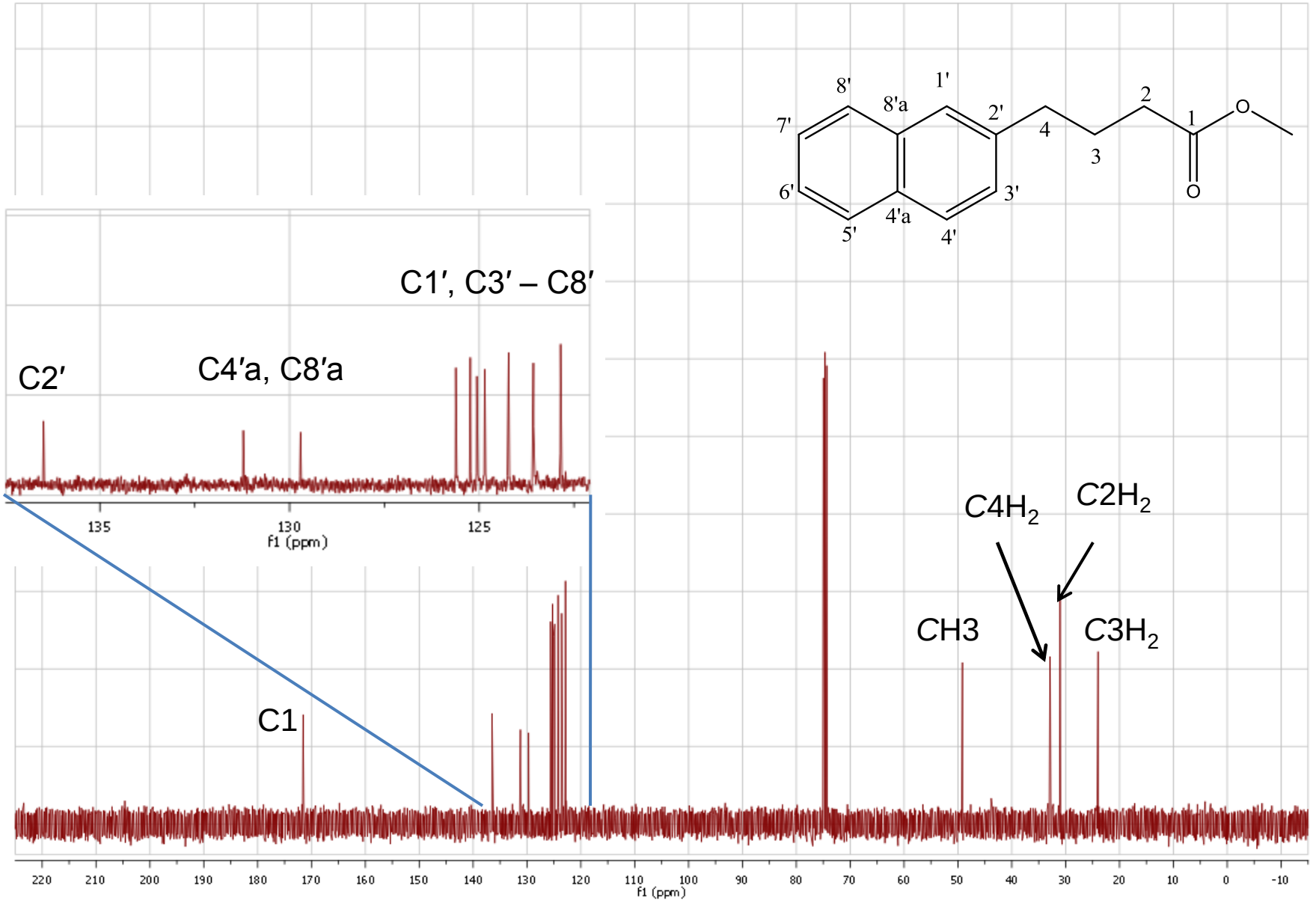


Figure S26. ^{13}C NMR (75 MHz, $CDCl_3$) of methyl NA-4-(naphthalen-2-yl)butanoate (**9**). (Assignments based on 2-methyl naphthalene: Salman, S. R. *Org. Magn. Reson*. **1982**, *19*, 181.)

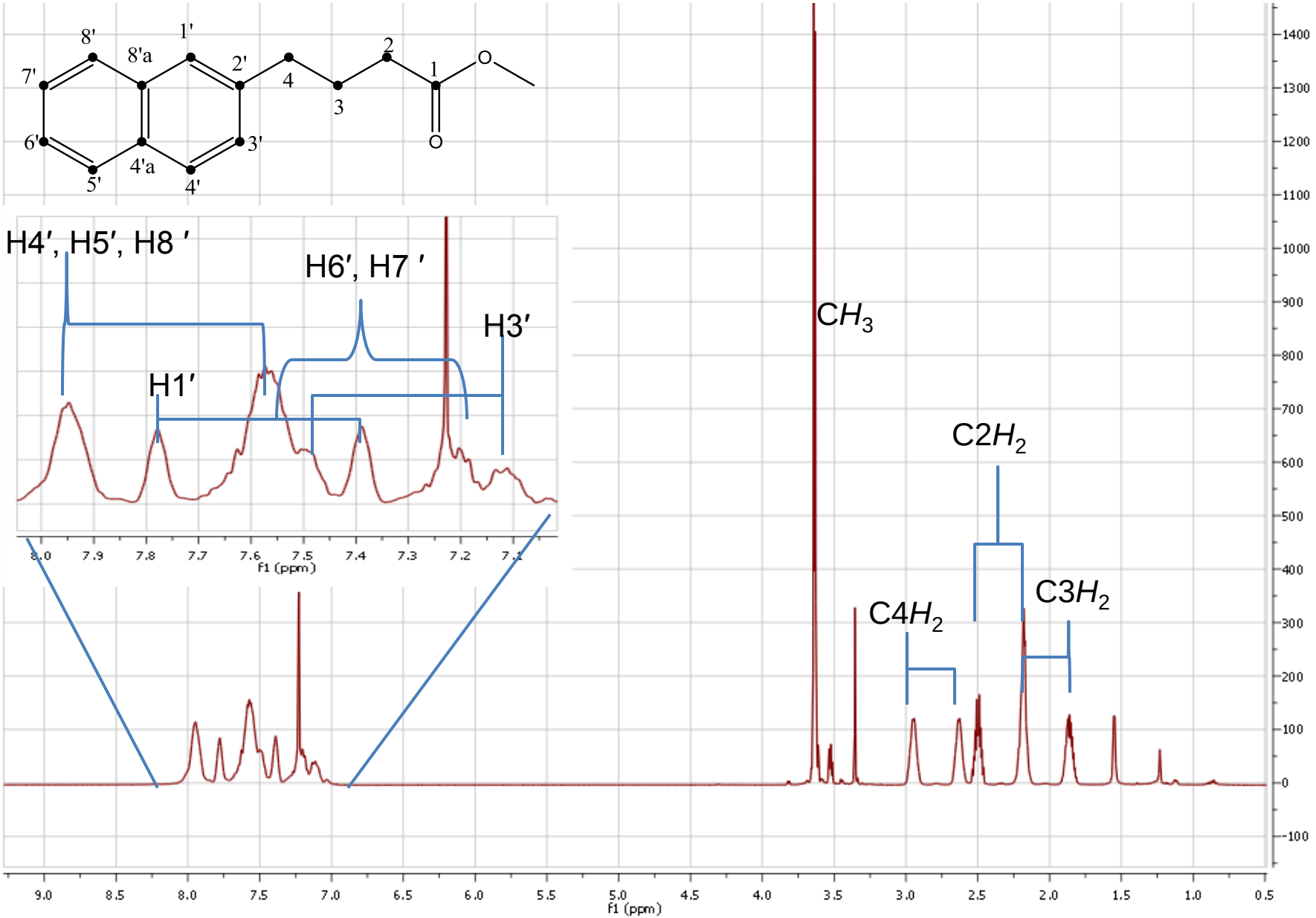


Figure S27. ^{1}H NMR (300 MHz, $CDCl_3$) of methyl U-^{13}C-4-(naphthalen-2-yl)butanoate (**9**). (Assignments based on 2-methyl naphthalene: Salman, S. R. *Org. Magn. Reson*. **1982**, *19*, 181.)

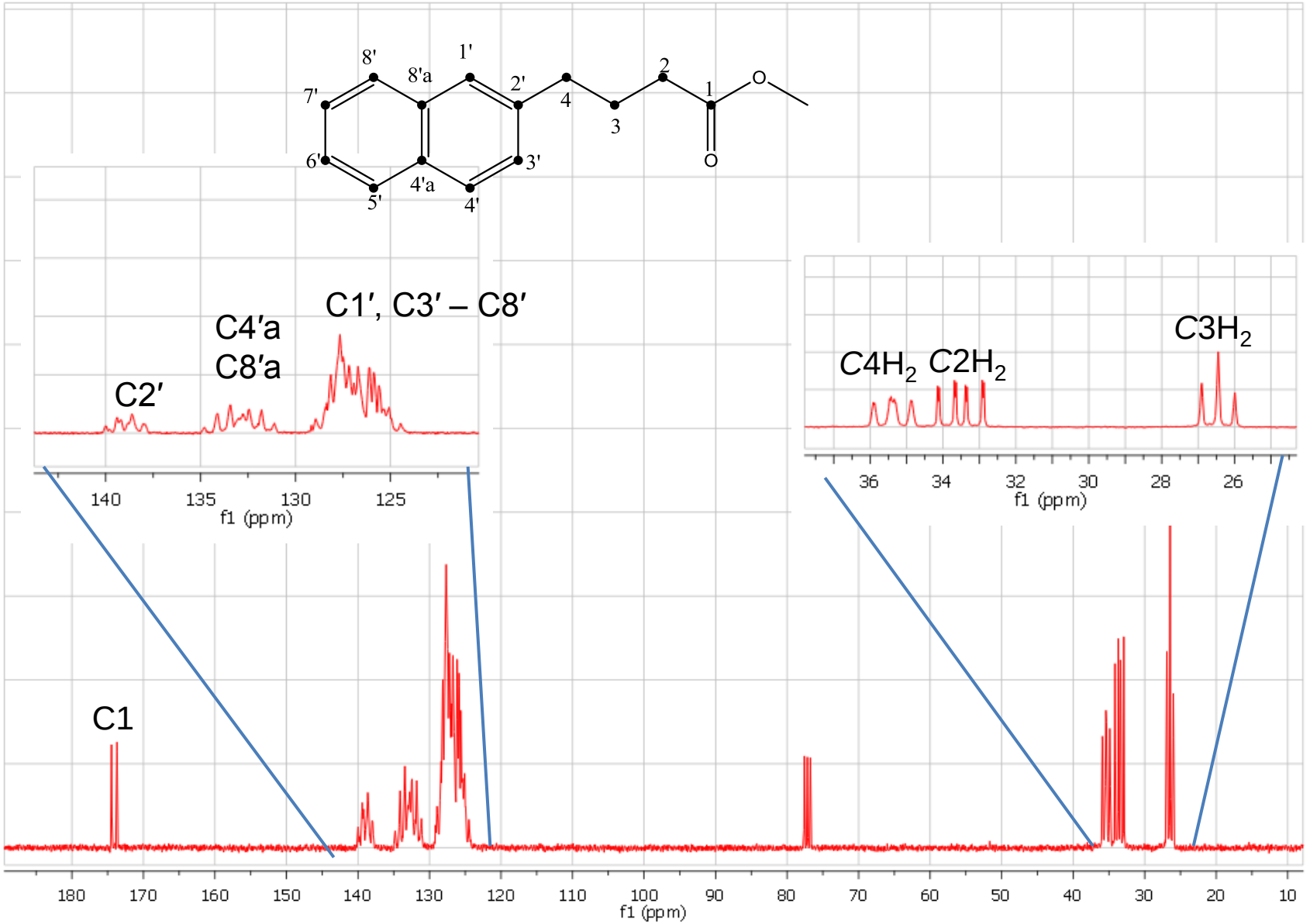


Figure S28. ^{13}C NMR (75 MHz, $CDCl_3$) of methyl U-^{13}C-4-(naphthalen-2-yl)butanoate (**9**). (Assignments based on 2-methyl naphthalene: Salman, S. R. *Org. Magn. Reson*. **1982**, *19*, 181.)

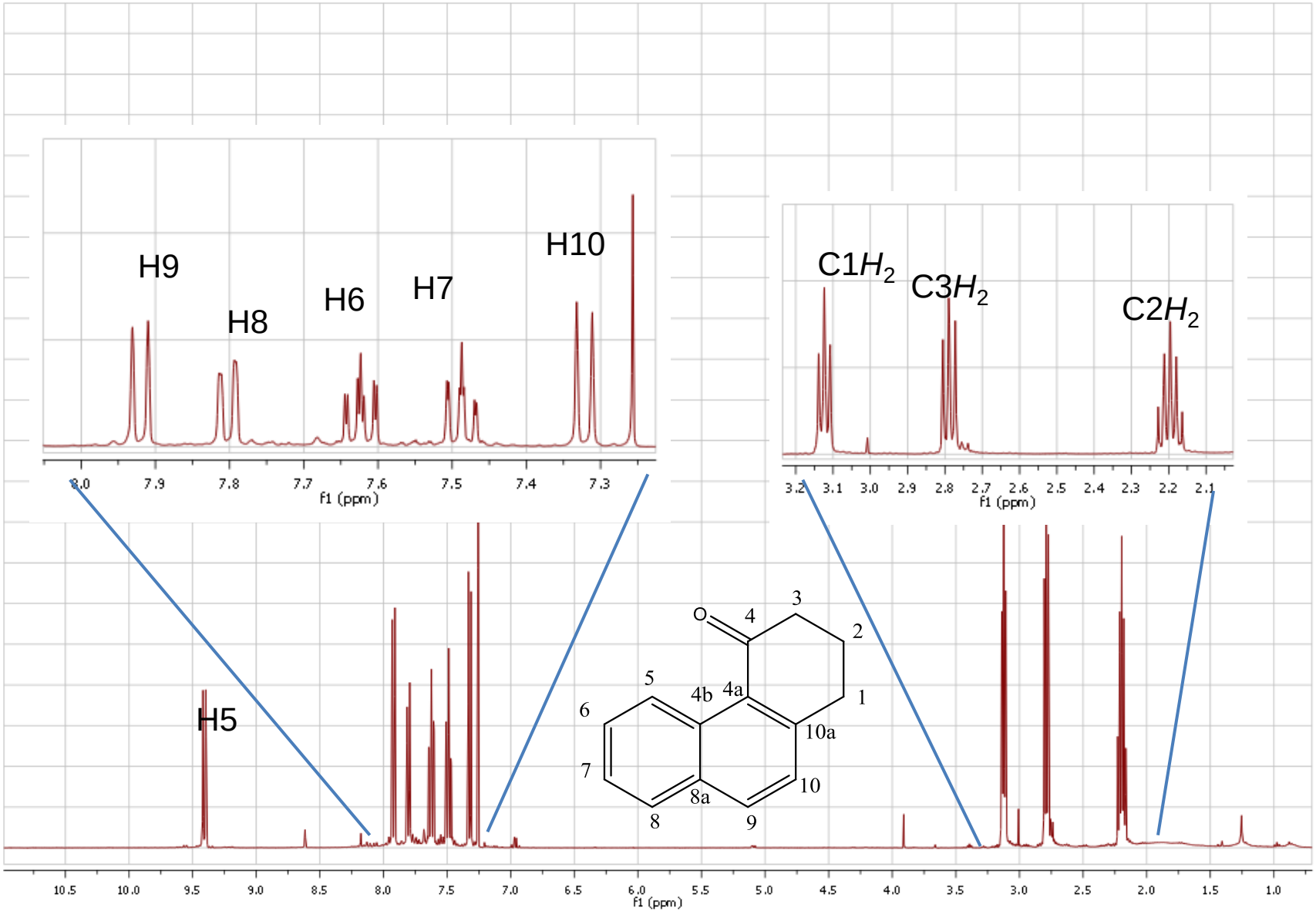


Figure S29. ^{1}H NMR (400 MHz, $CDCl_3$) of NA-2,3-dihydrophenanthren-4(1*H*)-one. (Chemical shifts identical to report with assignments: Ernst, L.; Stolle, R. *Magn. Reson. Chem*. **1989**, *27*, 796.)

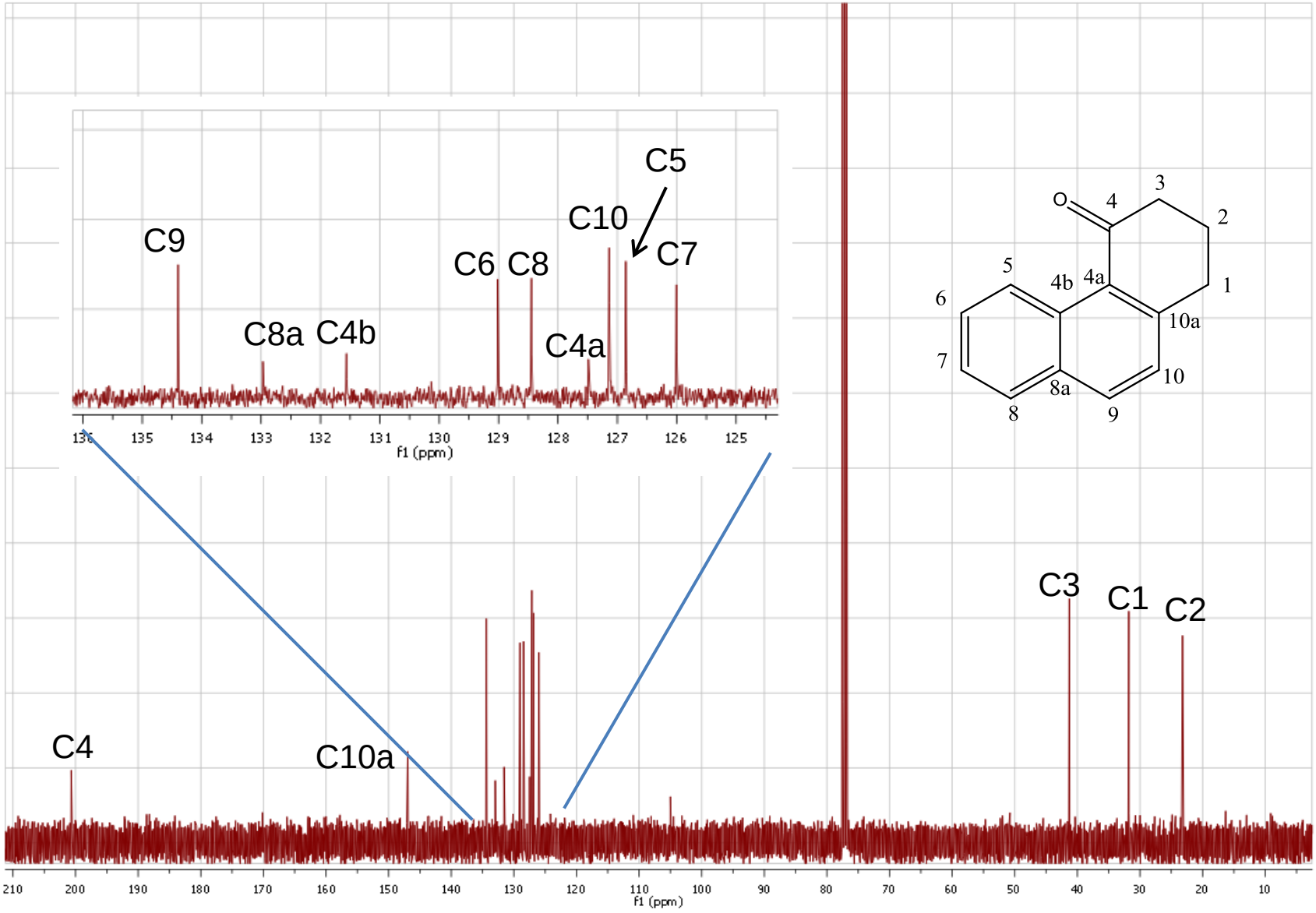


Figure S30. ^{13}C NMR (100 MHz, $CDCl_3$) of NA-2,3-dihydrophenanthren-4(1*H*)-one. (Chemical shifts identical to report with assignments: Ernst, L.; Stolle, R. *Magn. Reson. Chem*. **1989**, *27*, 796.)

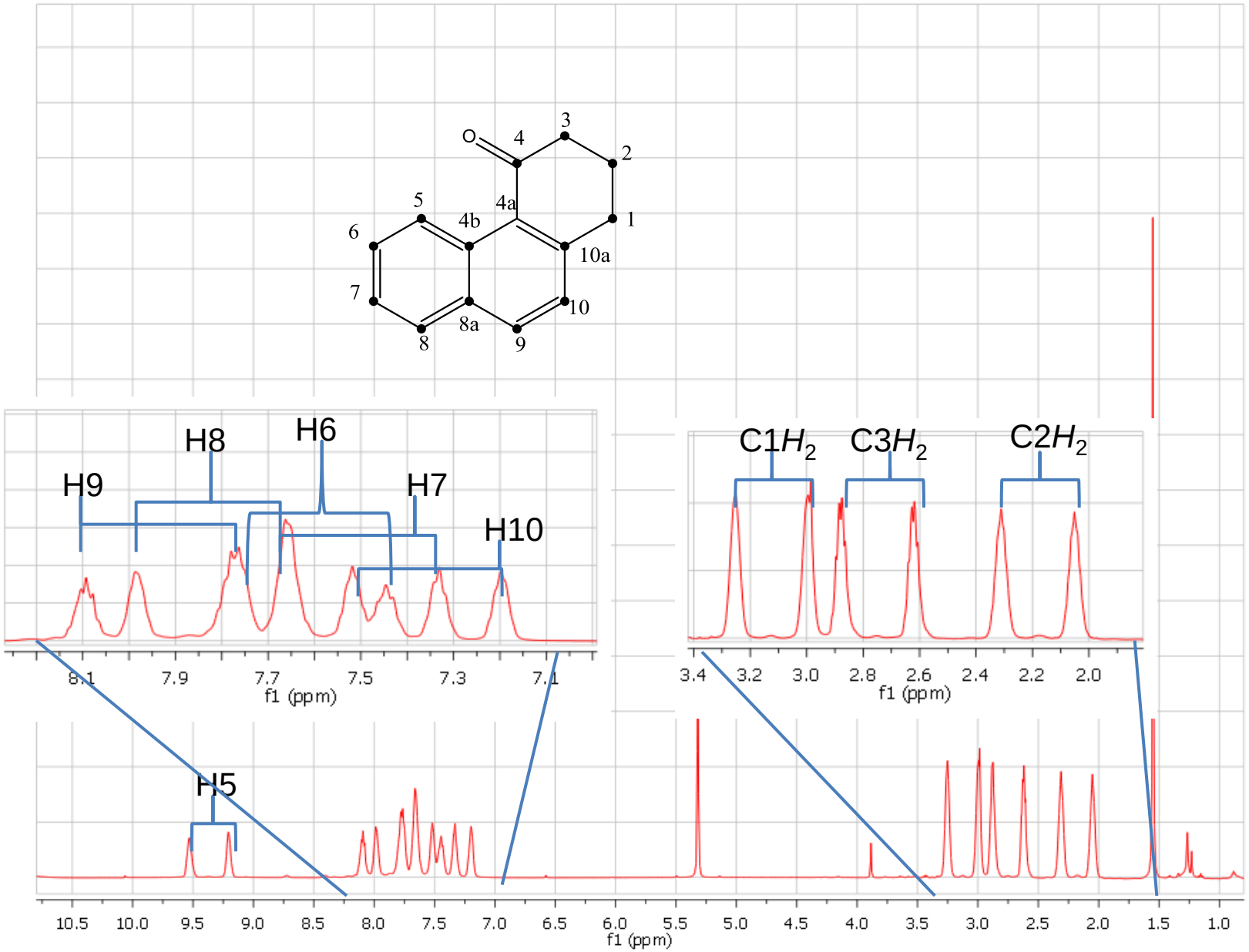


Figure S31. ^{1}H NMR (500 MHz, $CDCl_3$) of U-^{13}C- 2,3-dihydrophenanthren-4(1*H*)-one (**7**).

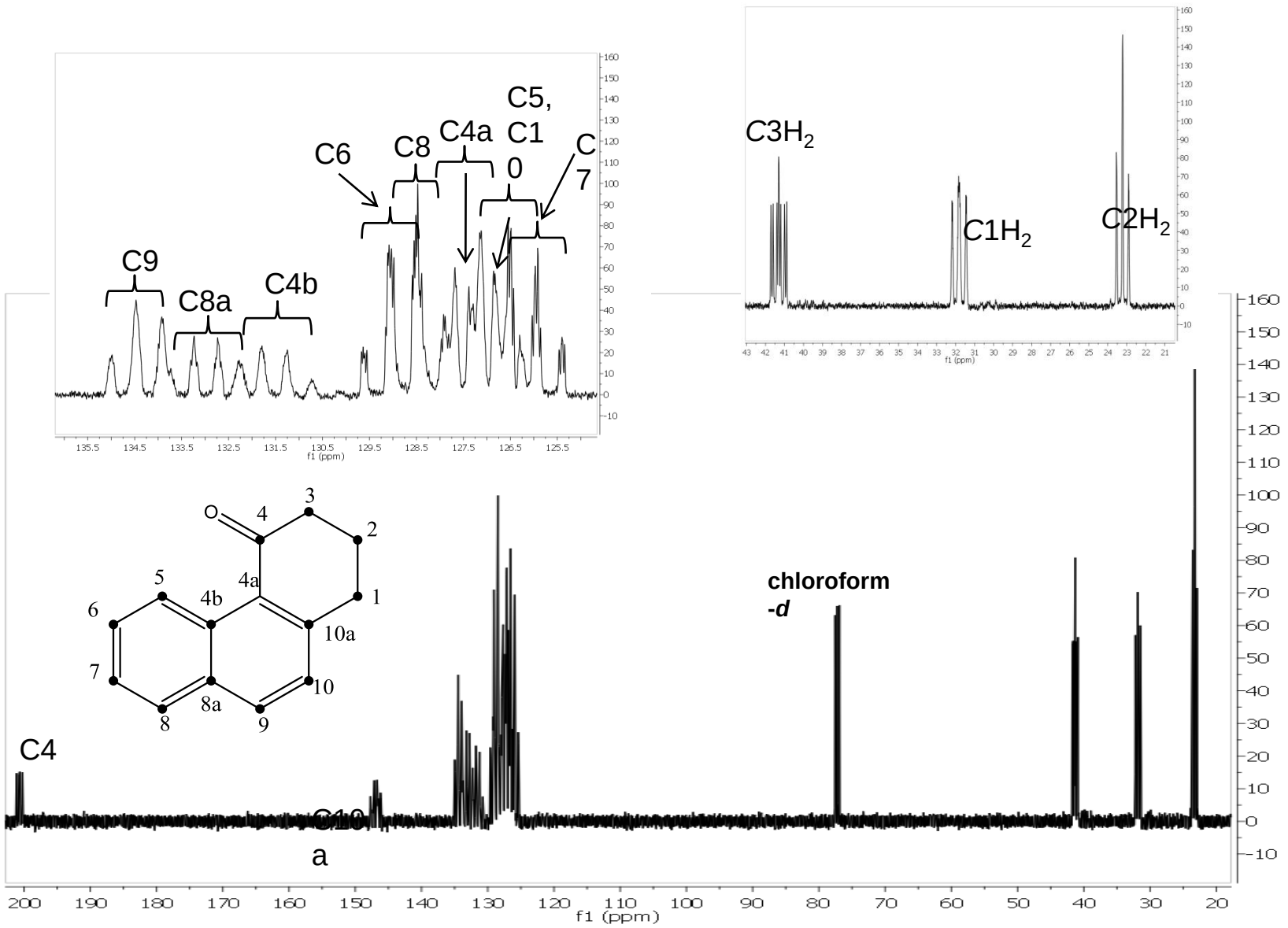


Figure S32. ^{13}C NMR (100 MHz, $CDCl_3$) of U-^{13}C- 2,3-dihydrophenanthren-4(1*H*)-one (**7**).

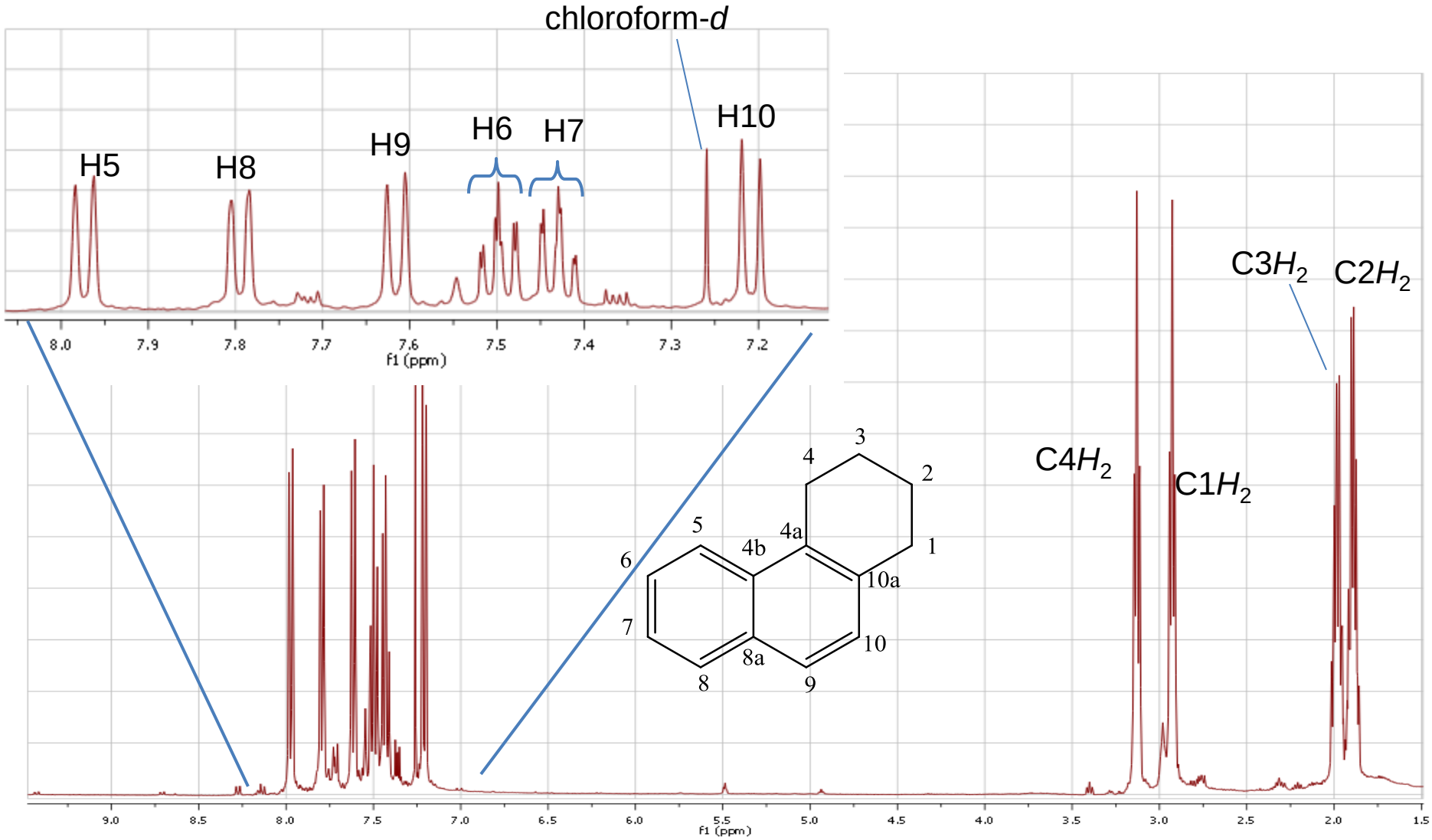


Figure S33. ^{1}H NMR (400 MHz, $CDCl_3$) of NA-1,2,3,4-tetrahydrophenanthrene. (Chemical shifts identical to report with assignments: Ernst, L.; Stolle, R. *Magn. Reson. Chem*. **1989**, *27*, 796.)

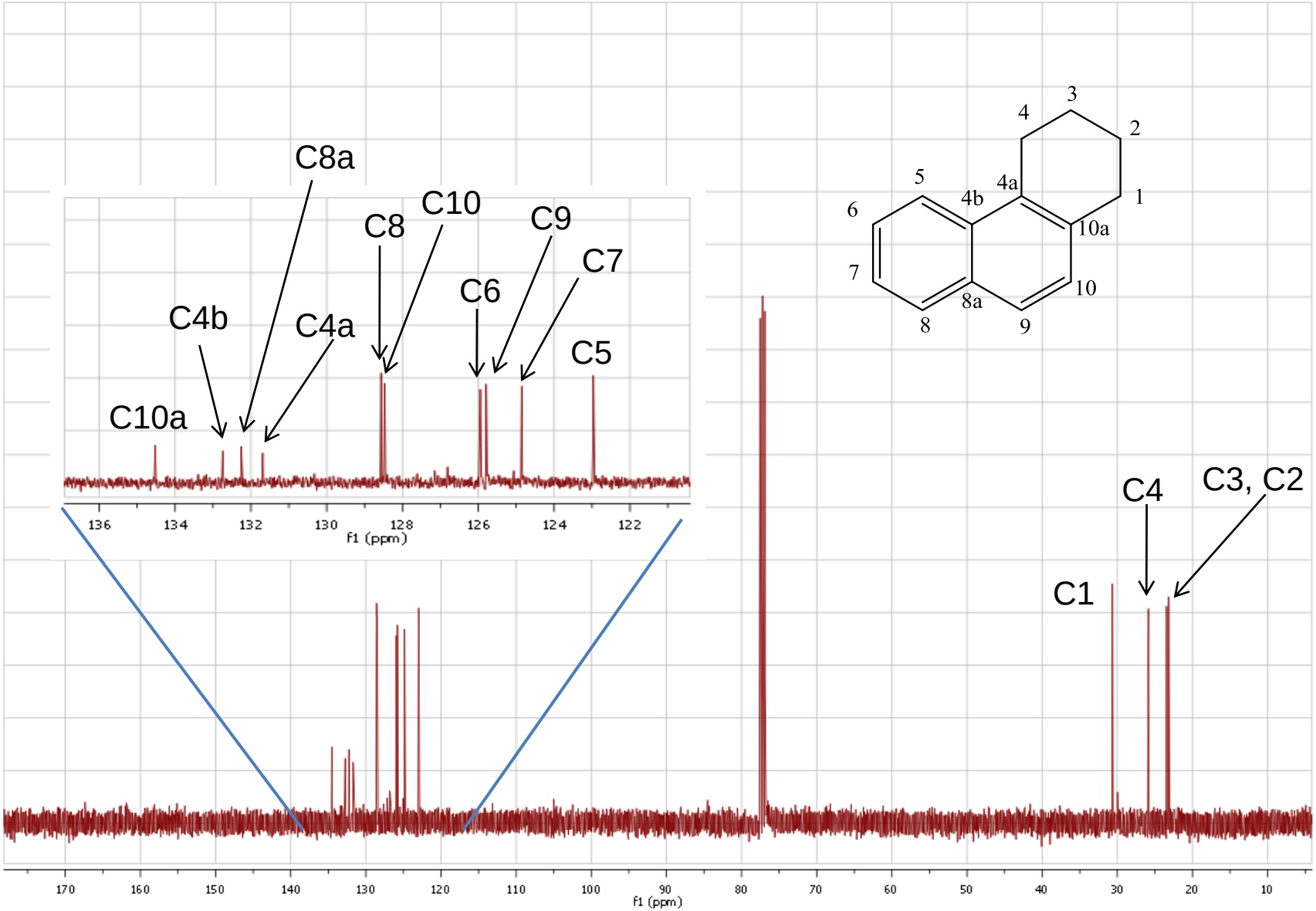


Figure S34. ^{13}C NMR (100 MHz, $CDCl_3$) of NA-1,2,3,4-tetrahydrophenanthrene. (Chemical shifts identical to report with assignments: Ernst, L.; Stolle, R. *Magn. Reson. Chem*. **1989**, *27*, 796.)

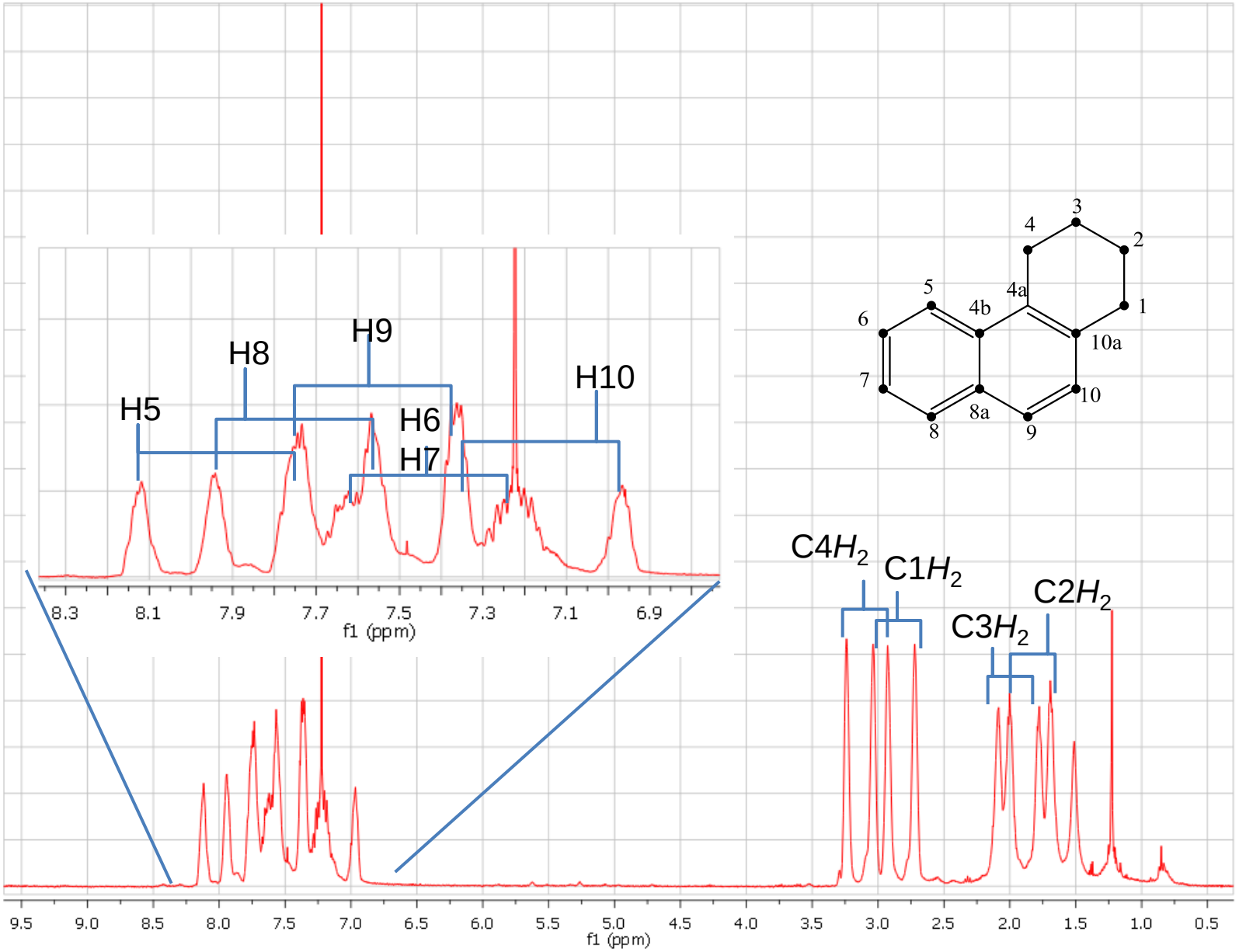


Figure S35. ^{1}H NMR (400 MHz, $CDCl_3$) of U-^{13}C-1,2,3,4-tetrahydrophenanthrene.

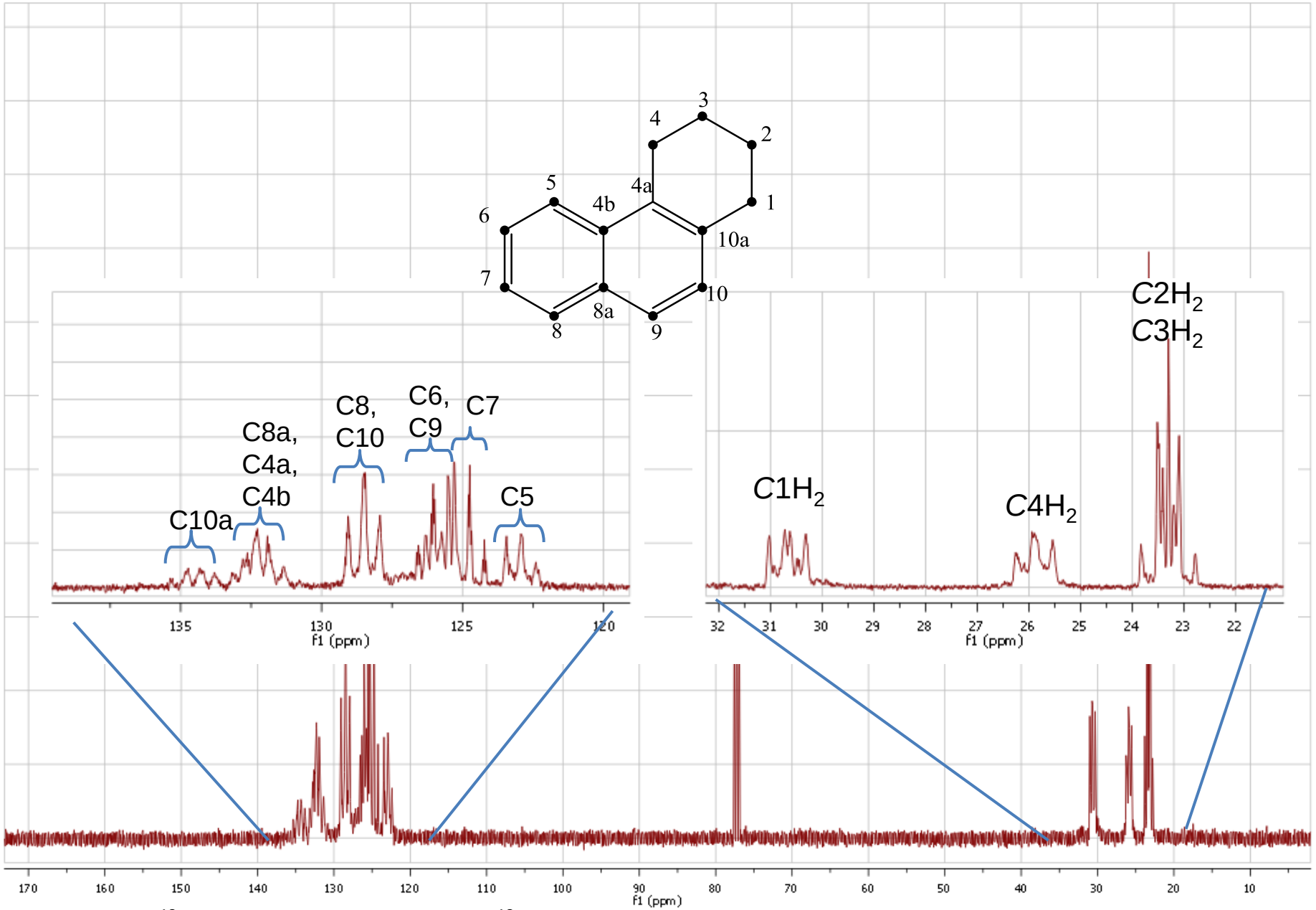


Figure S36. ^{13}C NMR (100 MHz, $CDCl_3$) of U-^{13}C-1,2,3,4-tetrahydrophenanthrene.

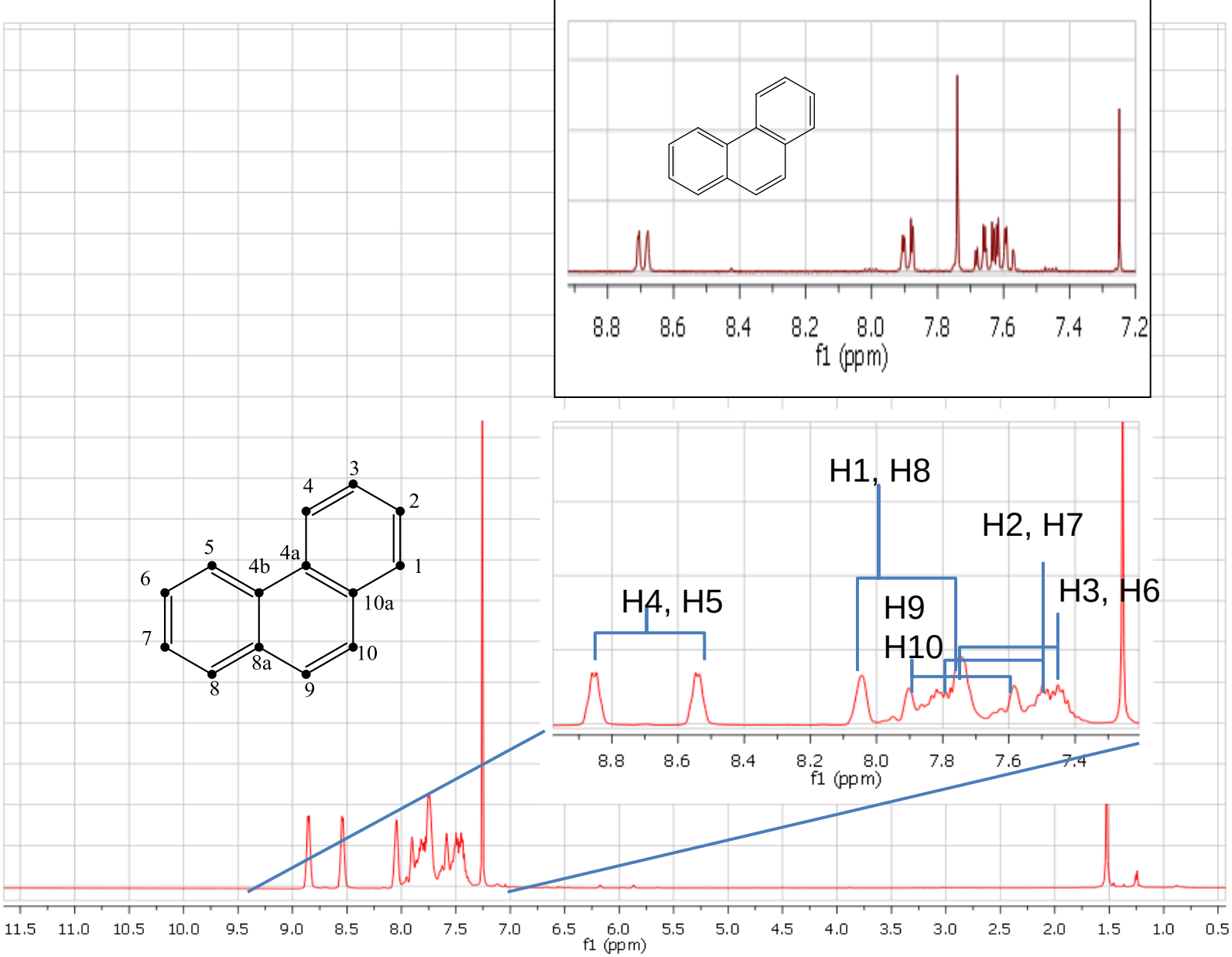


Figure S37. ^{1}H NMR (500 MHz, $CDCl_3$) of U-^{13}C-phenanthrene (**10**). (Inset: ^{1}H NMR of NA-phenanthrene. Chemical shifts identical to report with assignments: Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem*. **2005**, *43*, 588.)

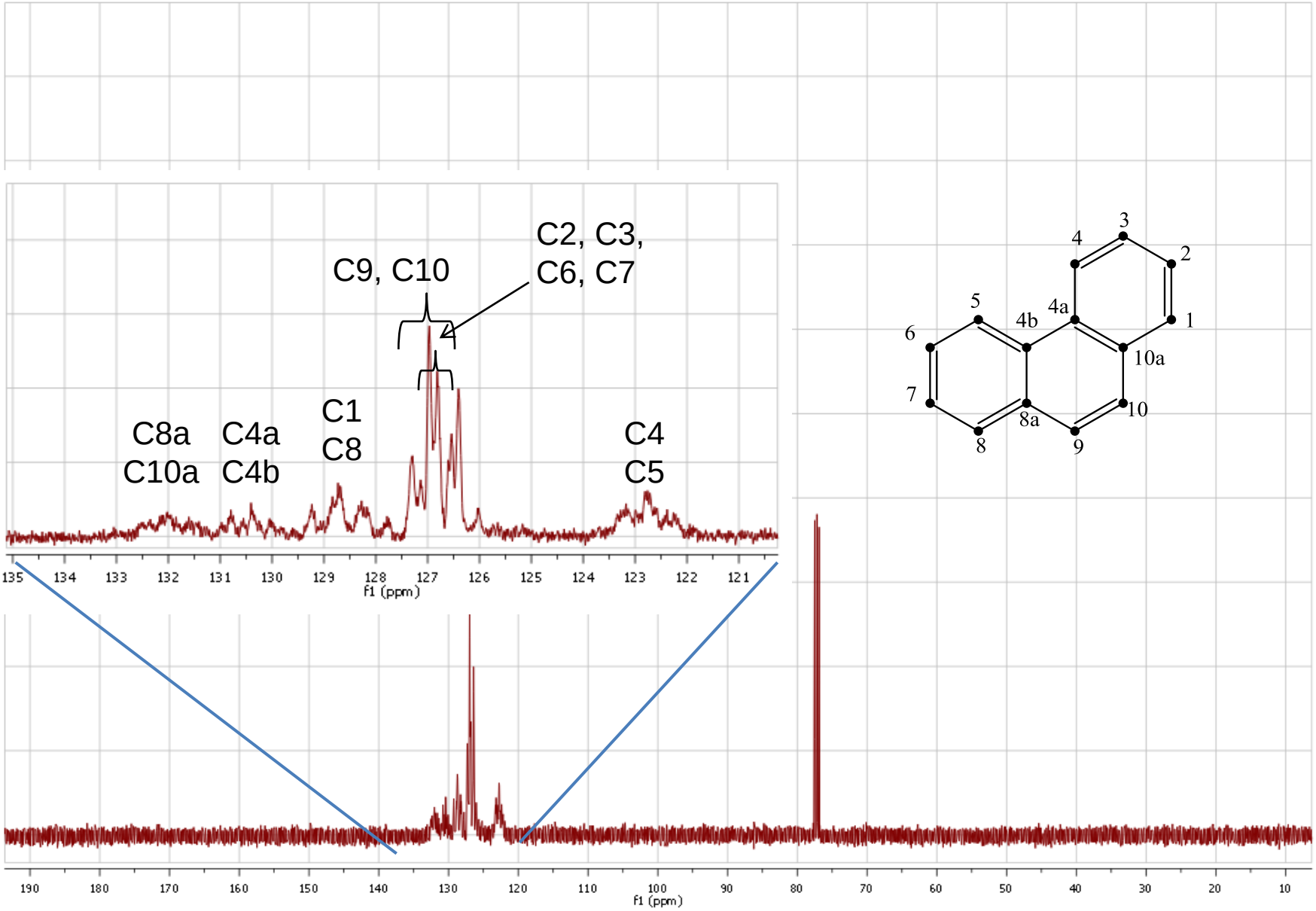


Figure S38. ^{13}C NMR (100 MHz, $CDCl_3$) of U-^{13}C-phenanthrene (**10**). (For ^{13}C NMR assignments of NA-phenanthrene see: Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem*. **2005**, *43*, 588.)

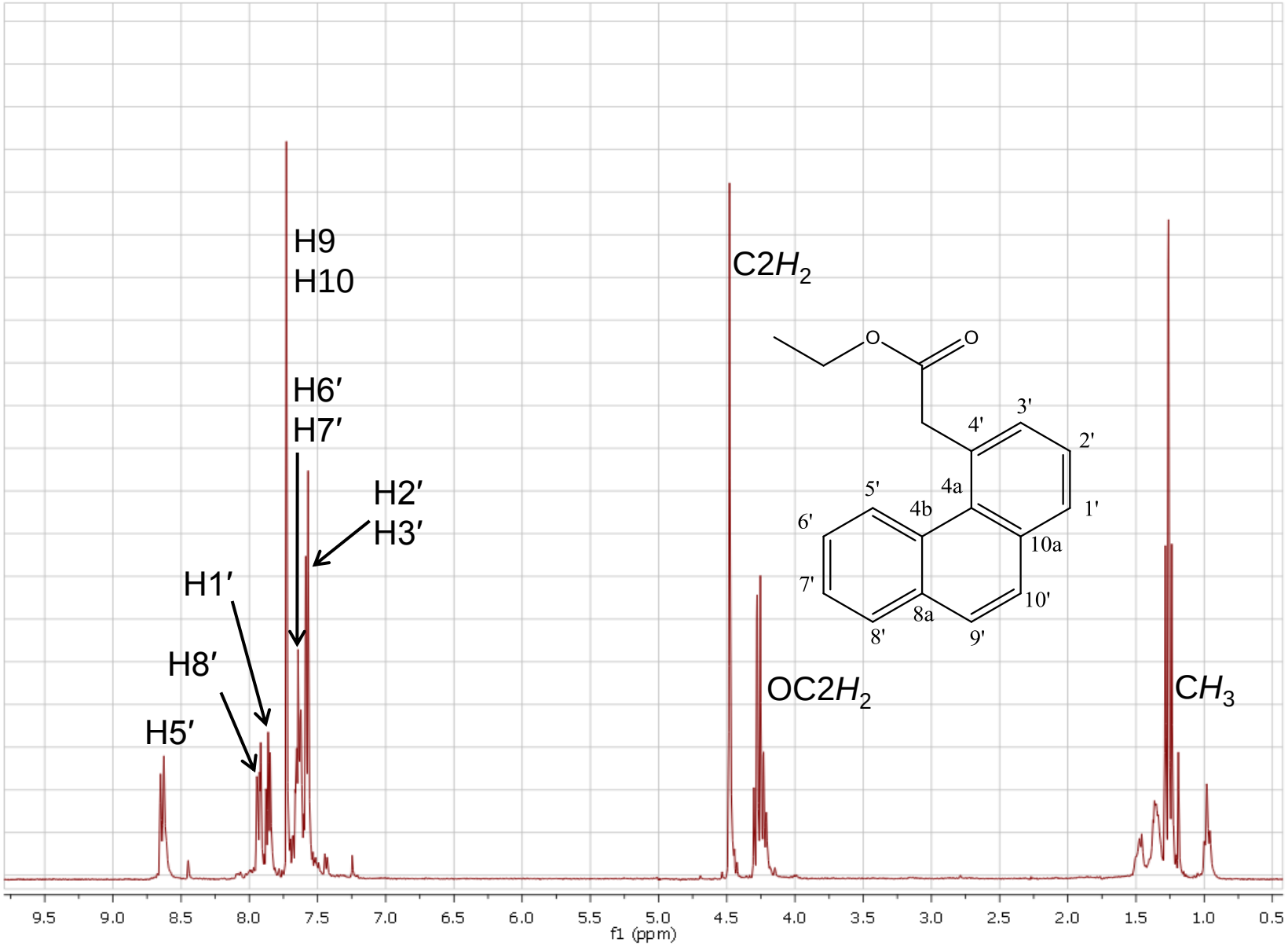


Figure S39. ^{1}H NMR (300 MHz, $CDCl_3$) of NA-ethyl 2-(phenanthren-4-yl)acetate. (Assignments based on 4-Me-phenanthrene: Ibrom, K.; Kohn, G. W.; Boeckmann, K.U.; Kraft, R.; Holba-Schulz, P.; Ernst, L. *Org. Lett*. **2000**, *2*, 4111.)

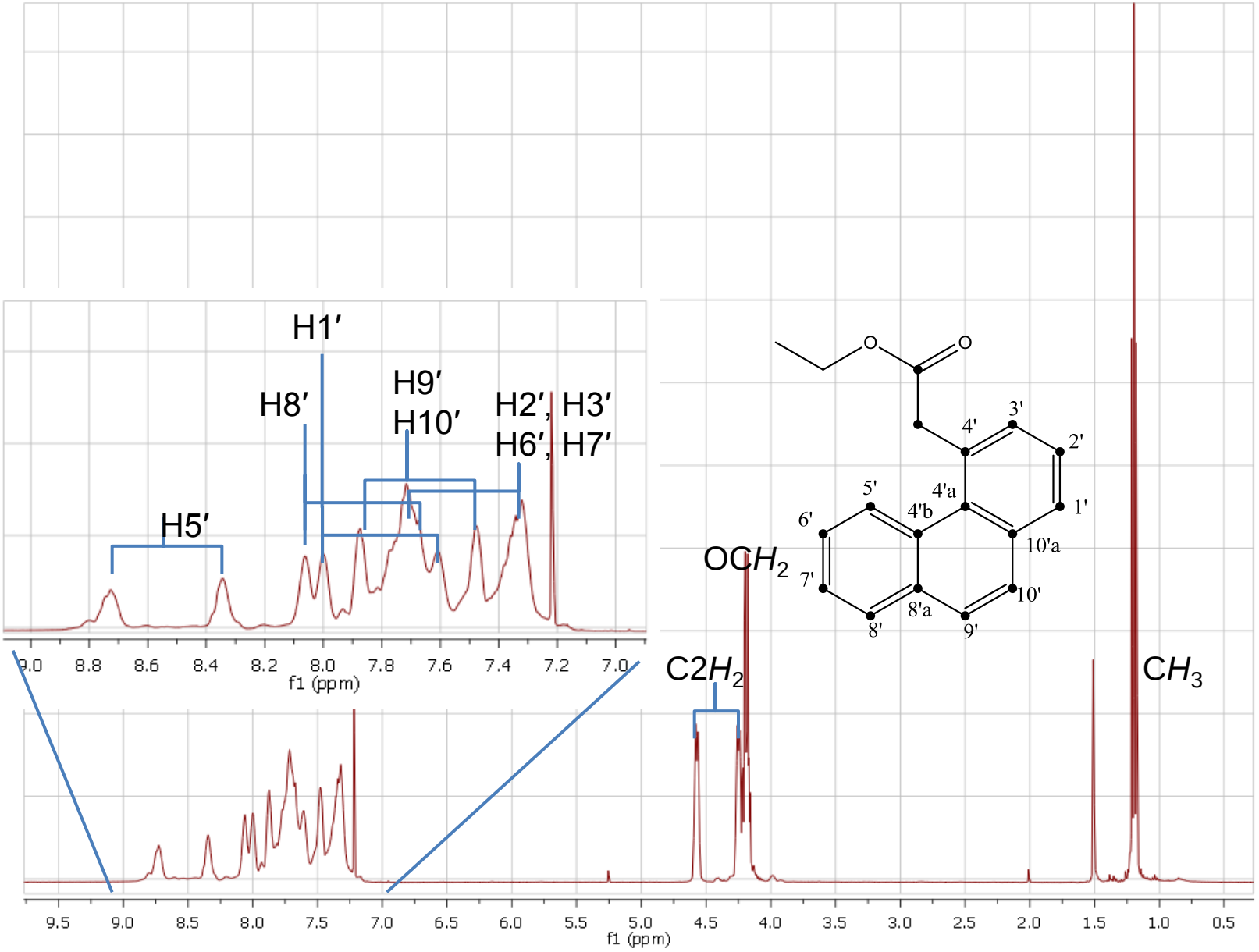


Figure S40. ^{1}H NMR (400 MHz, $CDCl_3$) of ethyl U-^{13}C-2-(phenanthren-4-yl)acetate (**12**).

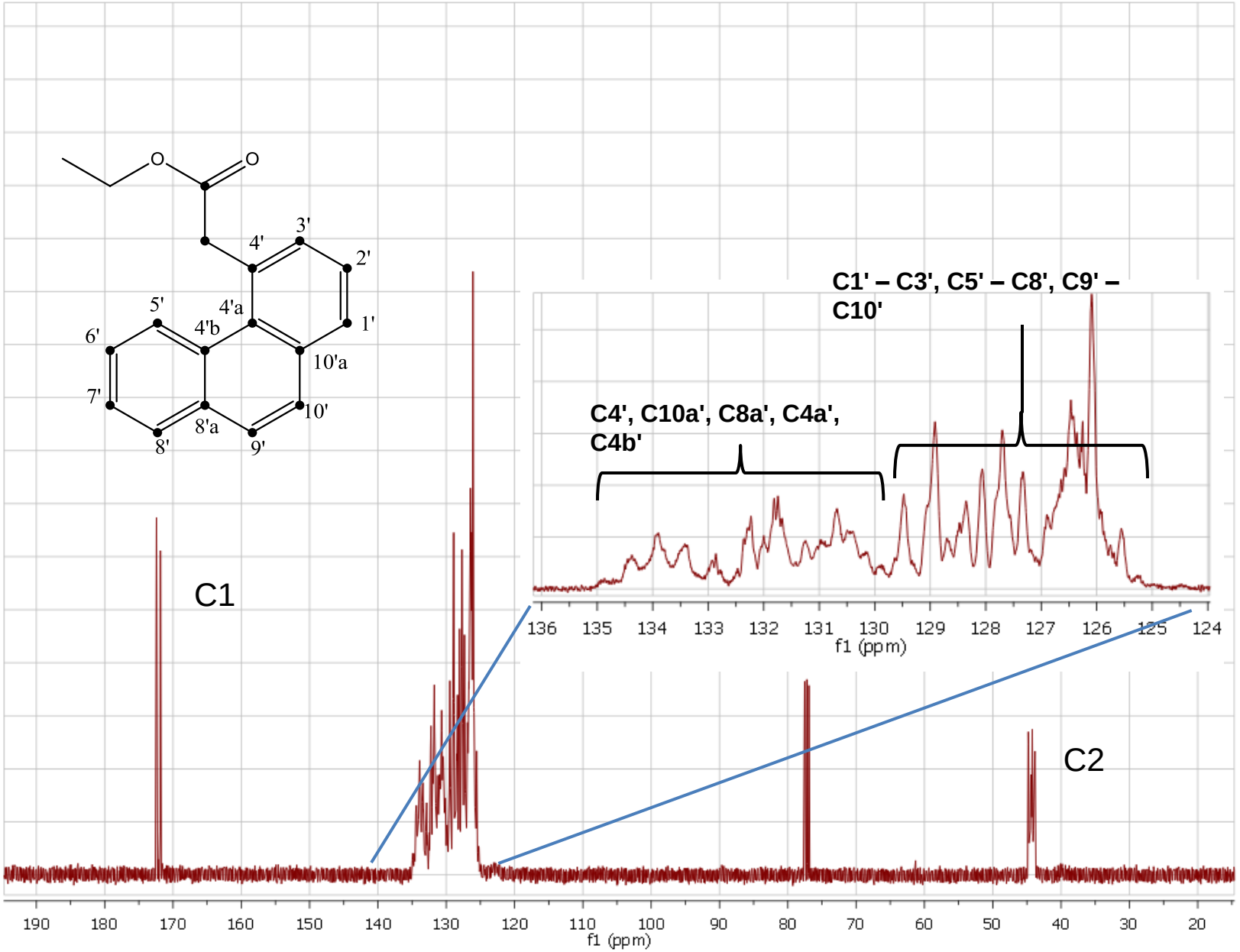


Figure S41. ^{13}C NMR (100 MHz, $CDCl_3$) of ethyl U-^{13}C-2-(phenanthren-4-yl)acetate (**12**). (Assignments based on 4-Me-phenanthrene: Ibrom, K.; Kohn, G. W.; Boeckmann, K.U.; Kraft, R.; Holba-Schulz, P.; Ernst, L. *Org. Lett*. **2000**, *2*, 4111.)

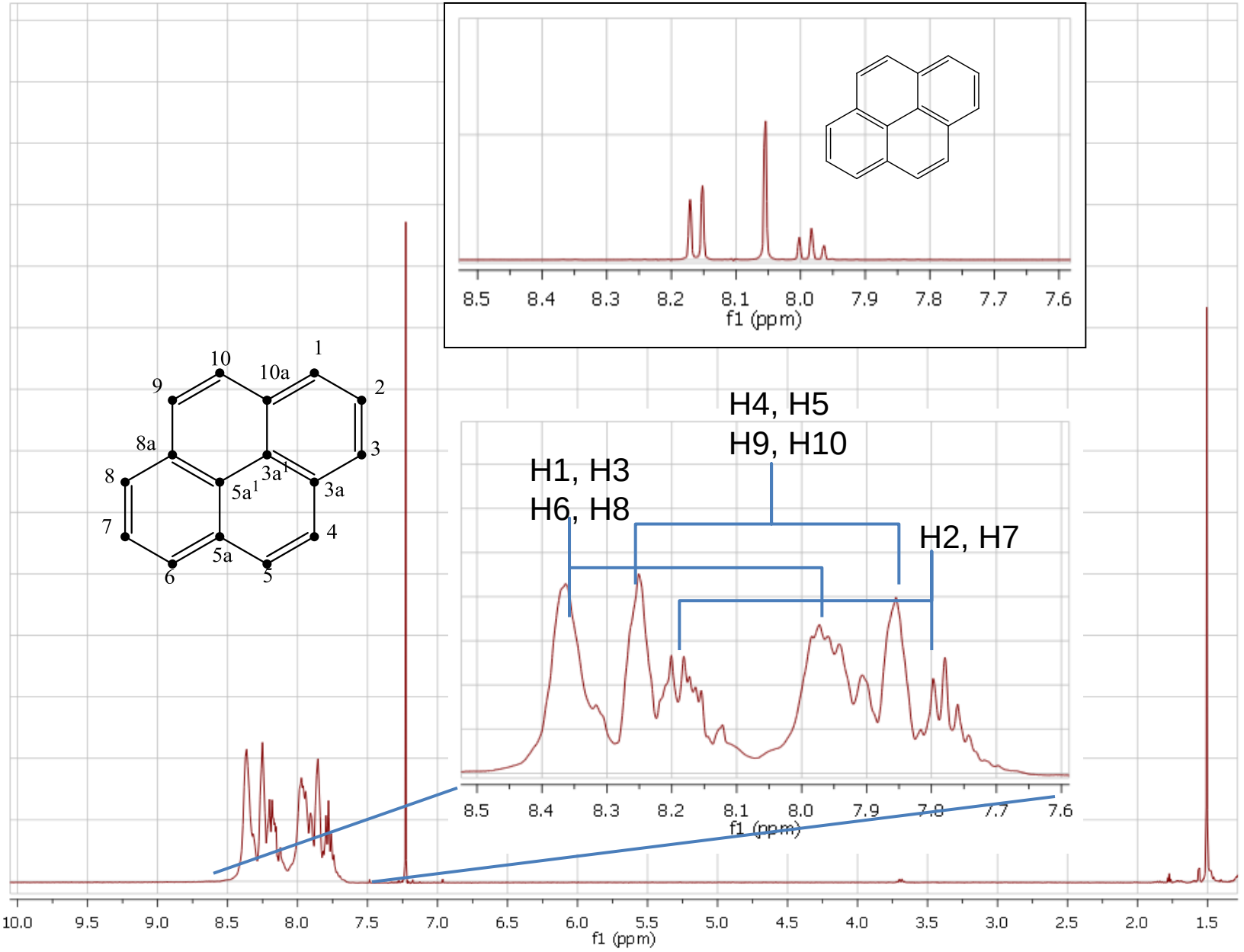


Figure S42. ^{1}H NMR (400 MHz, $CDCl_3$) of U-^{13}C-pyrene (**13**). (Inset: ^{1}H NMR of NA-pyrene. Chemical shifts identical to report with assignments: Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem*. **2005**, *43*, 588.)

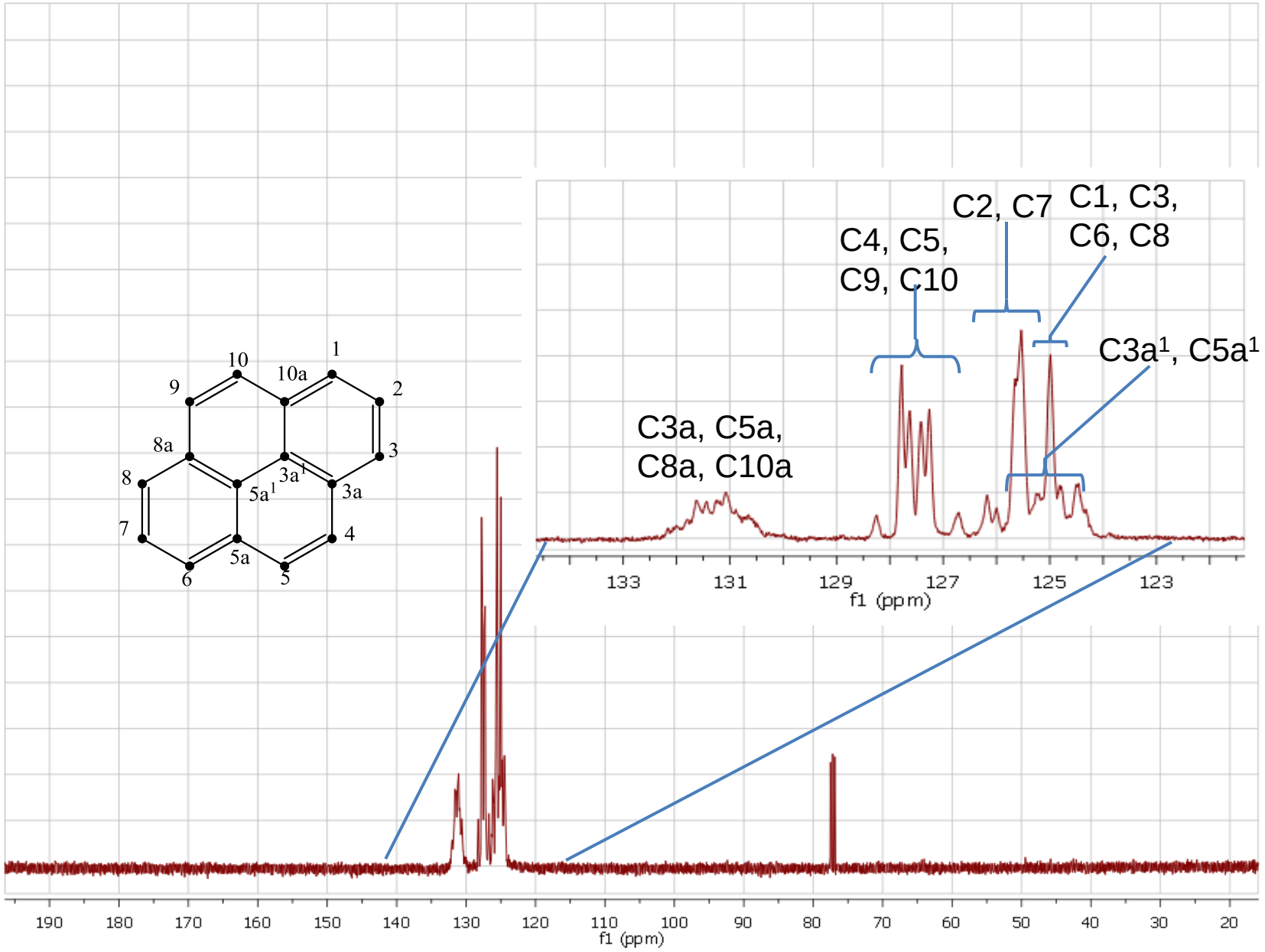


Figure S43. ^{13}C NMR (100 MHz, $CDCl_3$) of U-^{13}C-pyrene (**13**). (For ^{13}C NMR assignments of NA-pyrene see: Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem*. **2005**, *43*, 588.)

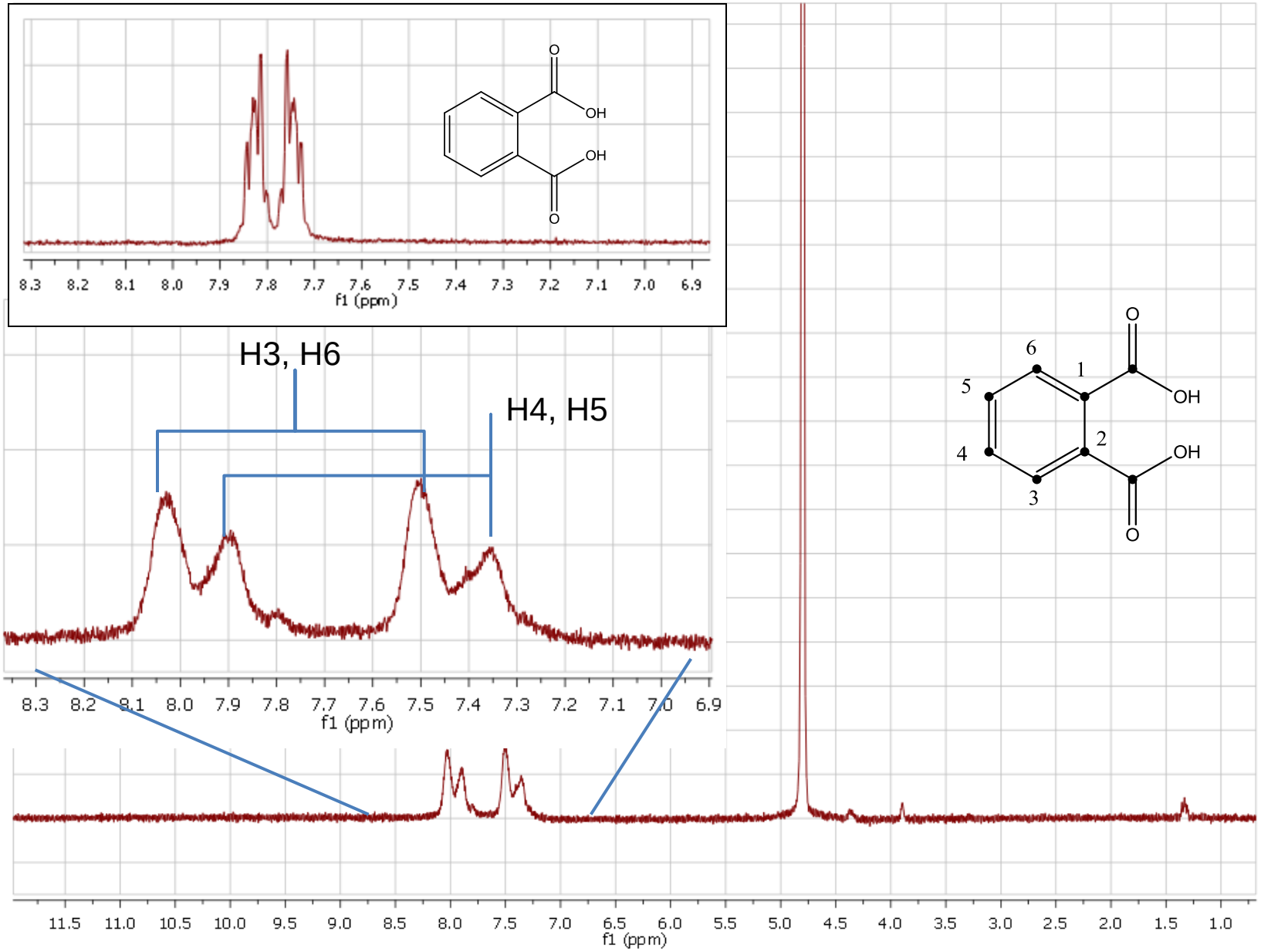


Figure S44. ^{1}H NMR (300 MHz, DMSO-d_6) of U-^{13}C-phthalic acid (**14**). (inset: ^{1}H NMR of NA-phthalic acid. Chemical shifts itentical to report: AIST: Integrated Spectral Database System of Organic Compounds.)

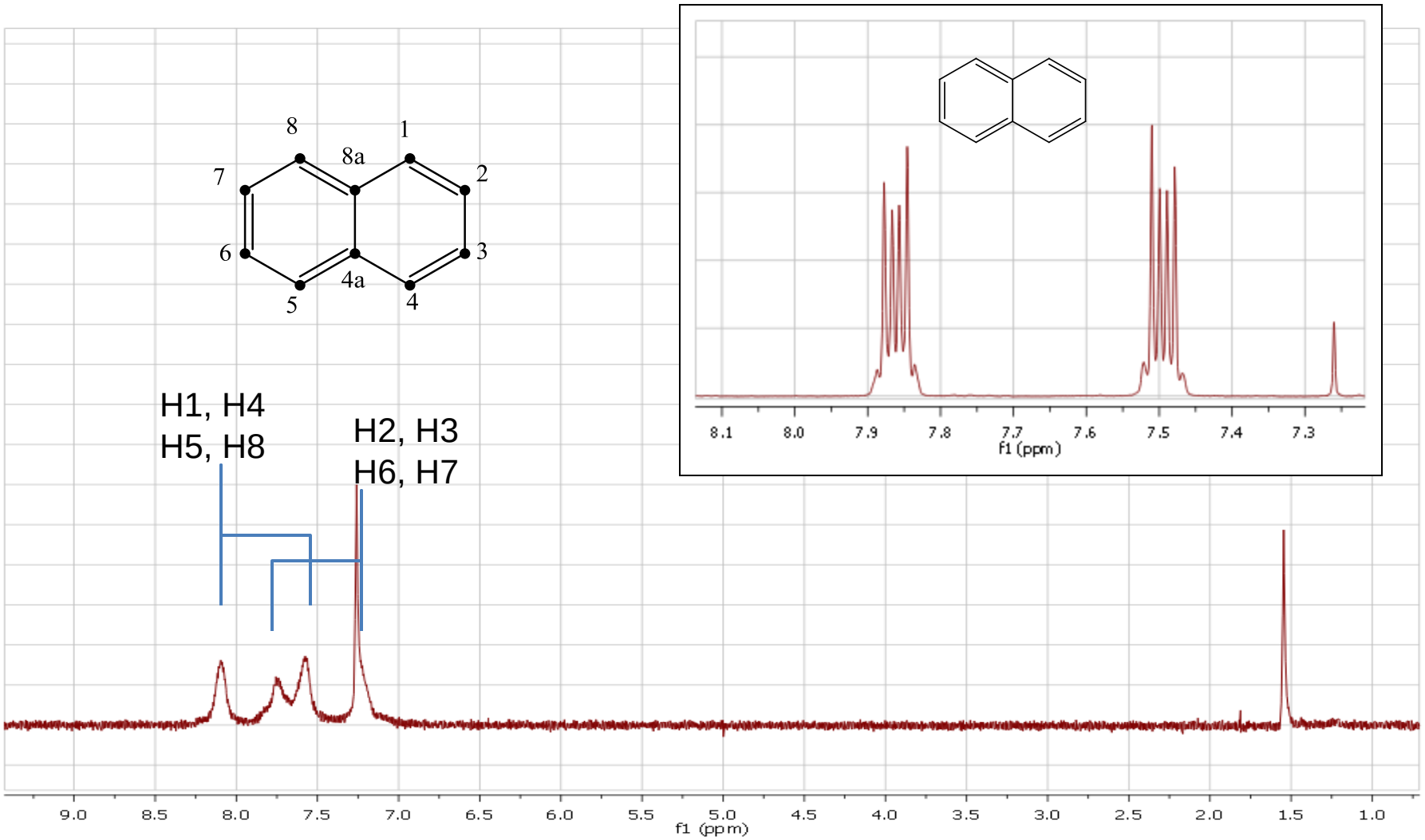


Figure S45. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-naphthalene (**6**). (Inset: ^{1}H NMR of NA-naphthalene. Chemical Shift identical to report with assignments: Pouchert, C.J.; Behnke, J. (eds). *The Aldrich Library of* ^{13}C *and* ^{1}H *FT NMR Spectra*. Aldrich Chemical: Milwaukee, WI, 1993.

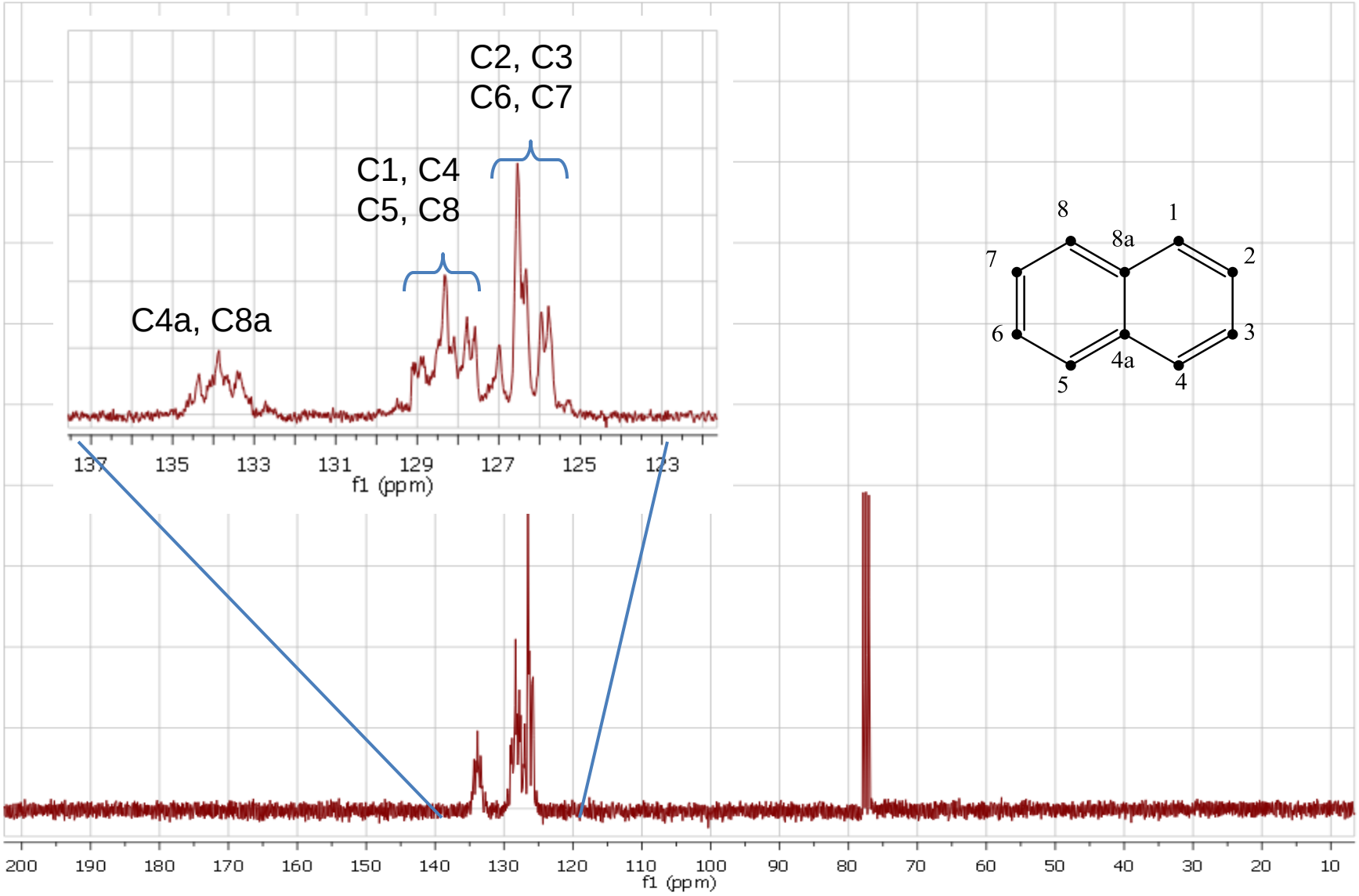


Figure S46. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-naphthalene (**6**). (For ^{13}C NMR assignments of NA-naphthalene see: Kitching, W.; Bullpitt, M.; Doddrell, D.; Adcock, W. *Org. Magn. Reson*. **1974**, *6*, 289. Seita, J.; Sandstrom, J.; Drakenberg, T. *Org. Magn. Reson.* **1978**, *11*, 239.)

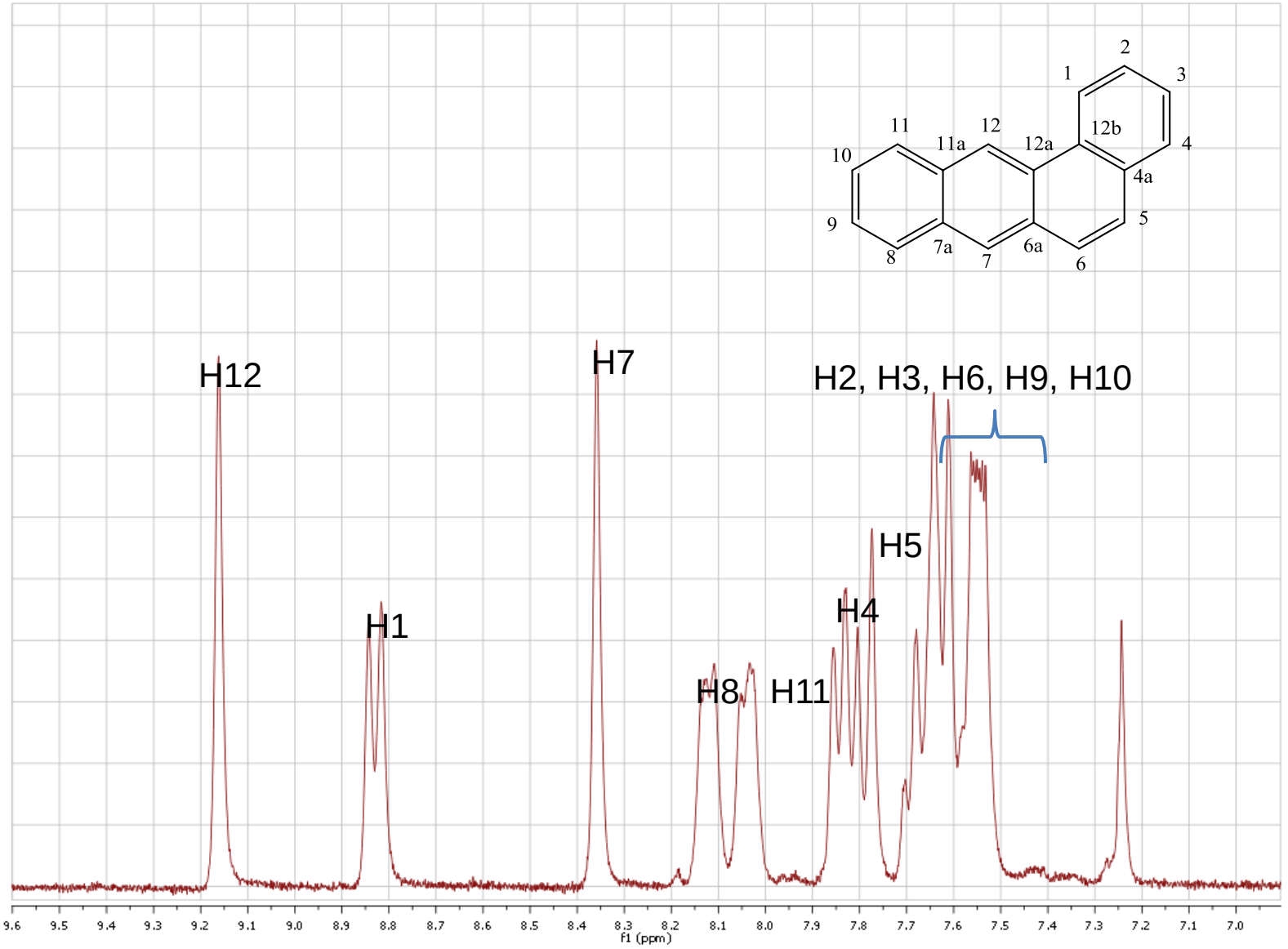


Figure S47. ^{1}H NMR (500 MHz, $CDCl_3$) of NA-benz[*a*]anthracene. (Chemical Shifts identical to report with assignment: Moody, J. D.; Freeman, J. P.; Cerniglia, C. E *Biodegradation* **2005**, *16*, 513.)

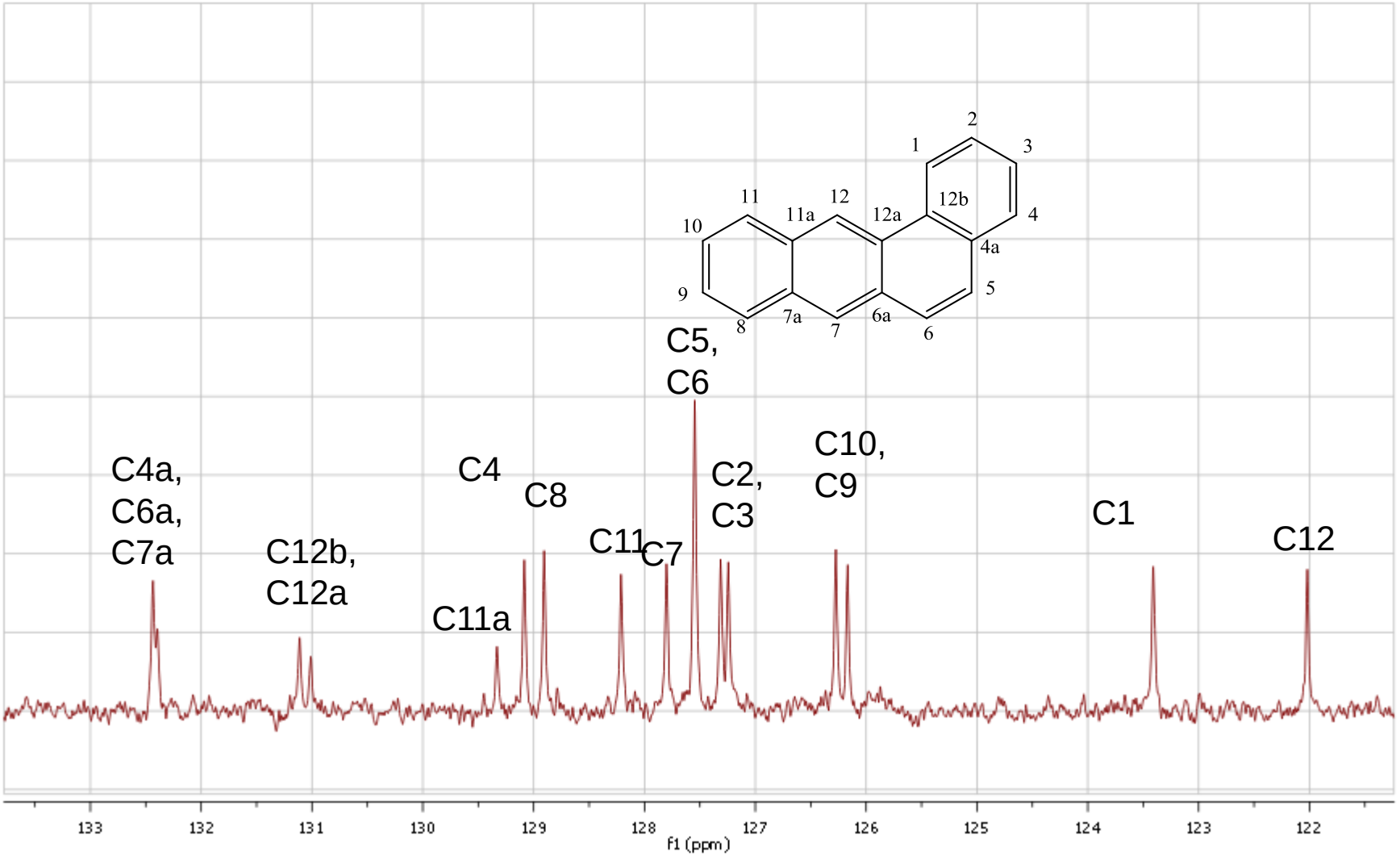


Figure S48. ^{13}C NMR (75 MHz, $CDCl_3$) of NA-benz[*a*]anthracene. (Chemical shifts identical to report with assignments: Cox, R. H.; Levy, L. A.; *Org. Megn. Reson.***1983**, *21,* 173.)

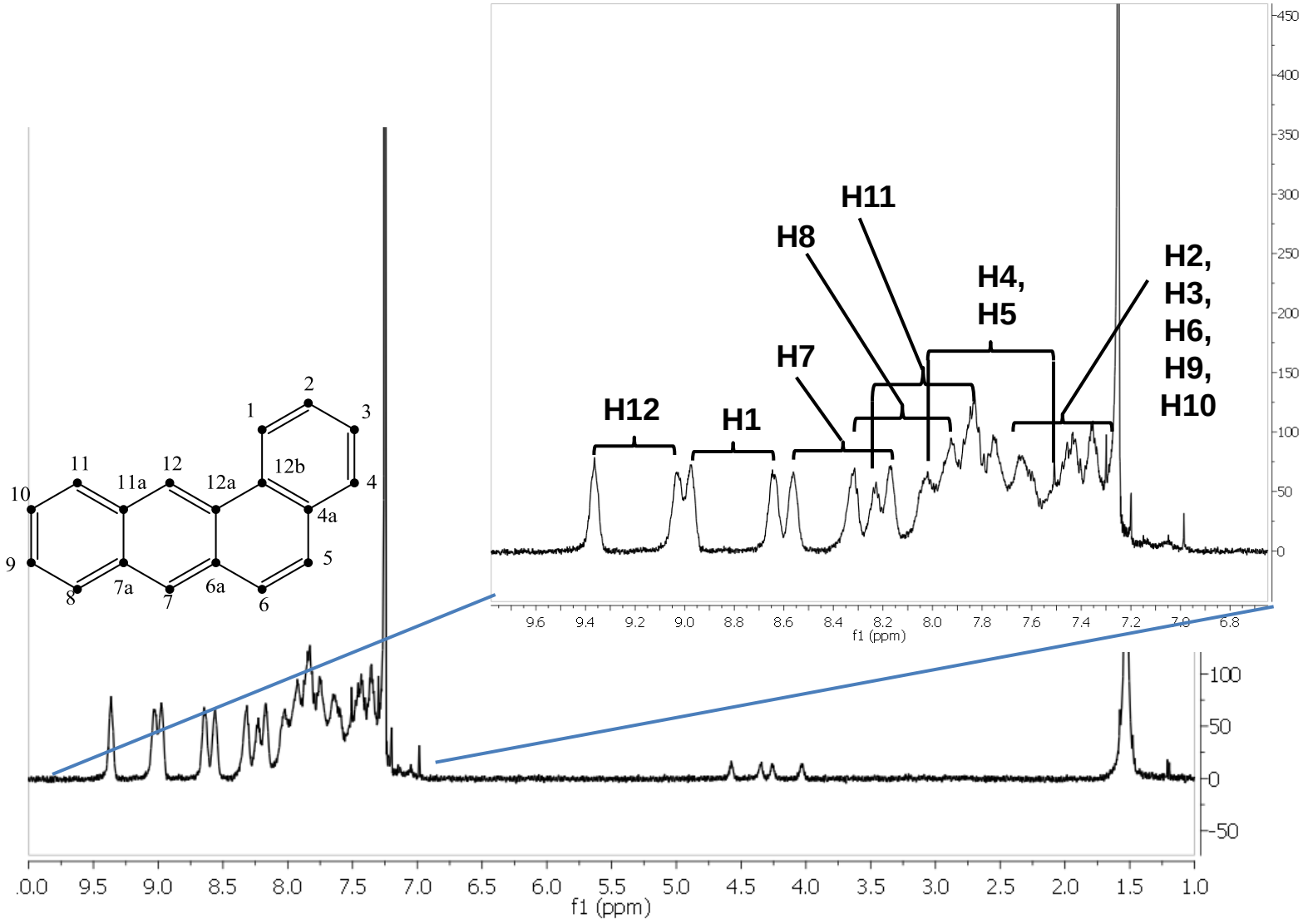
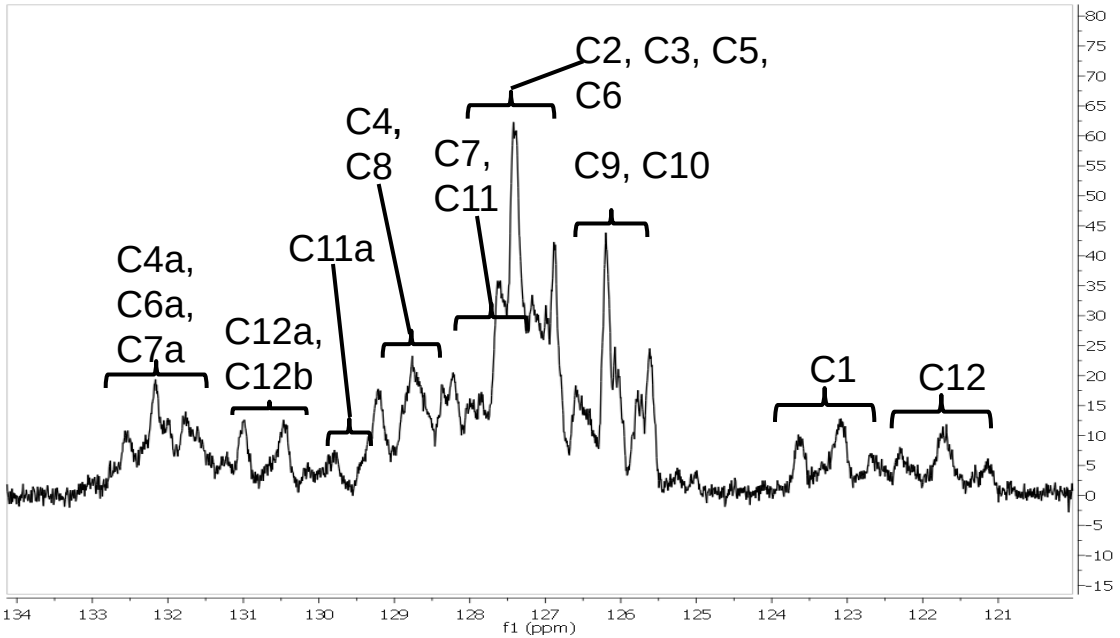
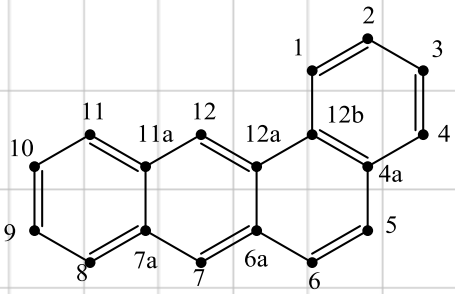


Figure S49. ^{1}H NMR (500 MHz, $CDCl_3$) of U-^{13}C-benz[*a*]anthracene (**16**).





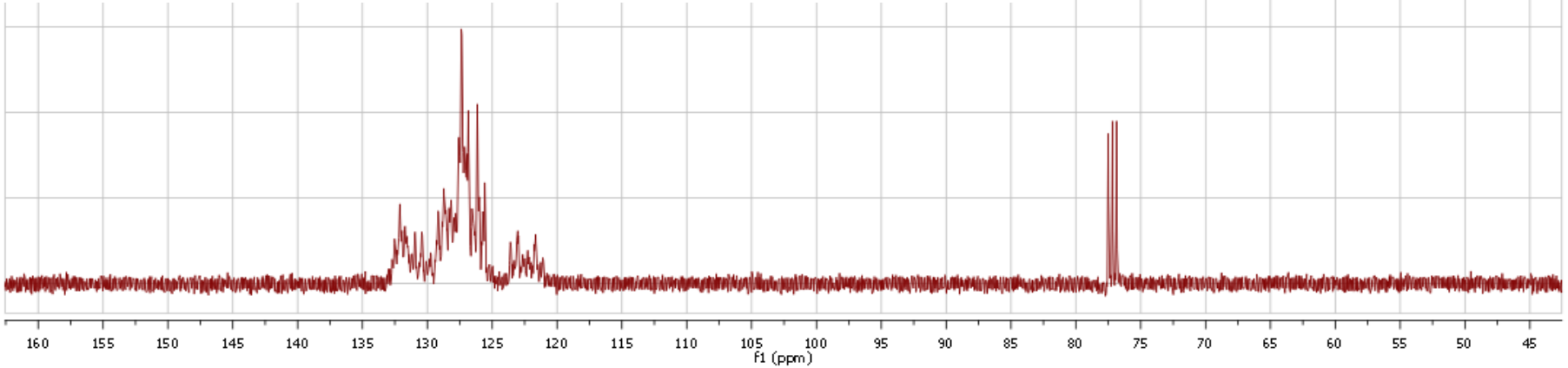


Figure S50. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-benz[*a*]anthracene (**16**).

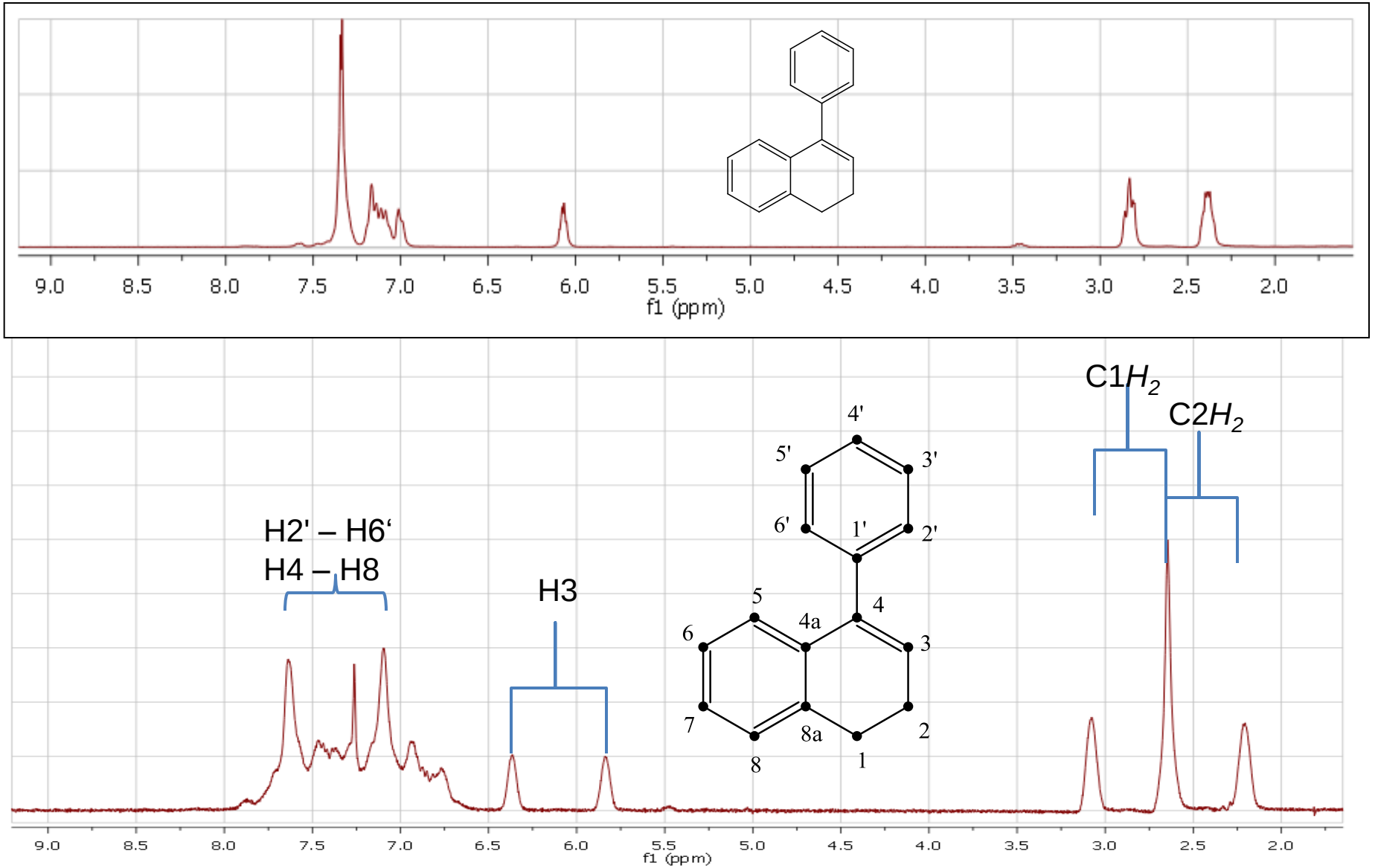


Figure S51. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-4-phenyl-1,2-dihydronaphthalene (**20**). (Inset: ^{1}H NMR of NA-4-phenyl-1,2-dihydronaphthalene. Chemical shifts identical report: Cook, M. J.; Katritzky, A. R.; Pennington, F. C.; Semple, B. M. *J. Chem. Soc.* B. **1969,** 523.)

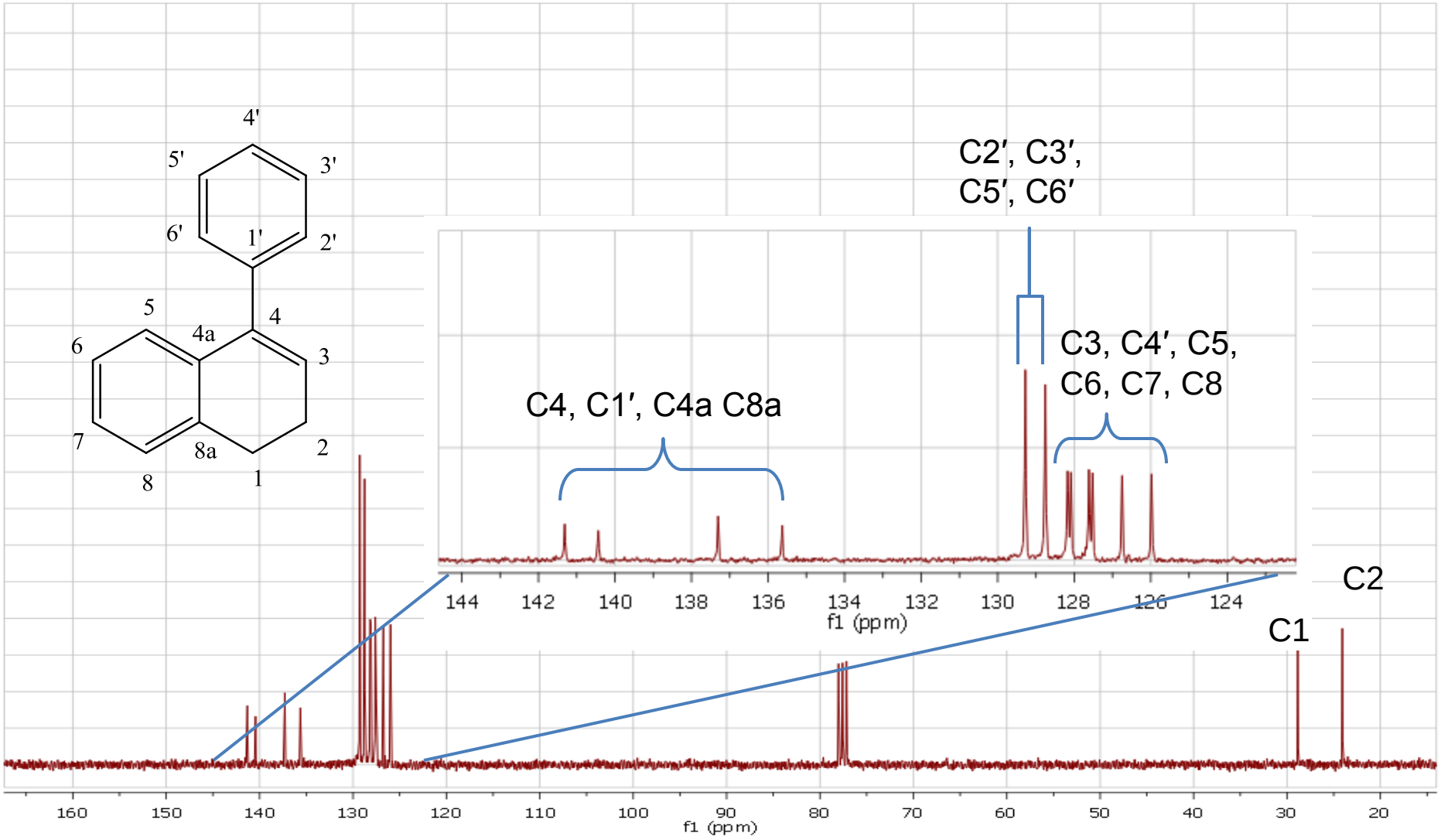


Figure S52. ^{13}C NMR (75 MHz, $CDCl_3$) of NA-4-phenyl-1,2-dihydronaphthalene. (Chemical shifts identical to report: Rao, M. L. N.; Jadhav, Deepak N.; Venkatesh, Varadhachari *Eur. J. Org. Chem*. **2009**, 4300.)

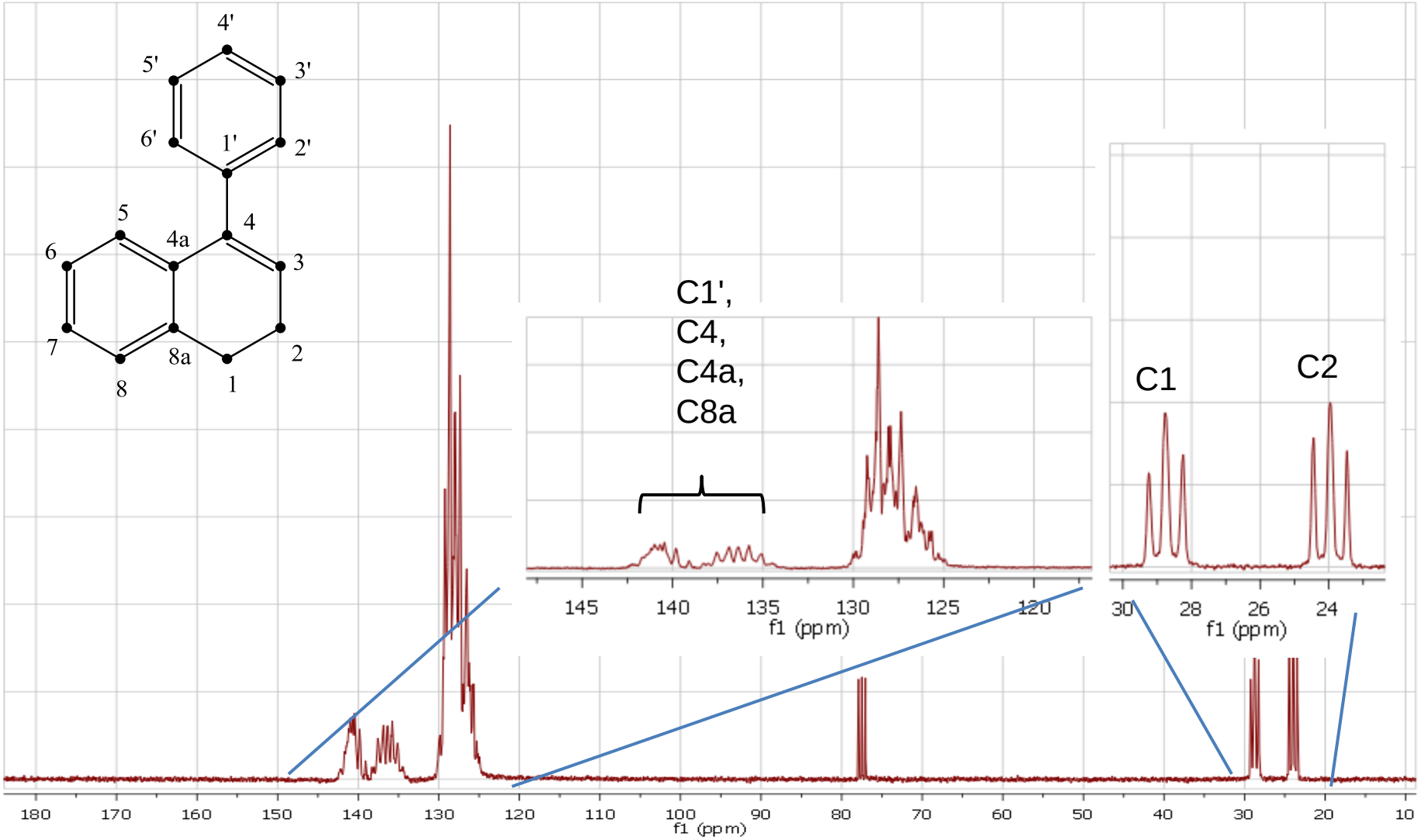


Figure S53. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-4-phenyl-1,2-dihydronaphtha lene (**20**).

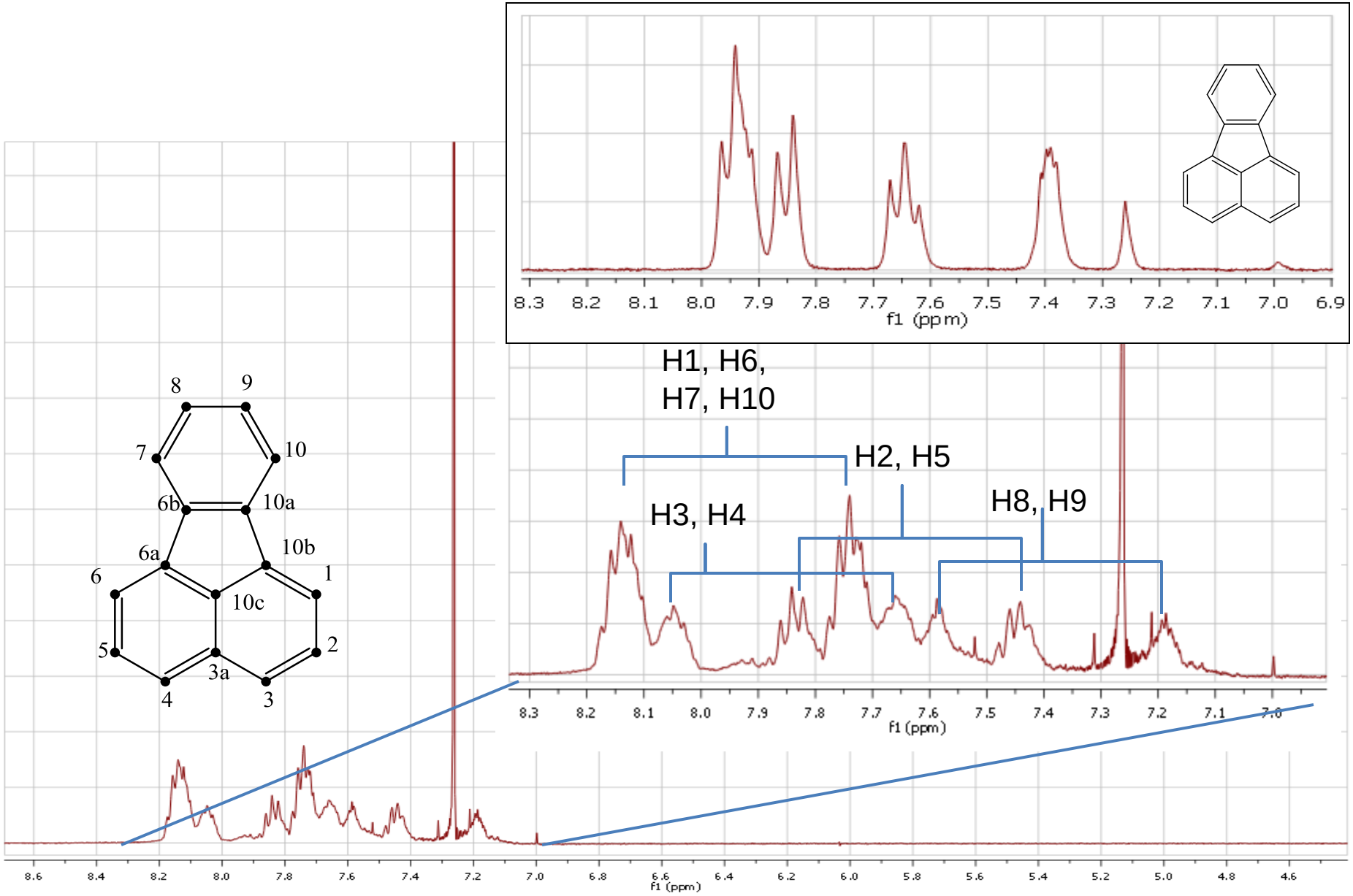


Figure S55. ^{1}H NMR (300 MHz, $CDCl_3$) of U-^{13}C-fluoranthene (**22**). (Inset: ^{1}H NMR of NA-fluoranthene. Chemical shifts identical to report with assignments : Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem*. **2005**, *43*, 588.)

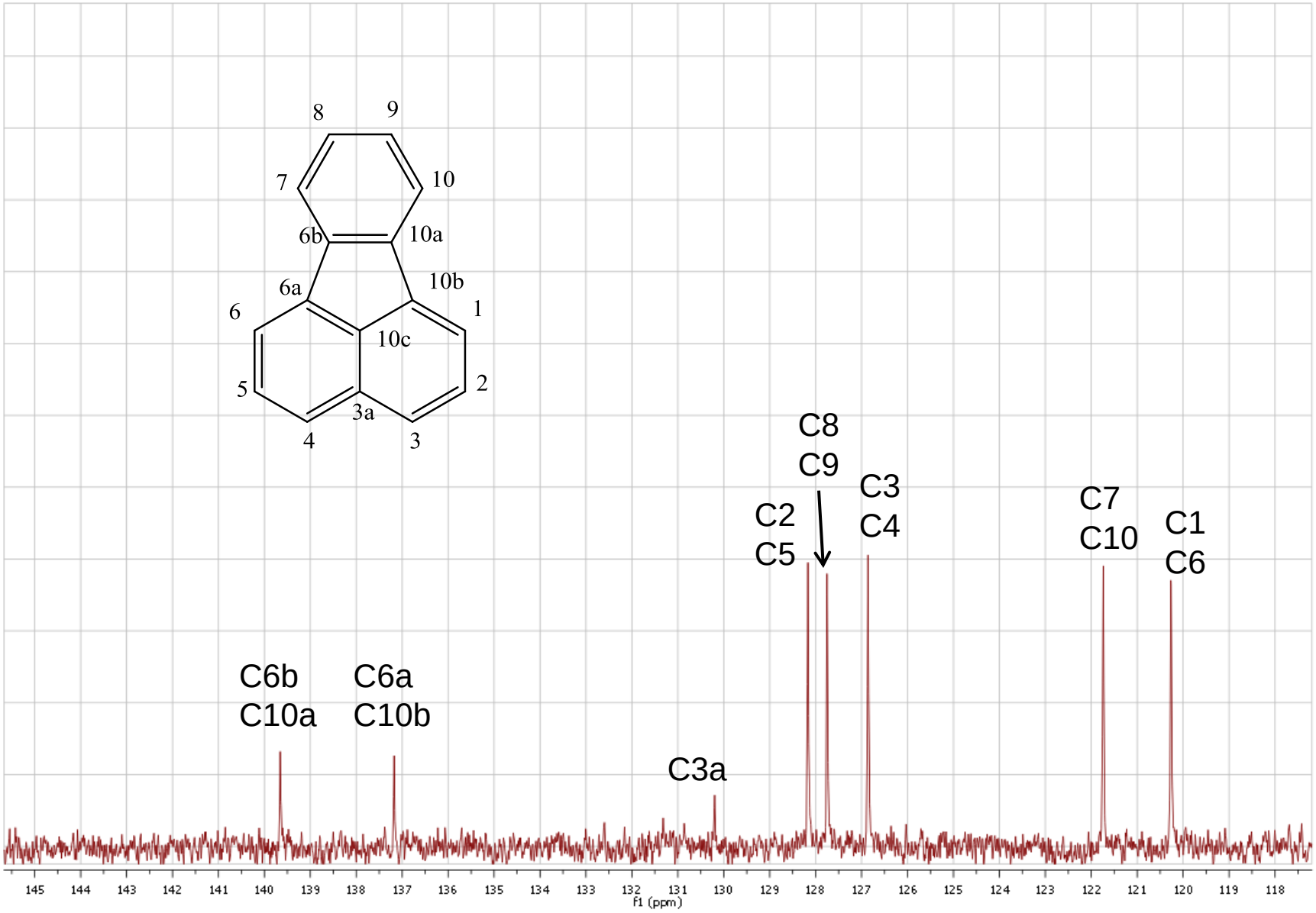


Figure S54. ^{13}C NMR (100 MHz, $CDCl_3$) of NA-fluoranthene . (Chemical shifts identical report with assignments : Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem*. **2005**, *43*, 588.)

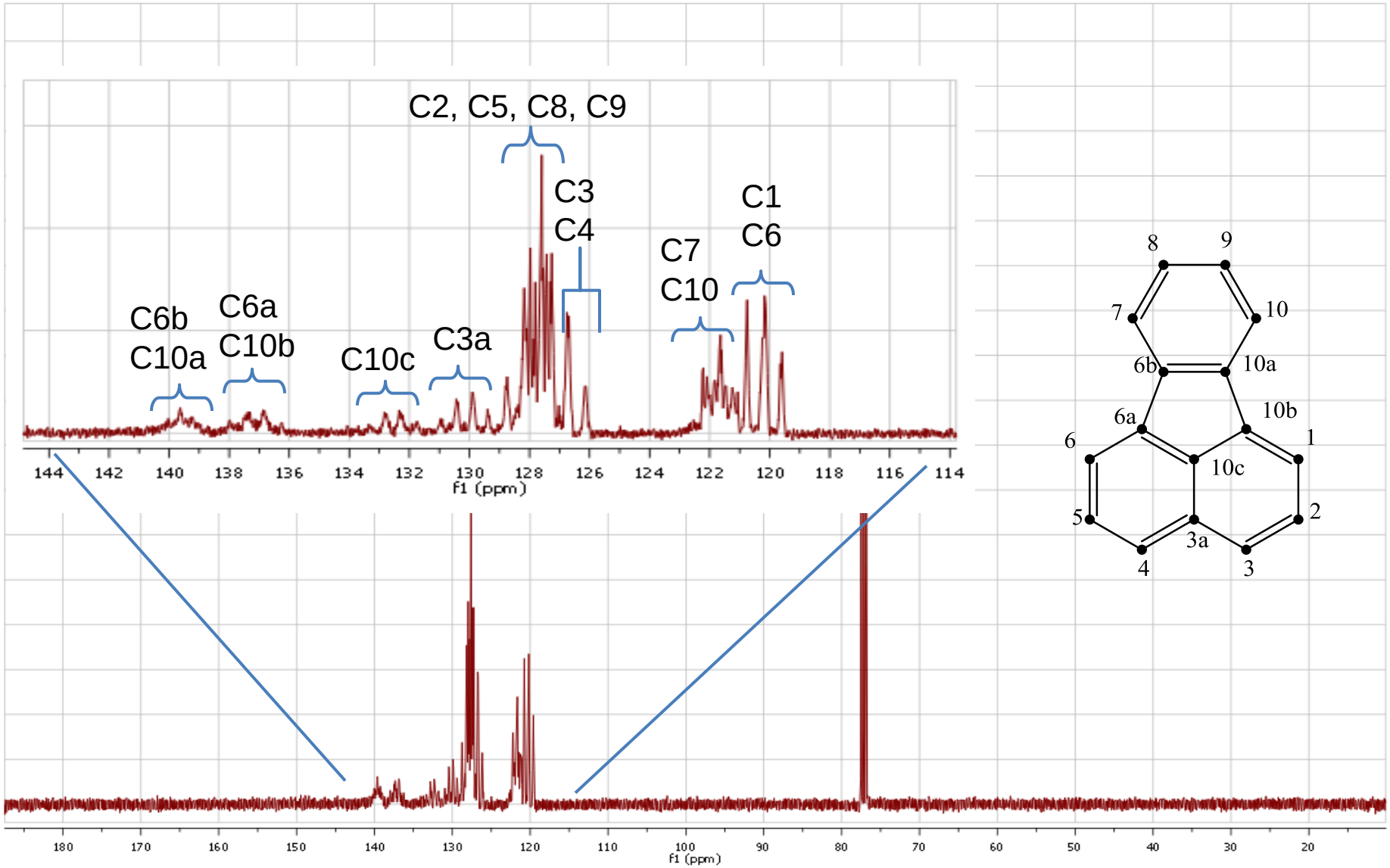


Figure S56. ^{13}C NMR (75 MHz, $CDCl_3$) of U-^{13}C-fluoranthene (**22**).