

Synthesis of Uniformly ¹³C-Labeled Polycyclic Aromatic Hydrocarbons

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Electronic Supplementary Information

Table of Contents

Page S2.	Experimental methods
Page S6.	Literature citations
Page S7.	Figure S1 ¹ H NMR spectrum of U- ¹³ C-4-phenylbutanoic acid
Page S8.	Figure S2 ¹³ C NMR spectrum of U- ¹³ C-4-phenylbutanoic acid
Page S9.	Figure S3 ¹ H NMR spectrum of U- ¹³ C-tetralone (1)
Page S10.	Figure S4 ¹³ C NMR spectrum of U- ¹³ C-tetralone (1)
Page S11.	Figure S5 ¹ H NMR spectrum of U- ¹³ C-tetralin (2)
Page S12.	Figure S6 ¹³ C NMR spectrum of U- ¹³ C-tetralin (2)
Page S13.	Figure S7 ¹ H NMR spectrum of U- ¹³ C-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid
Page S14.	Figure S8 ¹³ C NMR spectrum of NA-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid
Page S15.	Figure S9 ¹³ C NMR spectrum of U- ¹³ C-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid
Page S16.	Figure S10 ¹ H NMR spectrum of NA-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid
Page S17.	Figure S11 ¹³ C NMR spectrum of NA-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid
Page S18.	Figure S12 ¹ H NMR spectrum of U- ¹³ C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (3)
Page S19.	Figure S13 ¹³ C NMR spectrum of U- ¹³ C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (3)
Page S20.	Figure S14 ¹ H NMR spectrum of NA-3,4,5,6,7,8-hexahydroanthracen-1(2H)-one
Page S21.	Figure S15 ¹³ C NMR spectrum of NA-3,4,5,6,7,8-hexahydroanthracen-1(2H)-one
Page S22.	Figure S16 ¹ H NMR spectrum of U- ¹³ C-3,4,5,6,7,8-hexahydroanthracen-1(2H)-one (4)
Page S23.	Figure S17 ¹³ C NMR spectrum of U- ¹³ C-3,4,5,6,7,8-hexahydroanthracen-1(2H)-one (4)
Page S24.	Figure S18 ¹ H NMR spectrum of U- ¹³ C-1,2,3,4,5,6,7,8-octahydroanthracene
Page S25.	Figure S19 ¹³ C NMR spectrum of U- ¹³ C-1,2,3,4,5,6,7,8-octahydroanthracene
Page S26.	Figure S20 ¹ H NMR spectrum of U- ¹³ C-anthracene (6)
Page S27.	Figure S21 ¹³ C NMR spectrum of U- ¹³ C-anthracene (6)
Page S28.	Figure S22 ¹ H NMR spectrum of methyl NA-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate
Page S29.	Figure S23 ¹ H NMR spectrum of methyl U- ¹³ C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate
Page S30.	Figure S24 ¹³ C NMR spectrum of methyl U- ¹³ C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate
Page S31.	Figure S25 ¹ H NMR spectrum of methyl NA-4-(naphthalen-2-yl)butanoate
Page S32.	Figure S26 ¹³ C NMR spectrum of methyl NA-4-(naphthalen-2-yl)butanoate
Page S33.	Figure S27 ¹ H NMR spectrum of methyl U- ¹³ C-4-(naphthalen-2-yl)butanoate (10)
Page S34.	Figure S28 ¹³ C NMR spectrum of methyl U- ¹³ C-4-(naphthalen-2-yl)butanoate (10)
Page S35.	Figure S29 ¹ H NMR spectrum of NA-2,3-dihydrophenanthren-4(1H)-one
Page S36.	Figure S30 ¹³ C NMR spectrum of NA-2,3-dihydrophenanthren-4(1H)-one
Page S37.	Figure S31 ¹ H NMR spectrum of U- ¹³ C-2,3-dihydrophenanthren-4(1H)-one (8)
Page S38.	Figure S32 ¹³ C NMR spectrum of U- ¹³ C-2,3-dihydrophenanthren-4(1H)-one (8)
Page S39.	Figure S33 ¹ H NMR spectrum of NA-1,2,3,4-tetrahydrophenanthrene
Page S40.	Figure S34 ¹³ C NMR spectrum of NA-1,2,3,4-tetrahydrophenanthrene
Page S41.	Figure S35 ¹ H NMR spectrum of U- ¹³ C-1,2,3,4-tetrahydrophenanthrene
Page S42.	Figure S36 ¹³ C NMR spectrum of U- ¹³ C-1,2,3,4-tetrahydrophenanthrene
Page S43.	Figure S37 ¹ H NMR spectrum of U- ¹³ C-phenanthrene (11)
Page S44.	Figure S38 ¹³ C NMR spectrum of U- ¹³ C-phenanthrene (11)
Page S45.	Figure S39 ¹ H NMR spectrum of NA-ethyl 2-(phenanthren-4-yl)acetate
Page S46.	Figure S40 ¹ H NMR spectrum of ethyl U- ¹³ C-2-(phenanthren-4-yl)acetate (13)
Page S47.	Figure S41 ¹³ C NMR spectrum of ethyl U- ¹³ C-2-(phenanthren-4-yl)acetate (13)
Page S48.	Figure S42 ¹ H NMR spectrum of U- ¹³ C-pyrene (14)
Page S49.	Figure S43 ¹³ C NMR spectrum of U- ¹³ C-pyrene (14)
Page S50.	Figure S44 ¹ H NMR spectrum of U- ¹³ C-phthalic acid (15)
Page S51.	Figure S45 ¹ H NMR spectrum of U- ¹³ C-naphthalene (7)

- Page S52. Figure S46 ^{13}C NMR spectrum of U- ^{13}C -naphthalene (**7**)
Page S53. Figure S47 ^1H NMR spectrum of NA-benz[*a*]anthracene
Page S54. Figure S48 ^{13}C NMR spectrum of NA-benz[*a*]anthracene
Page S55. Figure S49 ^1H NMR spectrum of U- ^{13}C -benz[*a*]anthracene (**17**)
Page S56. Figure S50 ^{13}C NMR spectrum of U- ^{13}C -benz[*a*]anthracene (**17**)
Page S57. Figure S51 ^1H NMR spectrum of U- ^{13}C -4-phenyl-1,2-dihydronaphthalene(**21**)
Page S58. Figure S52 ^{13}C NMR spectrum of NA-4-phenyl-1,2-dihydronaphthalene
Page S59. Figure S53 ^{13}C NMR spectrum of U- ^{13}C -4-phenyl-1,2-dihydronaphthalene(**21**)
Page S60. Figure S54 ^1H NMR spectrum of U- ^{13}C -fluoranthene (**23**)
Page S61. Figure S55 ^{13}C NMR spectrum of NA- ^{13}C -fluoranthene
Page S62. Figure S56 ^{13}C NMR spectrum of U- ^{13}C -fluoranthene (**23**)

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 ^1H 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 -benzene (0.5 g 5.95 mmol) was followed by portion-wise addition of fresh AlCl_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 RotavaporTM 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 anhydrous 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 HCl 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 anhydrous MgSO_4 and the solvent evaporated to give a white solid (0.98 g, 95% yield): Mp 50 – 52 °C. ^1H NMR, Figure S1, (300 MHz) δ 7.18 (bd, J = 158.9 Hz, 5H, Ar), 2.67 (bd, J = 125.7 Hz, 2H, C_4H_2), 2.37 (t, J = 123.1 Hz, 2H, C_2H_2), 1.97 (p, J = 125.7 Hz, 2H, C_3H_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, C_4H_2), 33.5 (ddd, J = 55.5, 34.1, 3.0 Hz, C_2H_2), 26.6 (t, J = 34.6 Hz, C_3H_2). ^1H and ^{13}C chemical shifts are identical to those reported.¹² 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 anhydrous MgSO_4 and filtered. Evaporation of solvent yielded a yellowish liquid (0.835 g, 95 % yield). ^1H 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, C_4H_2), 2.64 (bd, J = 126.4 Hz, 2H, C_2H_2), 2.12 (bd, J = 132.7 Hz, 2H, C_3H_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, C_2H_2), 30.1 (dd, J = 42.4, 32.5

Hz, C4H₂), 23.71 (t, *J* = 32.6 Hz, C3H₂). ¹³C and ¹H NMR assignments are based on published NMR spectra of NA-tetralone.³

U-¹³C-Tetralin (2). Wolff-Kishner reduction of U-¹³C-tetralone (830 mg, 5.32 mmol) by the procedure described above for 4-oxo-4-phenylbutanoic acid afforded a brownish residue which was further purified by chromatography (SiO₂, 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 to evaporation. ¹H NMR, Figure S5, (300 MHz) δ 7.07 (bd, *J* = 150.4 Hz, 4H, Ar), 2.77 (bd, *J* = 126.1 Hz, 4H, C1H₂, C4H₂), 1.80 (bd, *J* = 123.3 Hz, 4H, C2H₂, C3H₂). ¹³C 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, C1H₂, C4H₂), 23.13 – 24.16 (m, C2H₂, C3H₂). ¹H NMR shifts corresponded to those of NA-tetralin.⁴ The ¹³C shifts are based on the ¹³C NMR spectrum of NA-tetralin.⁵

U-¹³C-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid. U-¹³C-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-¹³C-tetralin (600 mg 4.2 mmol) with U-¹³C-succinic acid (0.439 g, 4.2 mmol) afforded a yellowish solid (0.991 g 90 % yield): Mp 114 – 116 °C. ¹H 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, C3H₂), 2.79 (bd, *J* = 131.2 Hz, 6H, C2H₂, C5'H₂, C8'H₂), 1.81 ((bd, *J* = 123.7 Hz, 4H, C6'H₂, C7'H₂). ¹³C 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, C3H₂), 29.3 – 30.5 (m, C5'H₂, C8'H₂), 8.4 (dd, *J* = 55.0, 39.5 Hz, C2H₂), 22.7 – 23.8 (m, C6'H₂, C7'H₂).

For NA-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid: ¹H 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, C3H₂), 2.79 (m, 6H, C2H₂, C5'H₂, C8'H₂), 1.81 (br, 4H, C6'H₂, C7'H₂). ¹³C NMR Figure S8, (100 MHz) δ 198.0(C4), 179.2(C1), 143.7(C4'a), 137.7(C2'), 134.1(C8'a), 129.6(C1'), 129.2(C3'), 125.3(C4'), 33.2(C3H₂), 29.8 & 29.7(C5H₂, C8H₂), 28.4(C2H₂), 23.2 & 23.0(C6H₂, C7H₂).

NA-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid. Procedure was the same as described above for the reduction of U-¹³C-4-oxo-4-phenylbutanoic acid. ¹H 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'H₂, C8'H₂), 2.59 (t, 2H, *J* = 7.5 Hz, C4H₂), 2.36 (2H, t, *J* = 7.5 Hz, C2H₂), 1.92 (2H, q, *J* = 7.3 Hz, C3H₂), 1.76 (br 4H, C6'H₂, C7'H₂). Chemical shifts are in agreement with the published spectrum⁶; signal assignments are tentative. ¹³C NMR, Figure S11, (100 MHz) δ 180.3 (C1), 138.4(C2'), 137.3(C8'a), 135.0(C4'a), 129.3 (C1', C4'), 125.8(C3'), 34.8(C4H₂), 33.7(C2H₂), 29.6 & 29.2(C5H₂, C8H₂), 26.5(C3H₂), 23.5 & 23.4(C6H₂, C7H₂). ¹H NMR shifts are from the published spectrum.⁶ ¹³C shift assignments are based on 6-butanyltetralin.⁷

U-¹³C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (3). Compound **3** was prepared in 92% yield by reduction of U-¹³C-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid. ¹H 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'H₂, C8'H₂), 2.60 (bd, *J* = 131.2 Hz, 2H, C4H₂), 2.37 (bd, *J* = 127.2 Hz, 2H, C2H₂), 1.95 (bd, *J* = 128.7 Hz, 2H, C2H₂), 1.82 (bd, *J* = 125.2 Hz, 4H, C6'H₂, C7'H₂). ¹³C NMR, Figure S13, (75 MHz) δ 179.4 (dd, *J* = 55.2, 3.1 Hz, C1), 134.1 – 139.7 (m, C2', C4'a, C8'a), 129.5 (t, *J* = 57.2 Hz, C1'), 125.9 (t, C4'), 35.0 (ddq, *J* = 41.2, 34, 3.3 Hz, C4H₂), 33.7 (ddd, *J* = 55.5, 34.2, 3.6 Hz, C2H₂), 29.0 – 30.2 (m, C5'H₂, C8'H₂) 26.7 (t, *J* = 34.5 Hz, C3H₂), 23.1 – 24.2 (m, C6'H₂, C7'H₂).

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 ¹H NMR (Figure S14) and ¹³C NMR (Figure S15) spectra are identical to the published spectrum.⁸ For compound **4**, ¹H NMR, Figure S16; ¹³C NMR, Figure S17.

U-¹³C-1,2,3,4,5,6,7,8-octahydroanthracene. U-¹³C-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. ¹H NMR, Figure S18, (300 MHz) δ 6.78 (bd, *J* = 149.3 Hz, 2H, H5, H10), 2.70 (bd, *J* = 125.9 Hz, 8H, C1H₂, C4H₂, C5H₂, C8H₂), 1.76 (bd, *J* = 121.9 Hz, 8H, C2H₂, C3H₂, C6H₂, C7H₂). ¹³C 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, C1H₂, C4H₂, C5H₂, C8H₂), 23.6 – 24.1 (m, C2H₂, C3H₂, C6H₂, C7H₂). For NA-1,2,3,4,5,6,7,8-octahydroanthracene, the ¹H NMR, Figure S18 inset, is identical to the published spectrum.⁹

Methyl U-¹³C-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 NaHCO₃, water and brine, then dried over anhydrous MgSO₄. 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 %). ¹H 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, CH₃), 2.73 (bd, *J* = 127.4 Hz, 4H, C5'H₂, C8'H₂), 2.56 (bd, *J* = 126.2 Hz, 2H, C4H₂), 1.98 (bd, *J* = 119.0 Hz, 2H, C2H₂), 1.90

(bd, $J = 125.2$ Hz, 2H, C3H₂), 1.76 (bd, $J = 123.9$ Hz, 4H, C6'H₂, C7'H₂). ¹³C 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, C4H₂), 33.6 (ddd, $J = 57.5, 34.6, 3.4$ Hz, C2H₂), 28.3 – 30.3 (m, C5'H₂, C8'H₂) 26.7 (t, $J = 32.5$ Hz, C3H₂), 22.9 – 23.9 (m, C6'H₂, C7'H₂).

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-¹³C compound. ¹H 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, CH₃), 2.73 (br, 4H, C5'H₂, C8'H₂), 2.57 (t, $J = 7.5$ Hz, C4H₂), 1.98 (t, $J = 7.5$ Hz, C2H₂), 1.90 (p, $J = 7.5$ Hz, C3H₂), 1.72 – 1.80 (m, 4H, C6'H₂, C7'H₂).

NA-Methyl 4-(naphthalen-2-yl)butanoate: NA-Methyl 4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate was dehydrogenated as described in the Experimental Section for the synthesis of **11**. ¹H 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, CH₃), 2.82 (t, $J = 7.7$ Hz, 2H, C4H₂), 2.37 (t, $J = 7.7$ Hz, 2H, C2H₂), 2.08 (p, $J = 7.7$ Hz, 2H, C3H₂). ¹³C 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(CH₃), 35.5(C4H₂), 33.6(C2H₂), 26.6(C3H₂). ¹H NMR and ¹³C NMR assignments are based on 2-methyl naphthalene.¹⁰

Methyl U-¹³C- 4-(naphthalen-2-yl)butanoate (10). Compound **10** was prepared from **3** as described above for the synthesis of NA-methyl 4-(naphthalen-2-yl)butanoate. Compound **10** was isolated as an oil in 85% yield. ¹H 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, CH₃), 2.82 (bd, $J = 127.8$ Hz, 2H, C4H₂), 2.37 (bd, $J = 126.8$ Hz, 2H, C2H₂), 2.08 (bd, $J = 128.6$ Hz, 2H, C3H₂). ¹³C 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, C4H₂), 33.4 (ddd, $J = 57.3, 34.5, 3.8$ Hz, C2H₂), 26.4 (t, $J = 34.6$ Hz, C3H₂).

NA-2,3-dihydrophenanthren-4(1H)-one. Procedure was the same as that described in the Experimental Section for the synthesis of **4**. ¹H 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, C1H₂), 2.79 (t, $J = 7.2$ Hz, 2H, C3H₂), 2.20 (q, $J = 6.4$ Hz, 2H, C2H₂). ¹³C NMR, Figure S30, ¹H NMR and ¹³C NMR shift assignments are from published data.¹¹

U-¹³C-2,3-dihydrophenanthren-4(1H)-one (8). Compound **8** was prepared in 98% yield as described above for **4**. ¹H 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, C1H₂), 2.79 (bd, $J = 127.7$ Hz, 2H, C3H₂), 2.20 (bd, $J = 130.1$ Hz, 2H, C2H₂). ¹³C 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, C3H₂), 28.8 (dd, $J = 40.9, 35.6$ Hz, C1H₂), 20.2 (t, $J = 32.4$ Hz, C2H₂).

NA-1,2,3,4-tetrahydrophenanthrene. Procedure was the same as that described in the Experimental Section for the synthesis of tetralin. ¹H 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, C4H₂), 2.91 (t, $J = 6.2$ Hz, 2H, C1H₂), 1.95 (m, 2H, C3H₂), 1.87 (m, 2H, C2H₂). ¹³C 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 ¹H and ¹³C NMR spectra are identical to published spectra with assignments.¹¹

U-¹³C-1,2,3,4-tetrahydrophenanthrene. U-¹³C-1,2,3,4-Tetrahydrophenanthrene was prepared from **8** by the procedure described above for the reduction of U-¹³C-tetralone to U-¹³C-tetralin and isolated as an oil in 88% yield. ¹H 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, C4H₂), 2.91 (bd, $J = 128.2$ Hz, 2H, C1H₂), 1.95 (bd, $J = 126.7$ Hz, 2H, C3H₂), 1.87 (bd, $J = 124.7$ Hz, 2H, C2H₂). ¹³C NMR, Figure S36, (100 MHz) δ 122.7 – 134.5 (m, Ar), 30.2 (m, C1H₂), 25.4 – 26.2 (m, C4H₂), 22.7 – 23.8 (m, C2H₂, C3H₂).

NA-Phenanthrene. For procedure see the Experimental Section. ¹H 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 ¹H and ¹³C NMR spectra of NA-phenanthrene are identical to published spectra with assignments.¹² For U-¹³C-phenanthrene, ¹H NMR, Figure S37; ¹³C 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-¹³C-2-(phenanthren-4-yl)acetate. ¹H 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, ArCH₂), 4.28 (q, $J = 7.2$ Hz, 2H, OCH₂), 1.26 (t, $J = 7.1$ Hz, 3H, CH₃). Assignments are based on 4-Me-phenanthrene.¹³ For **13**, ¹H NMR, Figure S40; ¹³C NMR, Figure S41.

U-¹³C-Pyrene (14). The procedure is described in the Experimental Section. ¹H NMR, Figure S42; ¹³C NMR, Figure S43. The ¹H (Figure S42 inset) and ¹³C NMR spectra of NA-Pyrene are identical to published spectra.¹²

U-¹³C-Phthalic acid (15). The procedure is described in the Experimental Section. ¹H NMR, Figures S44. For NA-Phthalic acid: ¹H 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); ¹³C NMR (75 MHz, *d*₆-DMSO) δ 169.3, 133.4, 131.4, 128.9.

U-¹³C-Naphthalene (7). The procedure is described in the Experimental Section. ¹H NMR, Figure S45; ¹³C NMR, Figure S46. For NA-Naphthalene: ¹H 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 ¹H and ¹³C 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-¹³C-benz[*a*]anthracene (**17**). ¹H 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). ¹³C 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 ¹H and ¹³C NMR shift assignments are from published spectra.^{14,15} For compound **17**, ¹H NMR, Figure S49; ¹³C NMR, Figure S50.

NA-4-bromo-1,2-dihydronaphthalene. Triphenyl phosphite (1.3 mL, 5 mmol) was dissolved in anhydrous 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 was purified by chromatography (SiO₂, hexane) to yield 4-bromo-1,2-dihydronaphthalene (0.78 g, 98 %). ¹H 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, C1H₂), 2.34 (AB, *J* = 7.6 Hz, 2H, C2H₂). ¹³C 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 ¹H NMR and ¹³C NMR spectra are identical to the published spectra.¹⁶

NA-1-Bromonaphthalene (18). 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 (Al₂O₃, benzene) to yield (**19**, 0.344 g, 72%). ¹H 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). ¹³C 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 ¹H 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 °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. The combined organic extracts were washed with brine and dried over anhydrous MgSO₄. Evaporation of the solvent yielded NA-naphthalen-1-ylboronic acid as a solid (245 mg, 72%). ¹H NMR (300 MHz) δ 8.58 (s, 1H), 7.86–7.88 (m, 3H), 7.44–7.48 (m, 3H). The ¹H NMR spectrum is identical to the published spectrum.¹⁸

U-¹³C-4-phenyl-1,2-dihydronaphthalene (21). U-¹³C-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 Na₂CO₃, water and brine, dried over anhydrous Na₂SO₄. After evaporation of solvent, the residue was purified by chromatography (SiO₂, hexane) to give **21**. (266 mg, 90 % yield). ¹H 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, C1H₂), 2.42 (bd, *J* = 126.7 Hz, 2H, C2H₂). ¹³C NMR, Figure 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, C1H₂), 23.9 (t, *J* = 37.6 Hz, C2H₂). For NA-4-phenyl-1,2-dihydronaphthalene: ¹H NMR Figure S51 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, C1H₂), 2.35–2.41 (m, 2H, C2H₂). ¹³C 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. ¹H and ¹³C NMR shift assignments are from published spectra.^{19, 20}

U-¹³C-2-Iodo-1-phenyl-naphthalene (22). To **21** (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 Na₂S₂O₃ and dried over anhydrous MgSO₄. 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 Al₂O₃, hexane/ethyl acetate, 25:1) to give **22** (346 mg, 83% yield), which was used directly in the next step. ¹H and ¹³C NMR spectra are identical to published spectra.²¹

U-¹³C-Fluoranthene (23). The procedure is described in the Experimental Section. ¹H NMR and ¹³C NMR spectra, Figures S54, S56. For NA-fluoranthene: ¹H NMR, S54 inset, (300 MHz) δ 7.91 – 7.97 (m, 4H, H1, H6, H7H, H10H), 7.86 (d, 2H, H3, H4), 7.65 (t, *J* = 8 Hz, 2H, H2, H5), 7.38 – 7.41 (m, 2H, H8, H9). ¹³C NMR, Figure S55, δ 139.7, 137.2, 130.2, 128.2, 127.8, 126.9, 121.7, 120.3. The ¹H and ¹³C NMR shift assignments are from published spectra.^{12, 22}

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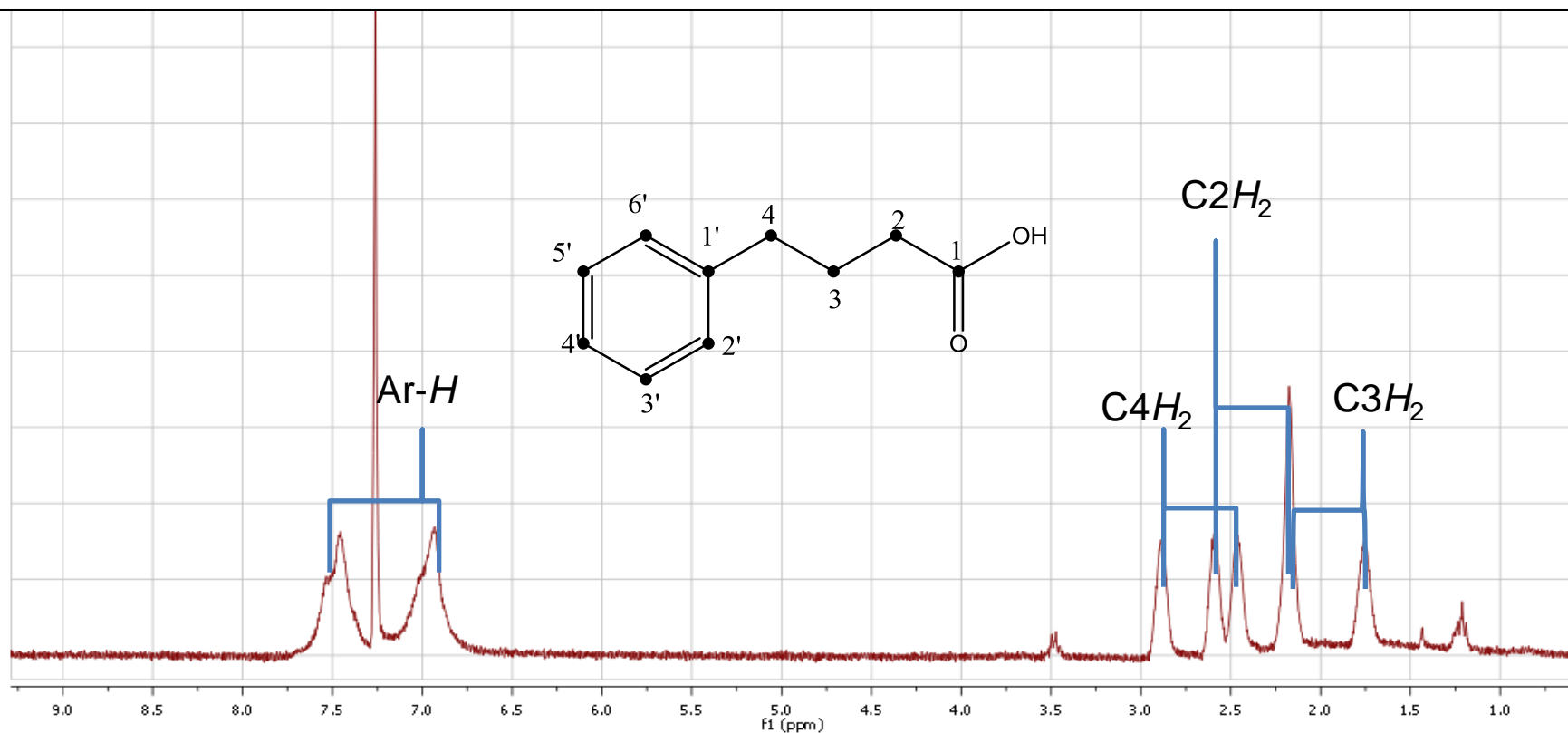
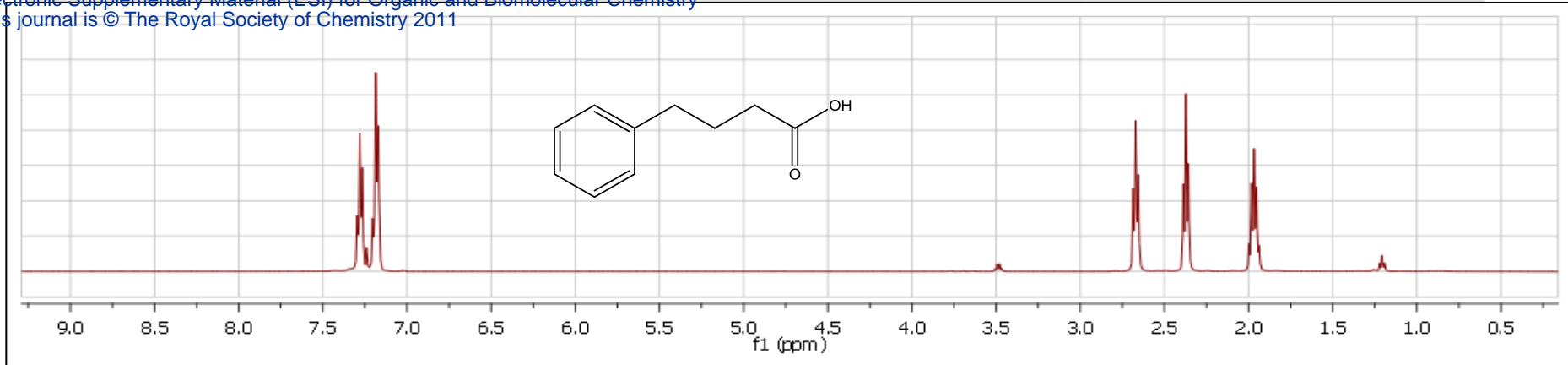


Figure S1. ¹H NMR (300 MHz, CDCl₃) of U-¹³C-4-phenylbutanoic acid. (Inset: ¹H NMR NA-4-phenylbutanoic acid. Chemical shifts identical to report: Auger P.; Malaiyandi, M.; Wightman, R. H. *J. Labelled Compounds Radiopharm.* **1993**, 33, 263.)

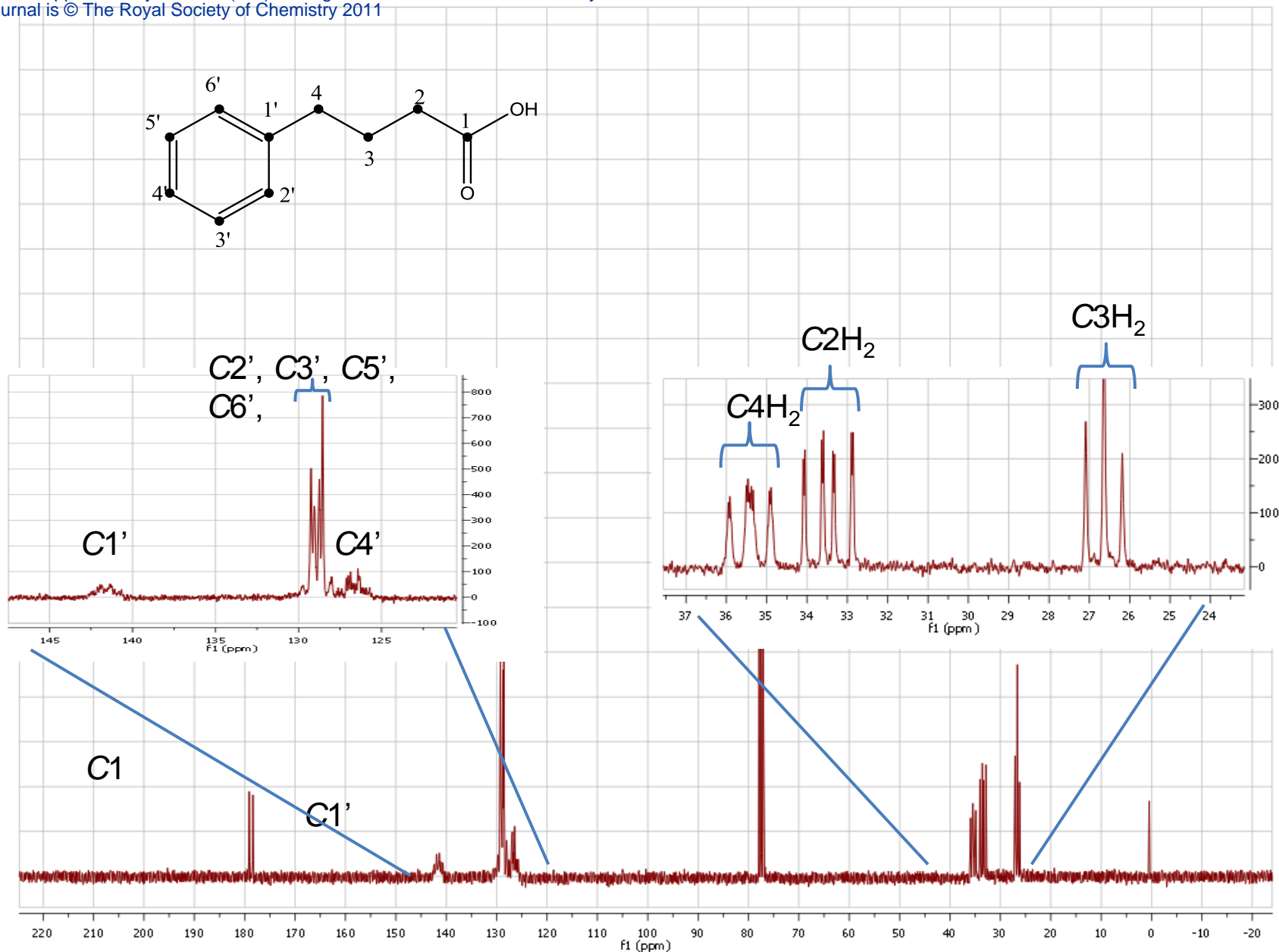


Figure S2. ¹³C NMR (75 MHz, CDCl₃) of U-¹³C-4-phenylbutanoic acid. (For ¹³C NMR assignments of NA-4-phenylbutanoic acid see: Hill, E. A.; Guenther, H. E. *Org. Magn. Reson.* **1981**, *16*, 177.)

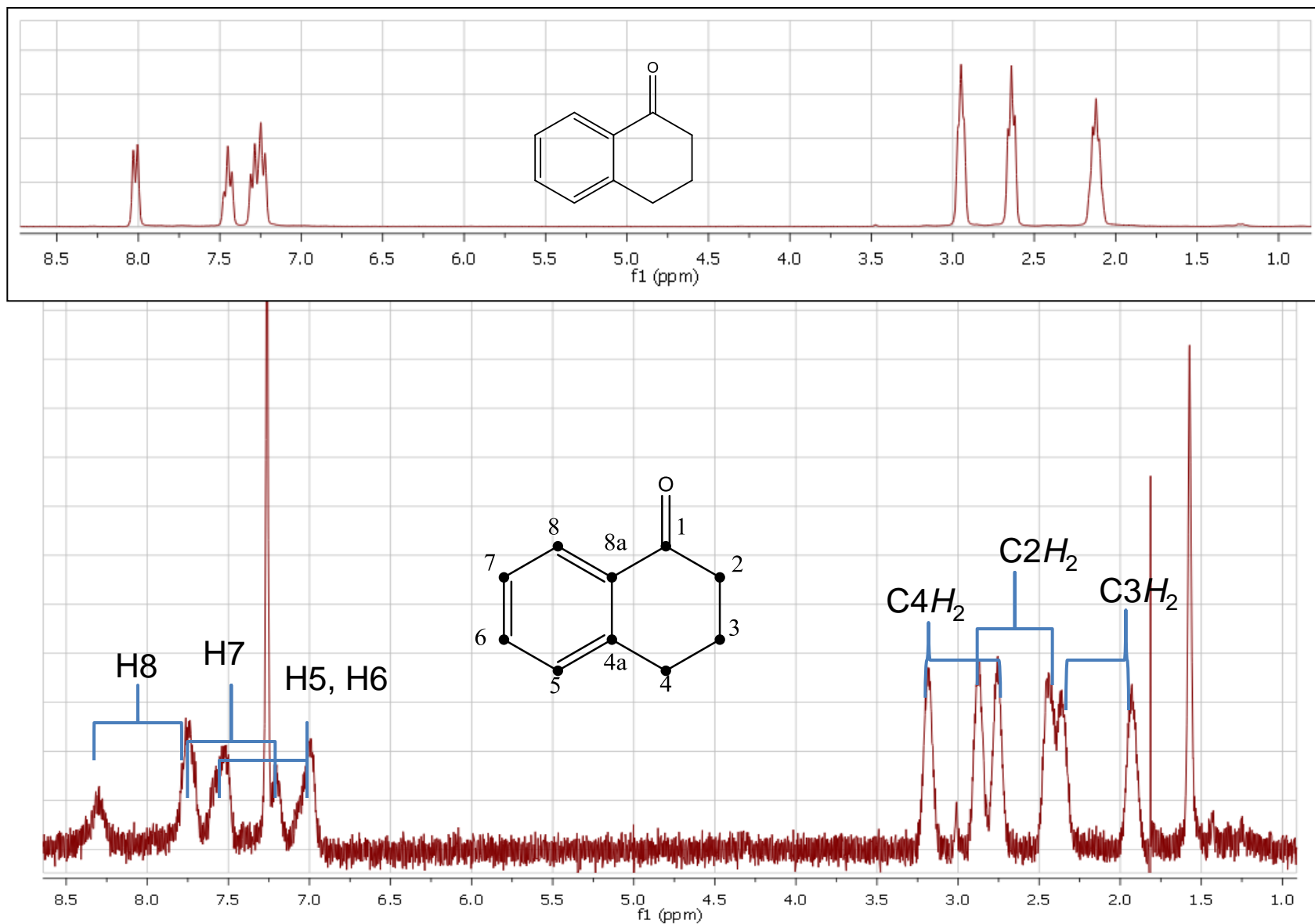


Figure S3. ¹H NMR (300 MHz, CDCl₃) of U-¹³C-tetralone (1). (Inset: ¹H NMR of NA-tetralone. Chemical shifts identical to report with assignments: Agrawal, P. K.; Thakur, R.S.; Frahm, A. W.; Schneider, M. *Magn. Reson. Chem.* **1990**, *28*, 931.)

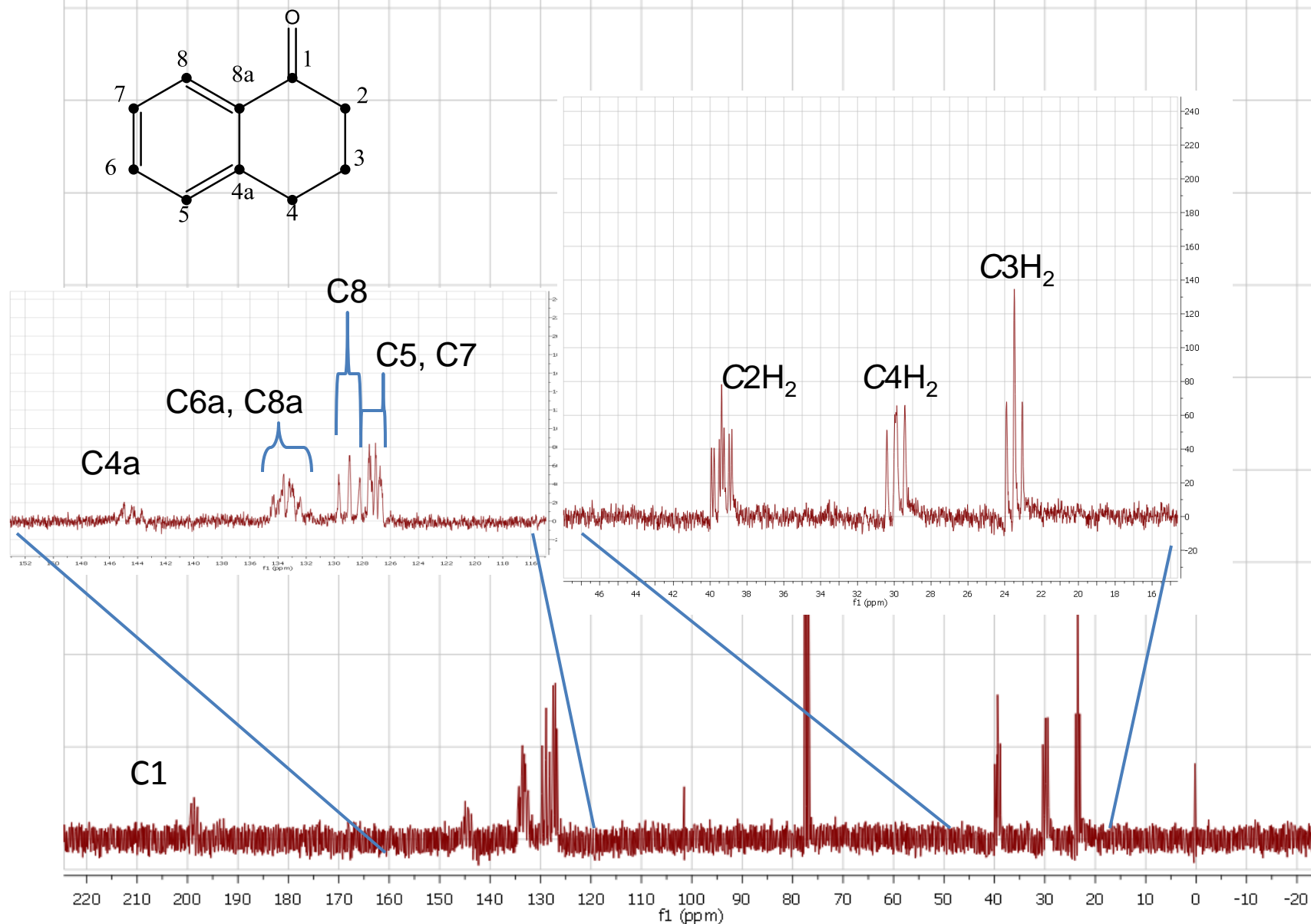


Figure S4. ^{13}C NMR (75 MHz, CDCl_3) of U- ^{13}C -tetralone (1). (For ^{13}C NMR assignments of NA-tetralone see: Agrawal, P. K.; Thakur, R.S.; Frahm, A. W.; Schneider, M. *Magn. Reson. Chem.* **1990**, *28*, 931.)

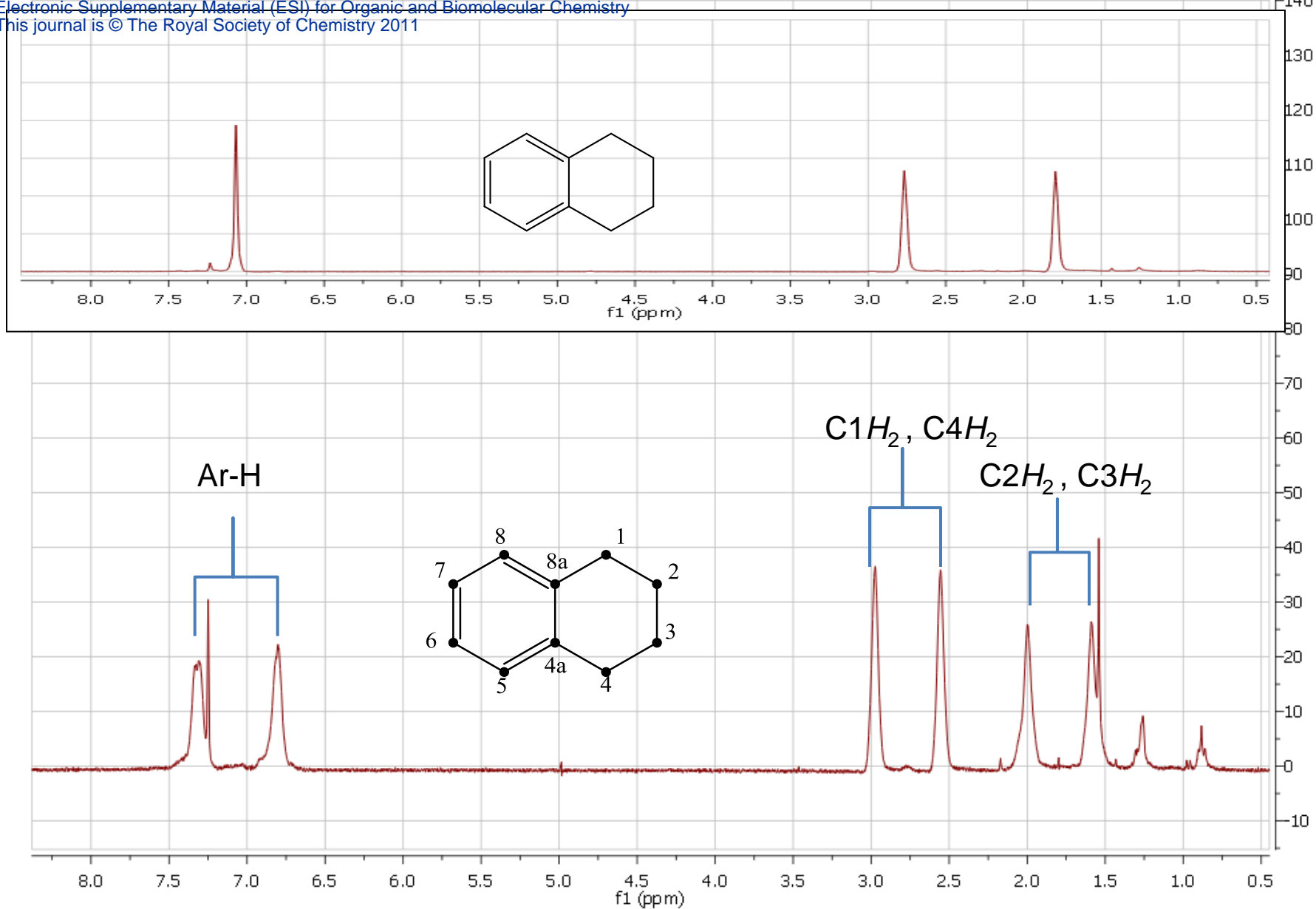


Figure S5. ¹H NMR (300 MHz, CDCl₃) of U-¹³C-tetralin (**2**). (Inset: ¹H NMR of NA-tetralin. For ¹H NMR of 1-¹³C₁-tetralin see: Pickering, R. E.; Bymaster, D. L.; Eisenbraun, E. J. *J. Labelled Compd. Radiopharm.* **1985**, *22*, 1149.)

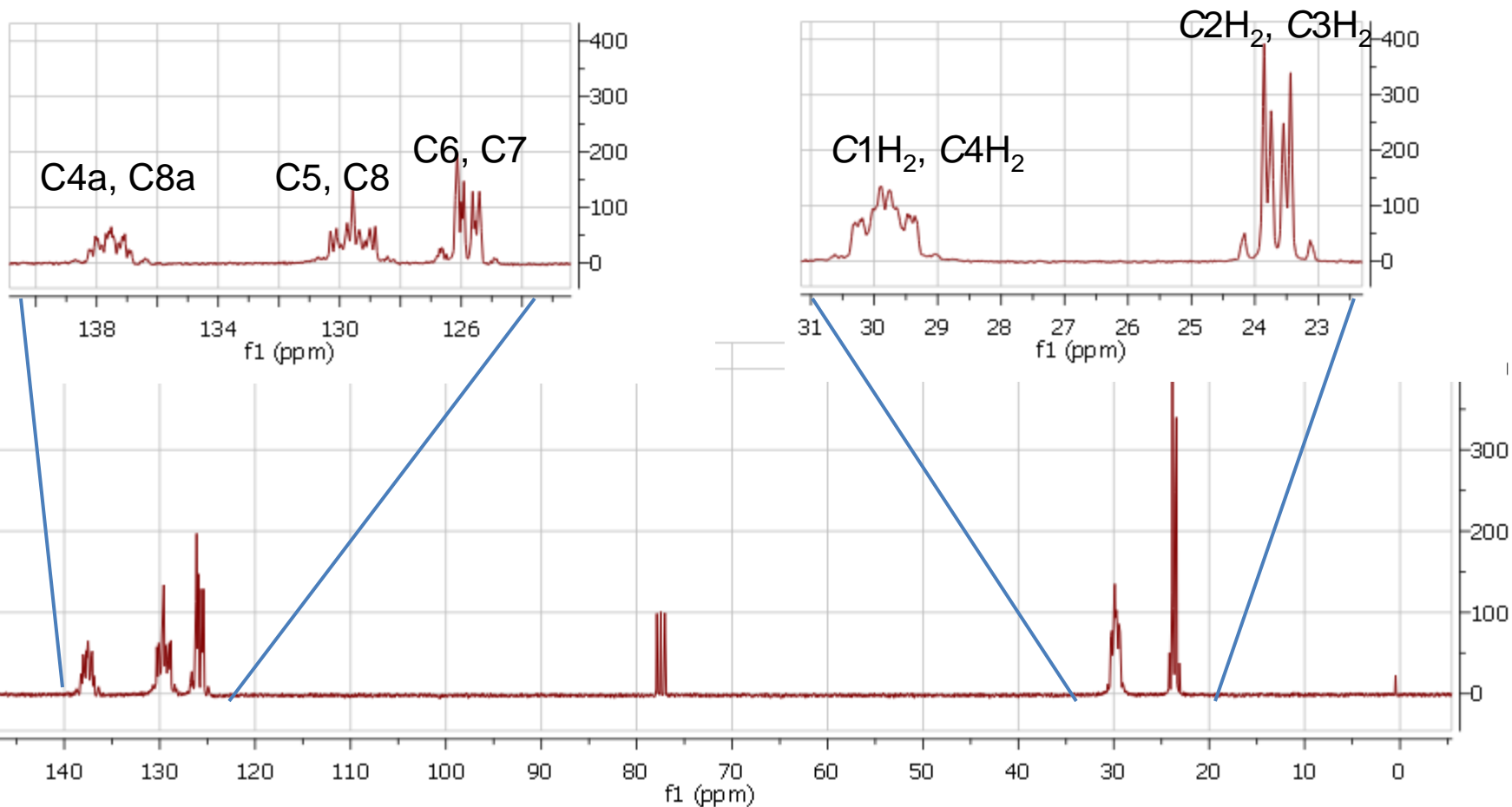
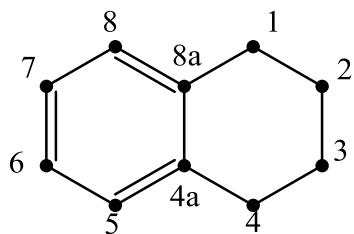


Figure S6. ^{13}C NMR (75 MHz, CDCl_3) of U- ^{13}C -tetralin (**2**). (For ^{13}C NMR assignments of NA-tetralin see: Lawens, T.; Schmit-Quih, F.; Nicole, D. *Mag. Res. Chem.* **1995**, 33, 523.)

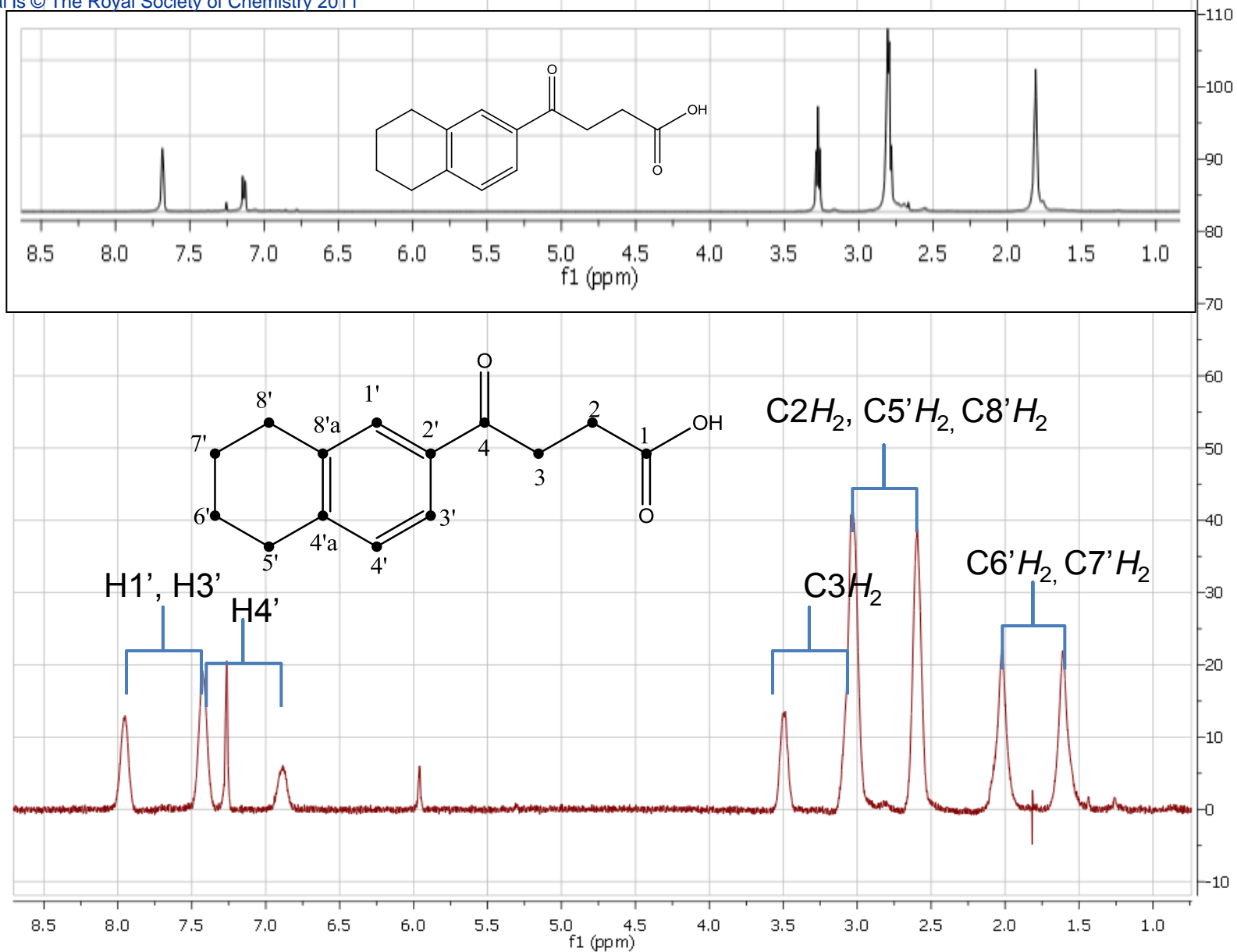


Figure S7. ¹H NMR (300 MHz, CDCl₃) of U-¹³C-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid. (Inset: ¹H NMR of NA-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid)

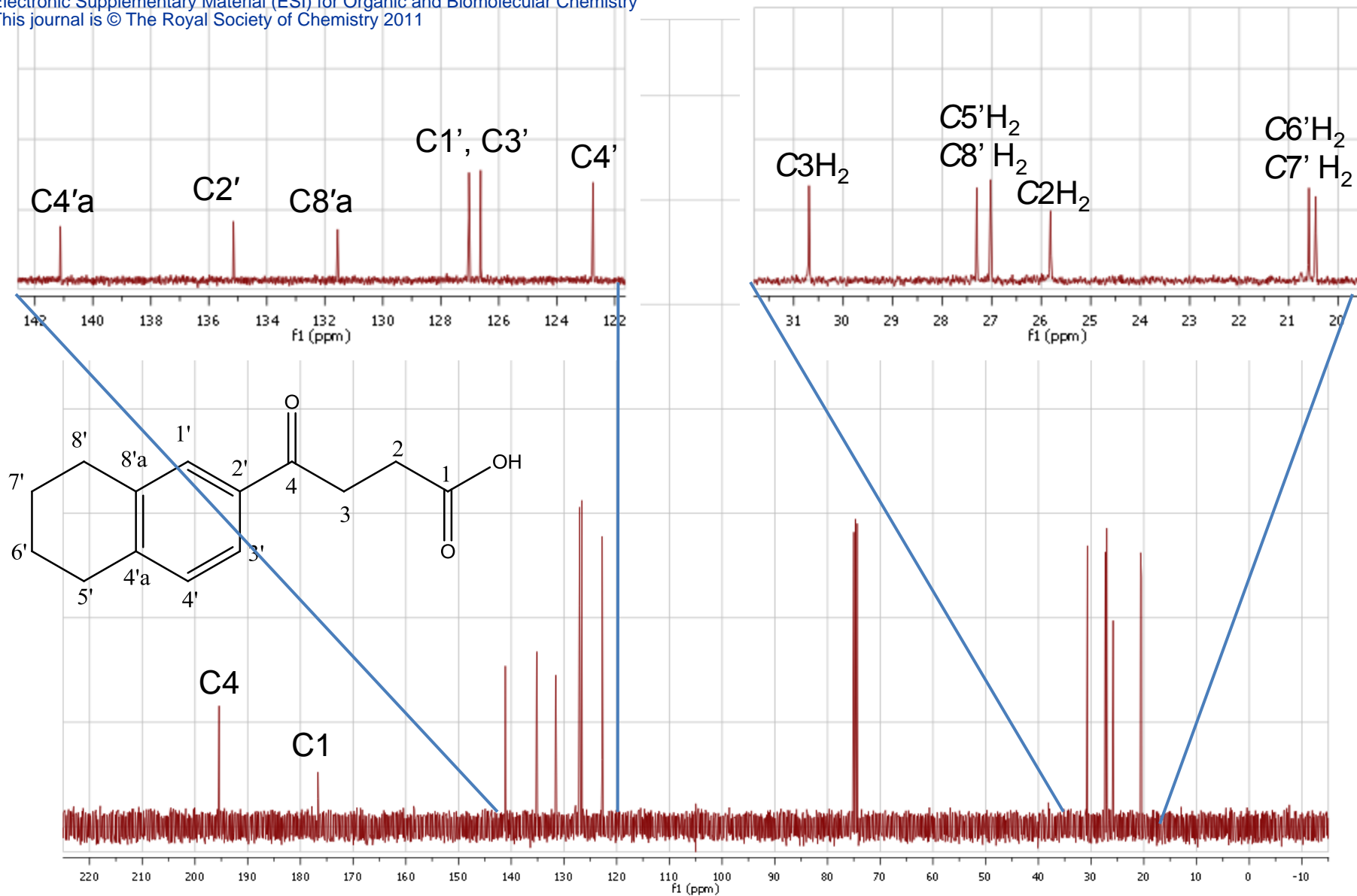


Figure S8. ^{13}C NMR (100 MHz, CDCl_3) of NA-4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid.

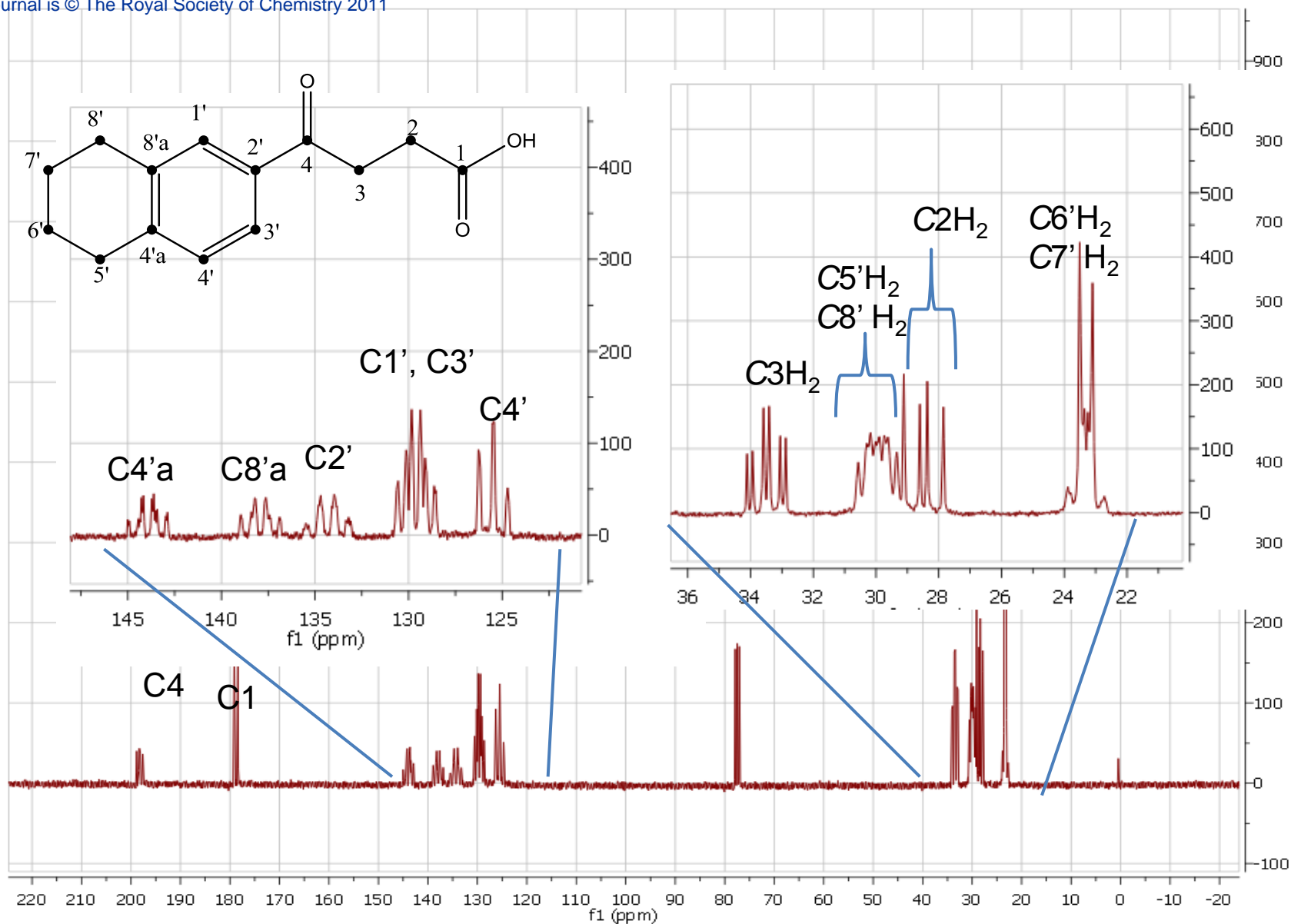


Figure S9. ^{13}C NMR (75 MHz, CDCl_3) of $\text{U-}^{13}\text{C}$ -4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid.

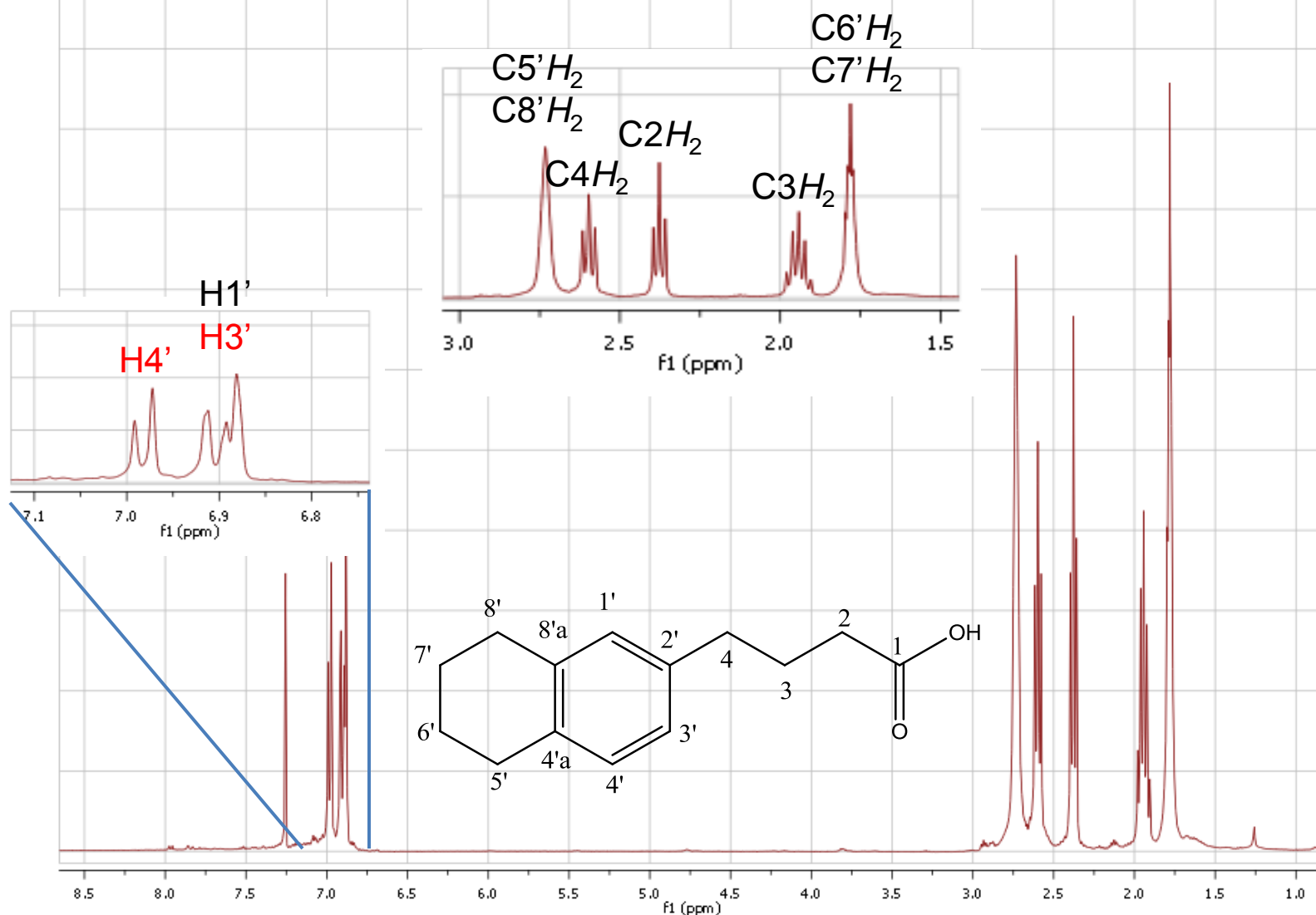


Figure S10. ^1H NMR (400 MHz, CDCl_3) of NA-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (**3**). (Chemical shifts identical to report: Murphy, P. V.; Hubbard, R. E.; Manallack, D. T.; Wills, R. E.; Montanaby, J. G.; Taylor, R. J. K. *Bioorg. Med. Chem.* **1998**, *6*, 2421.)

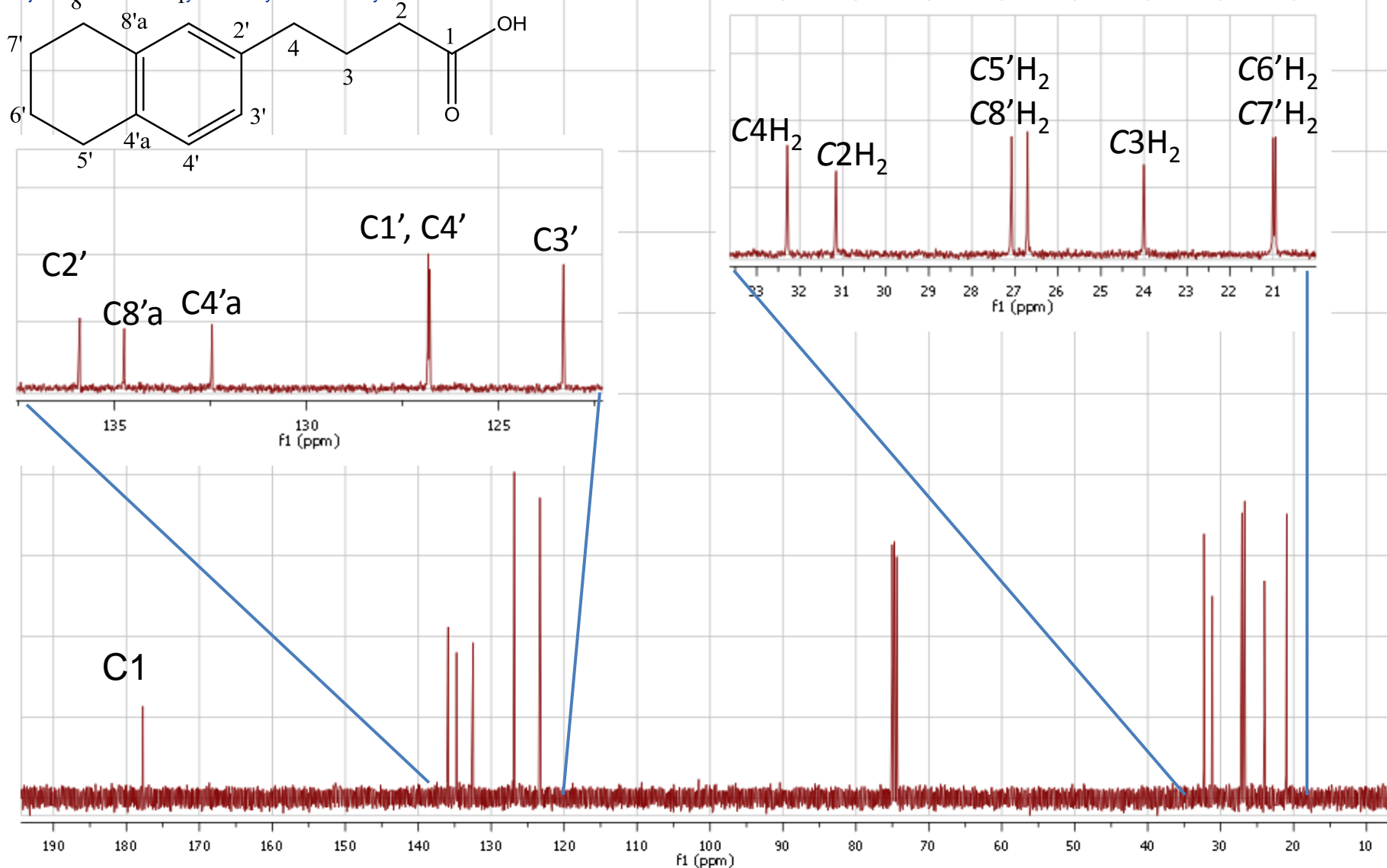


Figure S11. ^{13}C NMR (100 MHz, CDCl_3) of NA-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (**3**). (Chemical shifts identical to report: Murphy, P. V.; Hubbard, R. E.; Manallack, D. T.; Wills, R. E.; Montanaby, J. G.; Taylor, R. J. K. *Bioorg. Med. Chem.* **1998**, *6*, 2421. Assignments based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

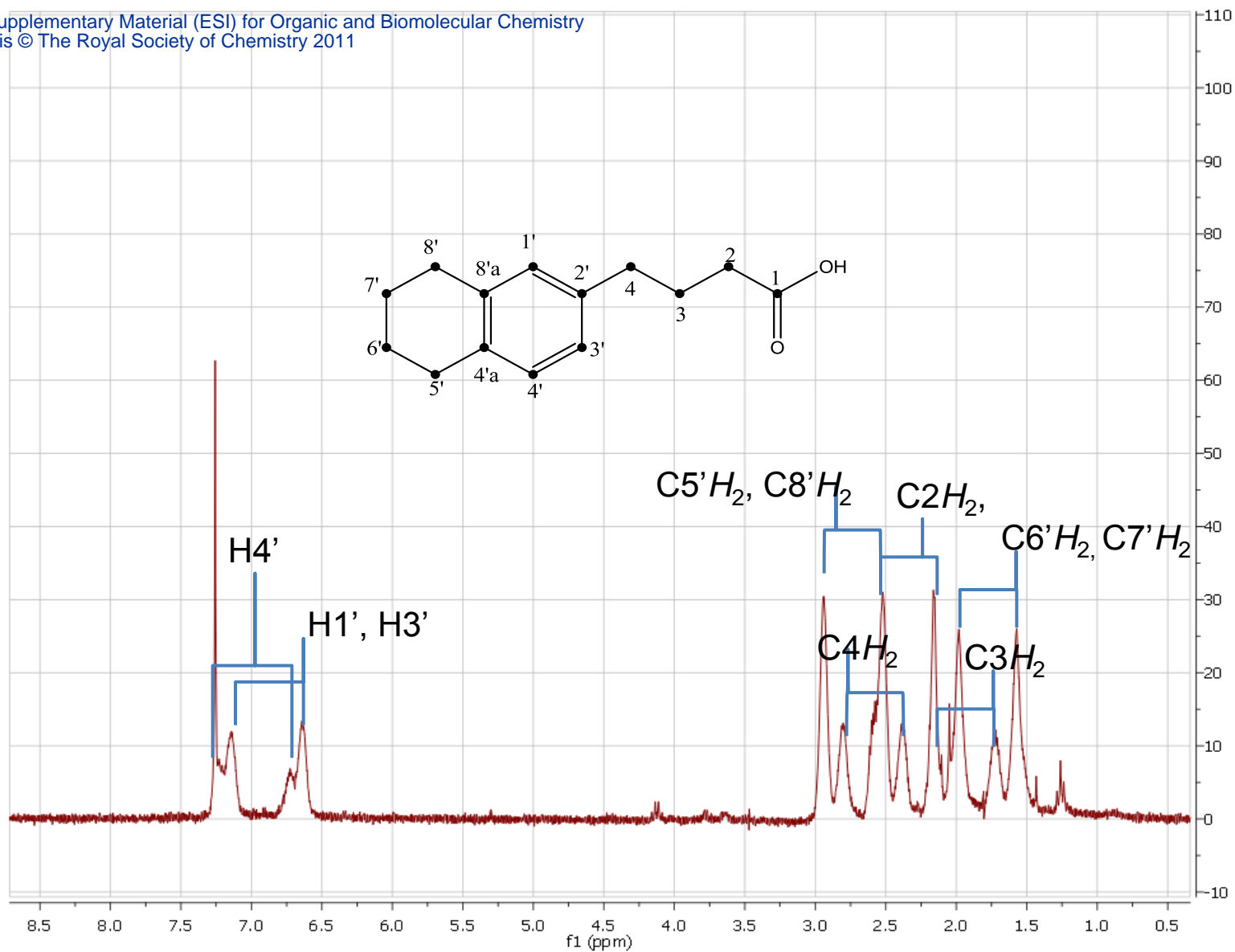


Figure S12. ¹H NMR (300 MHz, CDCl₃) of U-¹³C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (3).

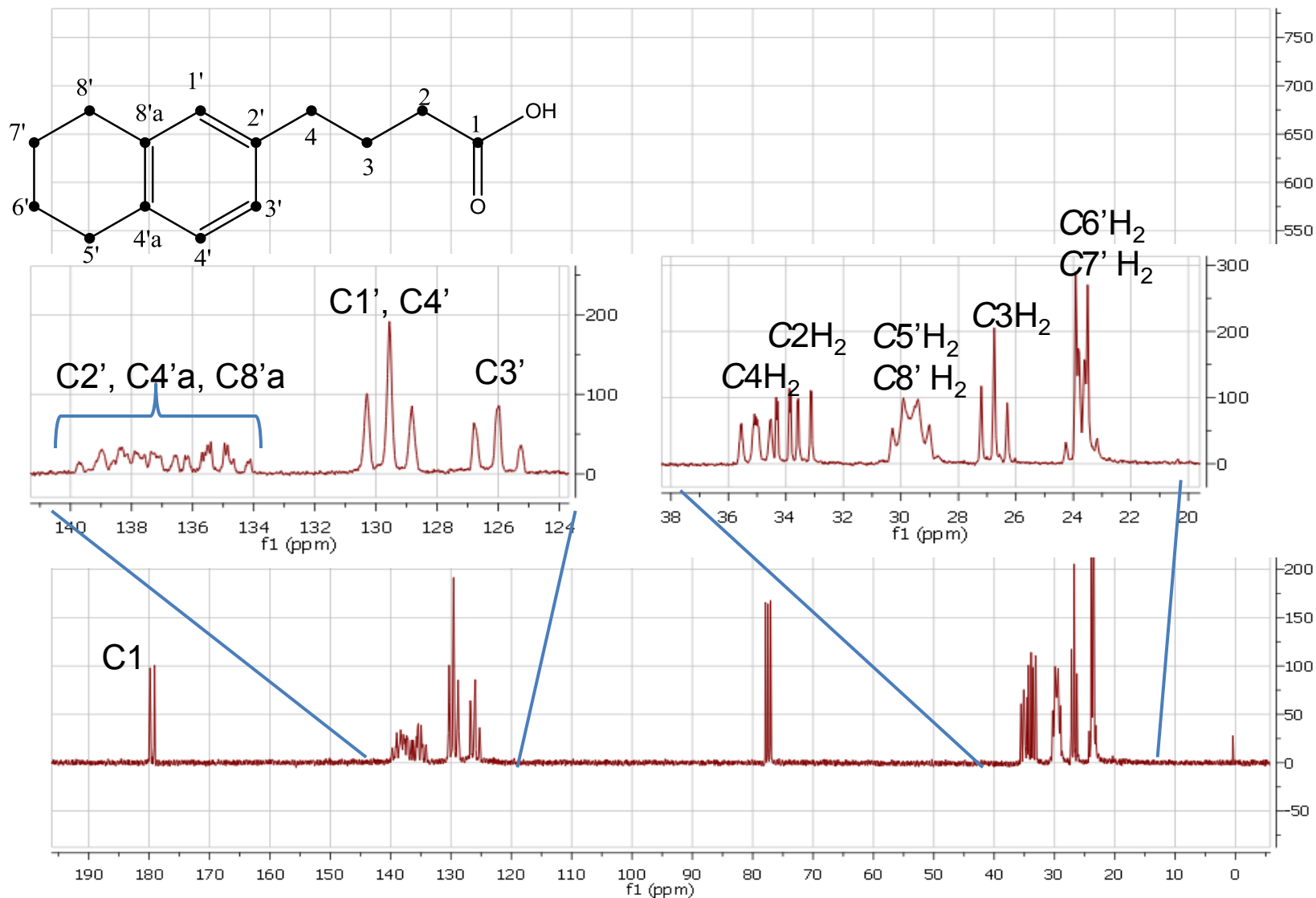


Figure S13. ¹³C NMR (75 MHz, CDCl₃) of U-¹³C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (**3**).

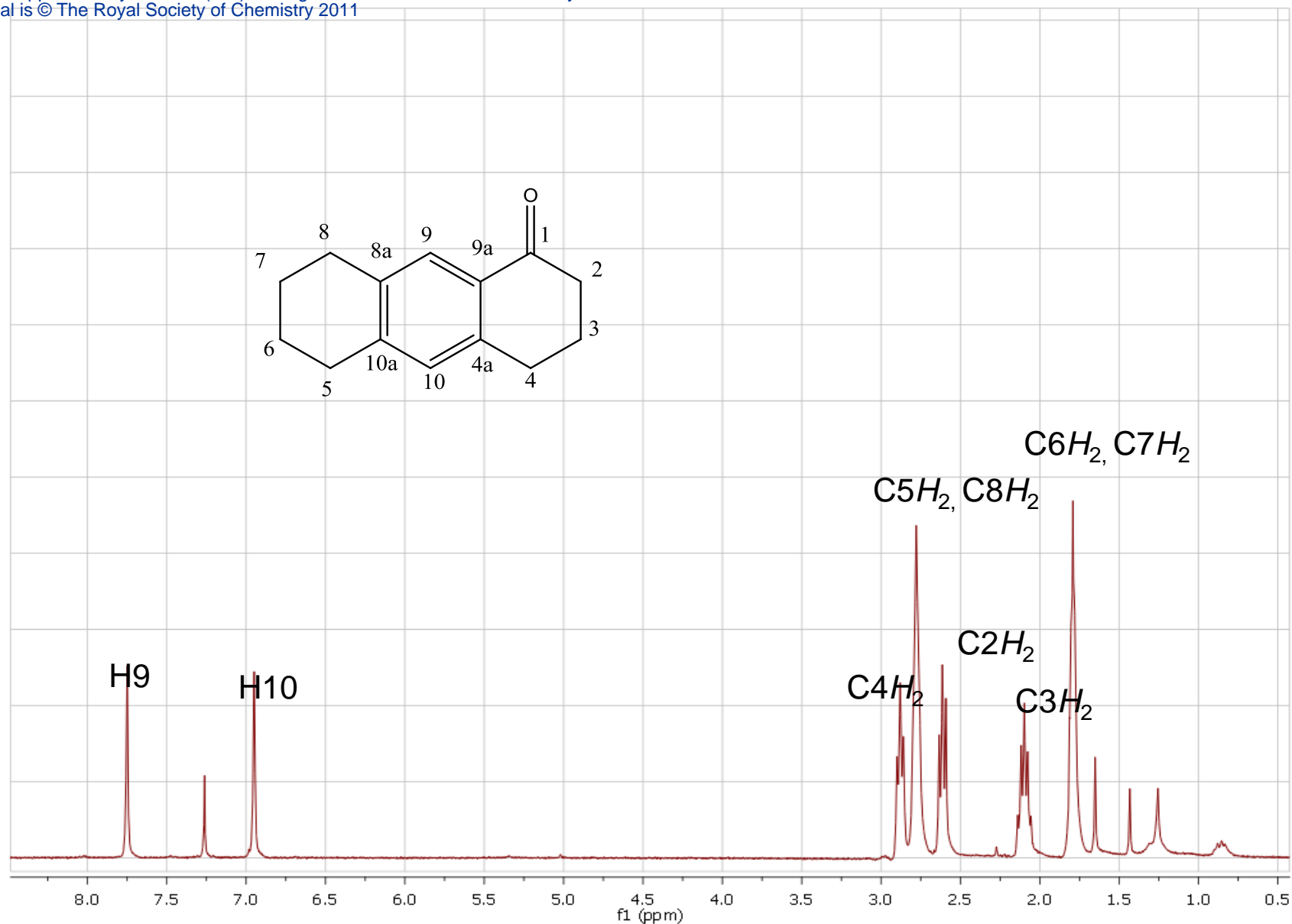


Figure S14. ¹H NMR (300 MHz, CDCl₃) of NA-3,4,5,6,7,8-hexahydroanthracen-1(2H)-one. (Chemical shifts identical to report: Boykin, D. W. *J. Org. Chem.* **1990**, *55*, 425.)

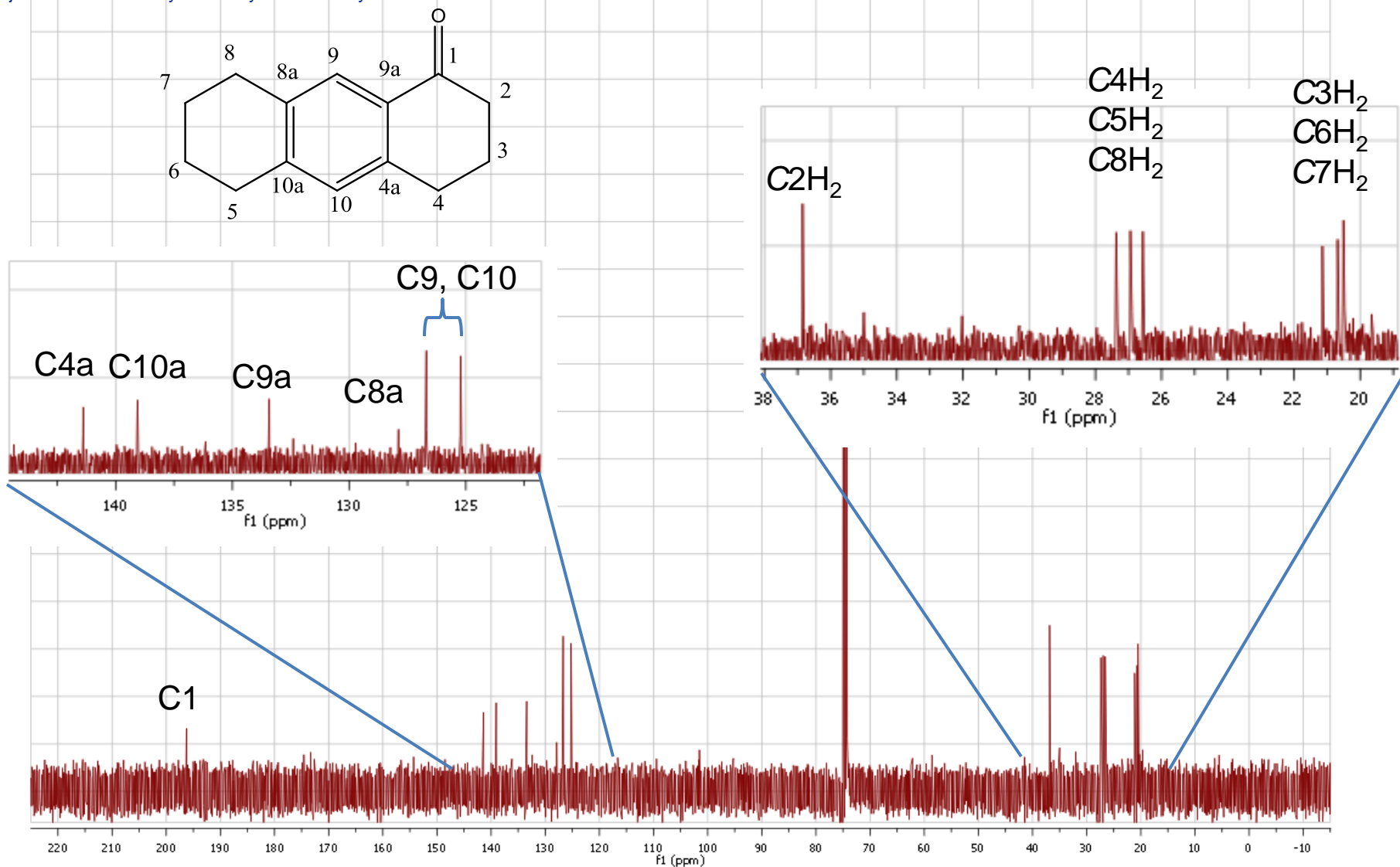


Figure S15. ^{13}C NMR (100 MHz, CDCl_3) of NA-3,4,5,6,7,8-hexahydroanthracen-1(2H)-one. (Chemical shifts identical to report: Boykin, D. W. *J. Org. Chem.* **1990**, *55*, 425.)

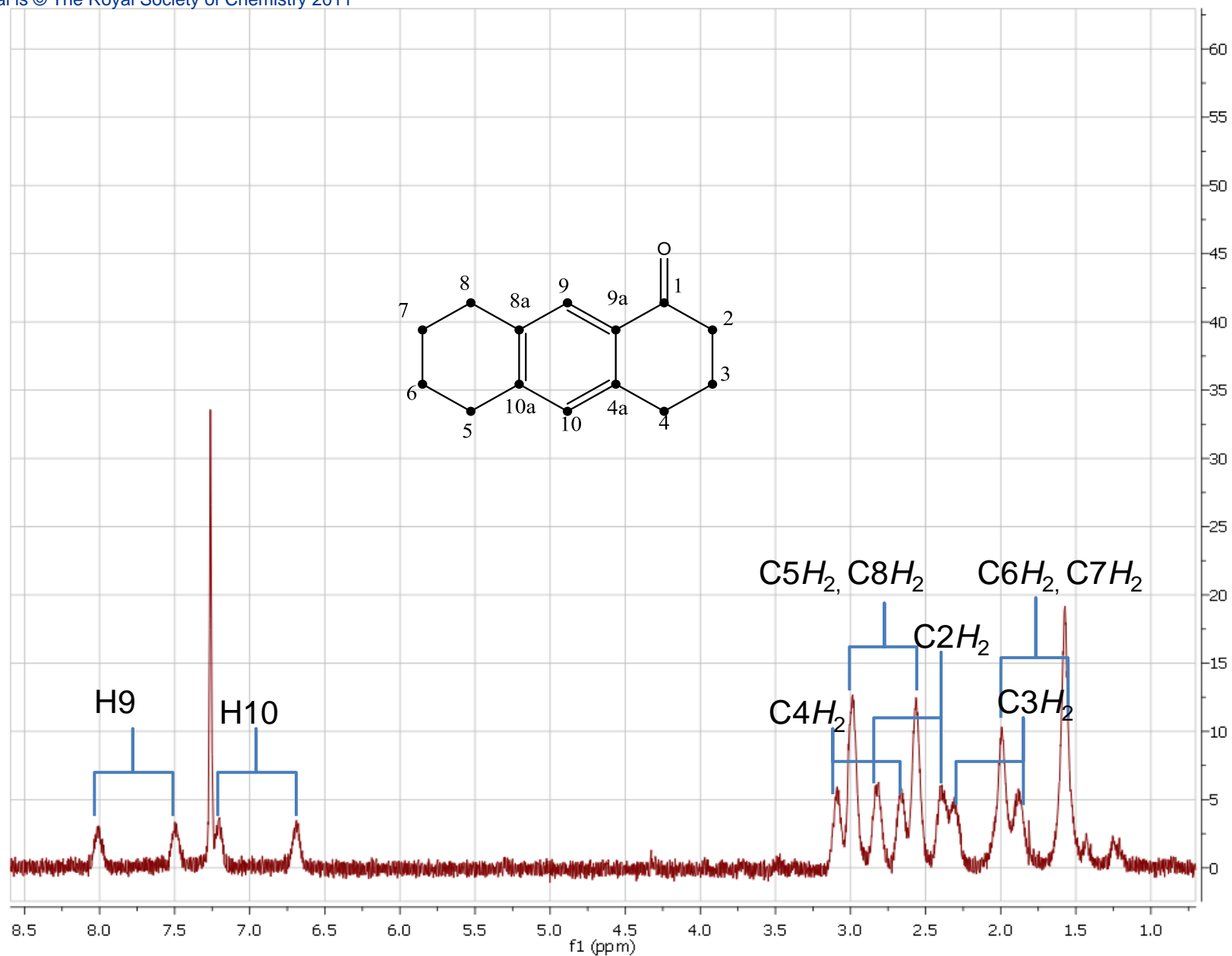


Figure S16. ^1H NMR (300 MHz, CDCl_3) of $\text{U-}^{13}\text{C}$ -3,4,5,6,7,8-hexahydroanthracen-1(2H)-one (**4**).

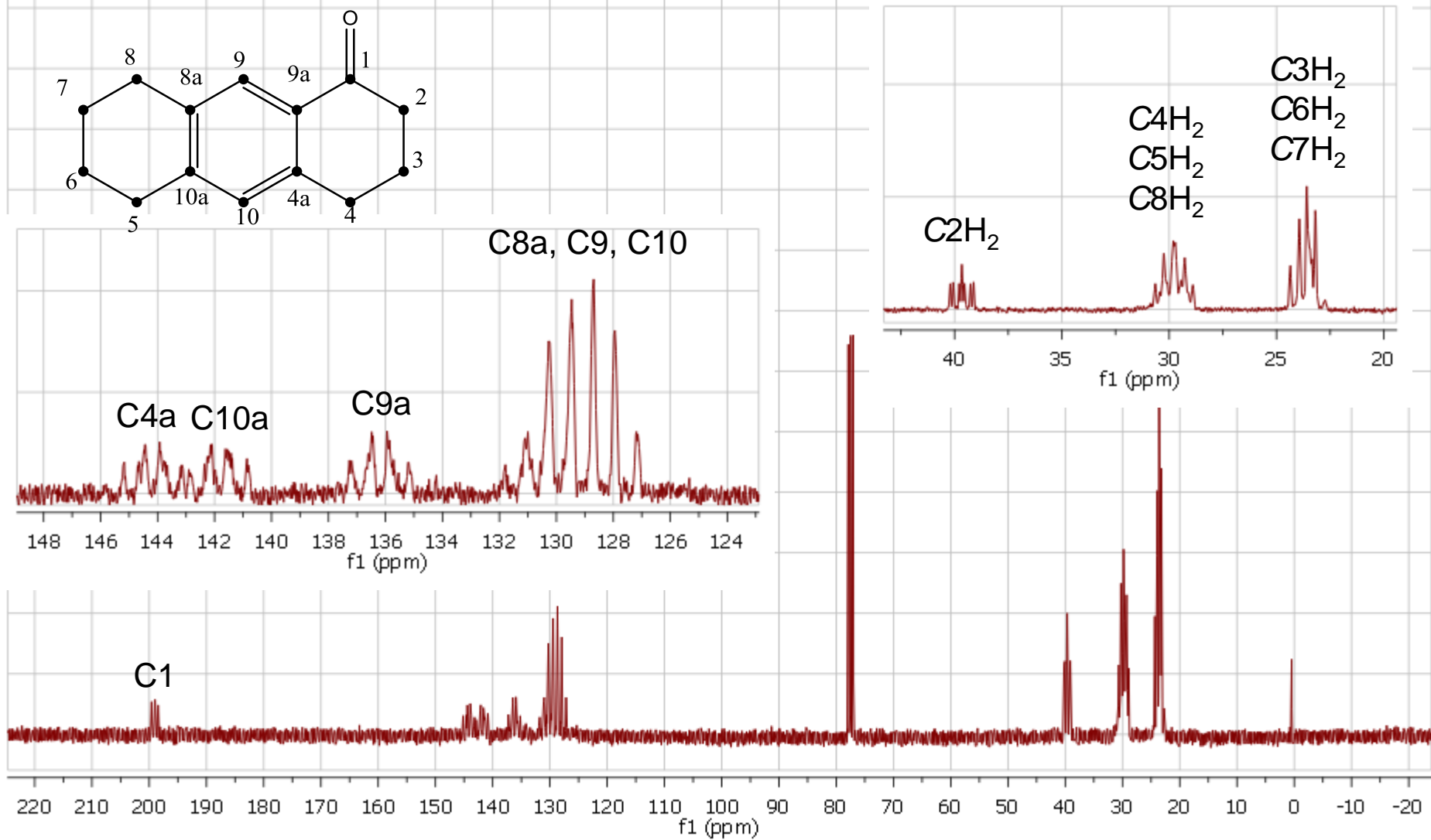


Figure S17. ¹³C NMR (75 MHz, CDCl₃) of U-¹³C-3,4,5,6,7,8-hexahydroanthracen-1(2H)-one (4).

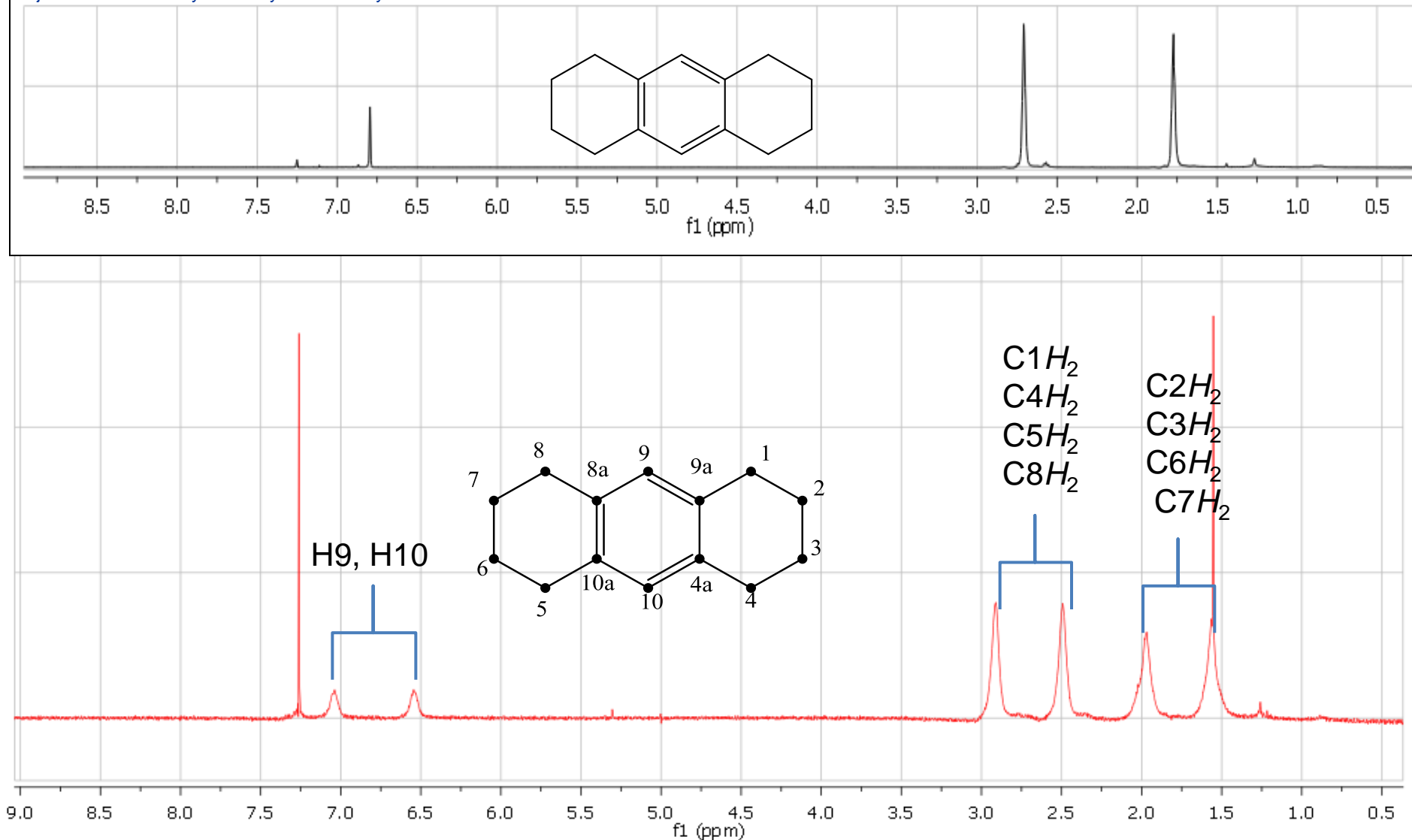


Figure S18. ¹H NMR (300 MHz, CDCl₃) of U-¹³C-1,2,3,4,5,6,7,8-octahydroanthracene: (Inset: ¹H NMR of NA-1,2,3,4,5,6,7,8-octahydroanthracene. Chemical shifts identical to report with assignments: Thummel, R. P.; Nutakul, W. *J. Org. Chem.*, **1978**, 43, 3170.)

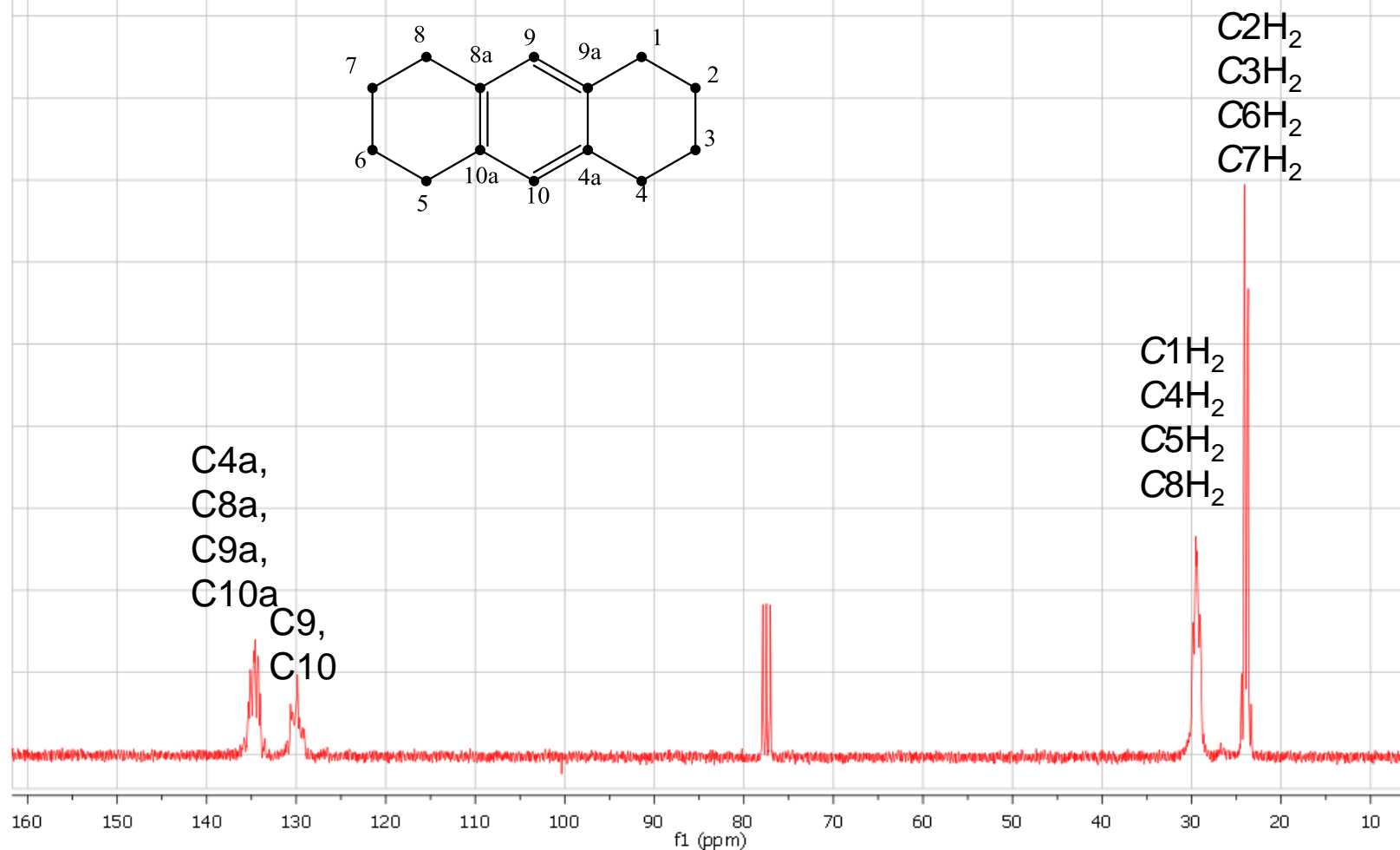


Figure S19. ^{13}C NMR (75 MHz, CDCl_3) of U- ^{13}C -1,2,3,4,5,6,7,8-octahydroanthracene. (For ^{13}C NMR assignments of NA-1,2,3,4,5,6,7,8-octahydroanthracene see: Thummel, R. P.; Nutakul, W. *J. Org. Chem.*, **1978**, 43, 3170.)

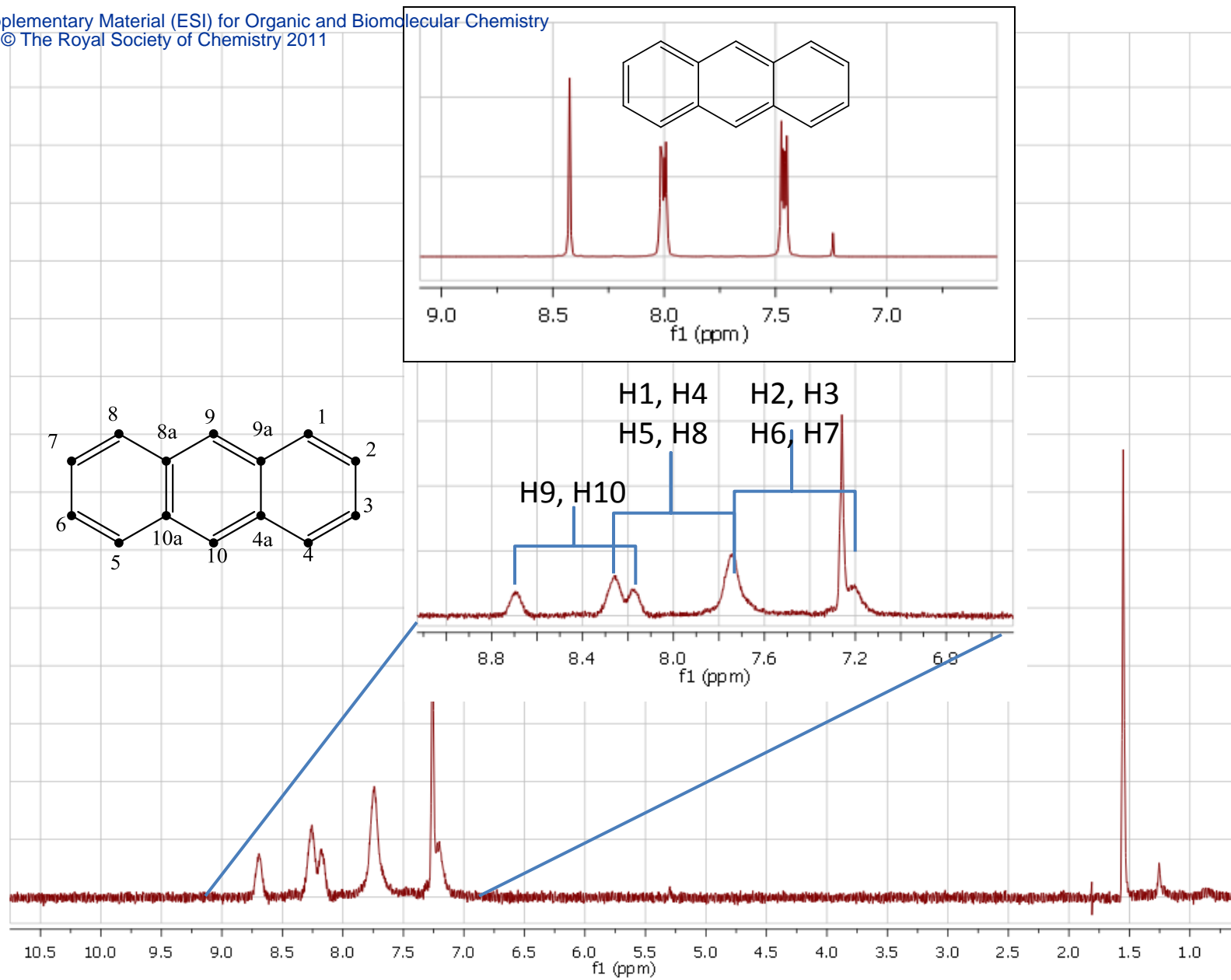


Figure S20. ¹H NMR (300 MHz, CDCl₃) of U-¹³C-anthracene (**6**). (Inset: ¹H NMR of NA-anthracene. Chemical shifts identical report with assignments: Gobert, F.; Combrisson, S.; Platzer, N.; Ricard M. *Org. Magn. Reson.* **1976**, *8*, 293.)

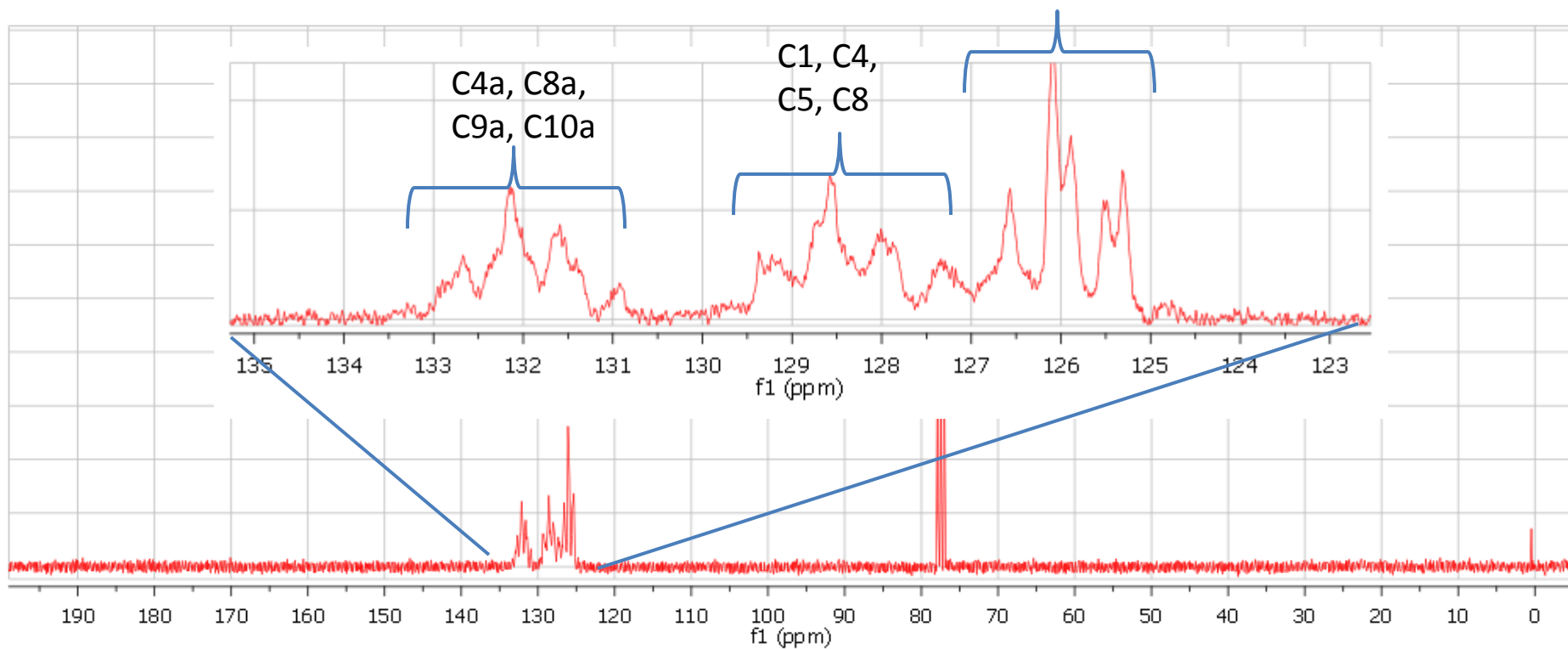
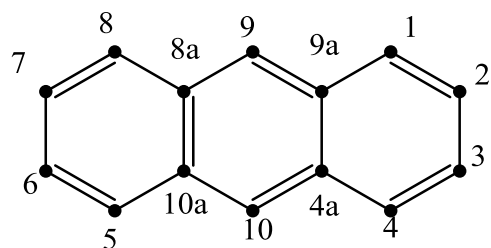


Figure S21. ^{13}C NMR (100 MHz, CDCl_3) of U- ^{13}C -anthracene (6). (For ^{13}C NMR assignments of NA-anthracene see: Gobert, F.; Combrisson, S.; Platzer, N.; Ricard M. *Org. Magn. Reson.* **1976**, 8, 293.)

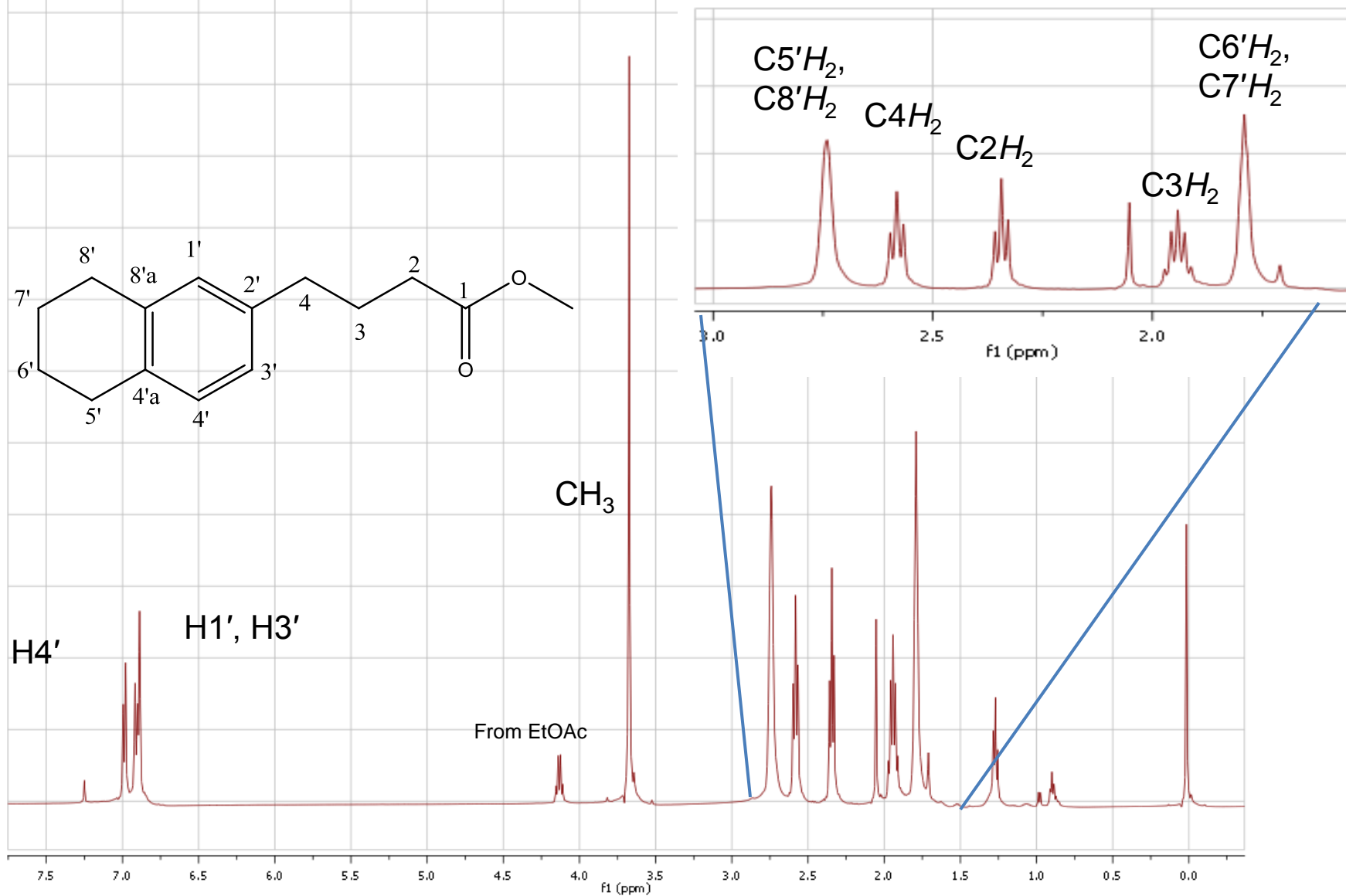


Figure S22. ^1H NMR (300 MHz, CDCl_3) of NA methyl 4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate.

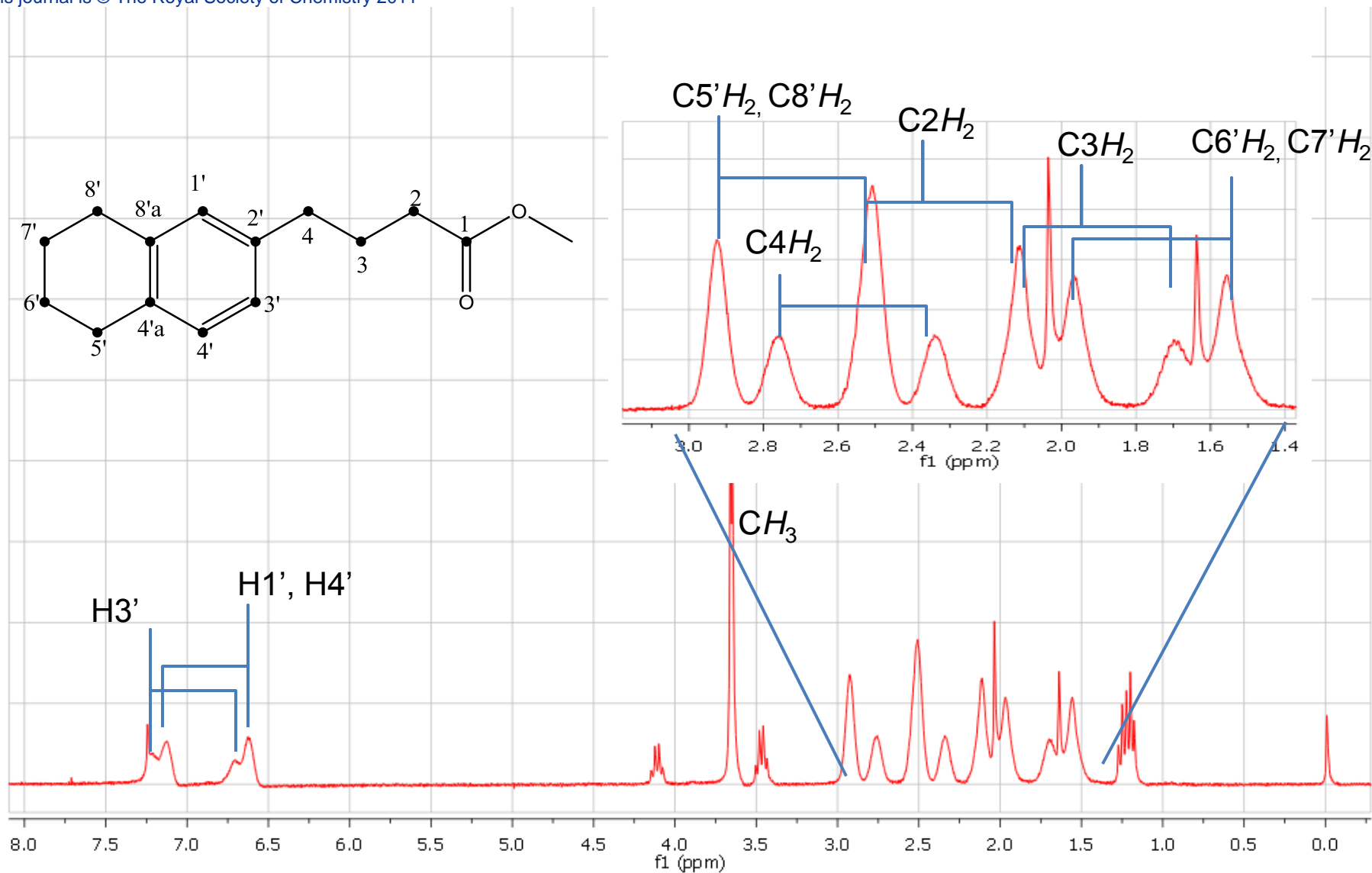


Figure S23. ¹H NMR (300 MHz, CDCl₃) of methyl U-¹³C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate.

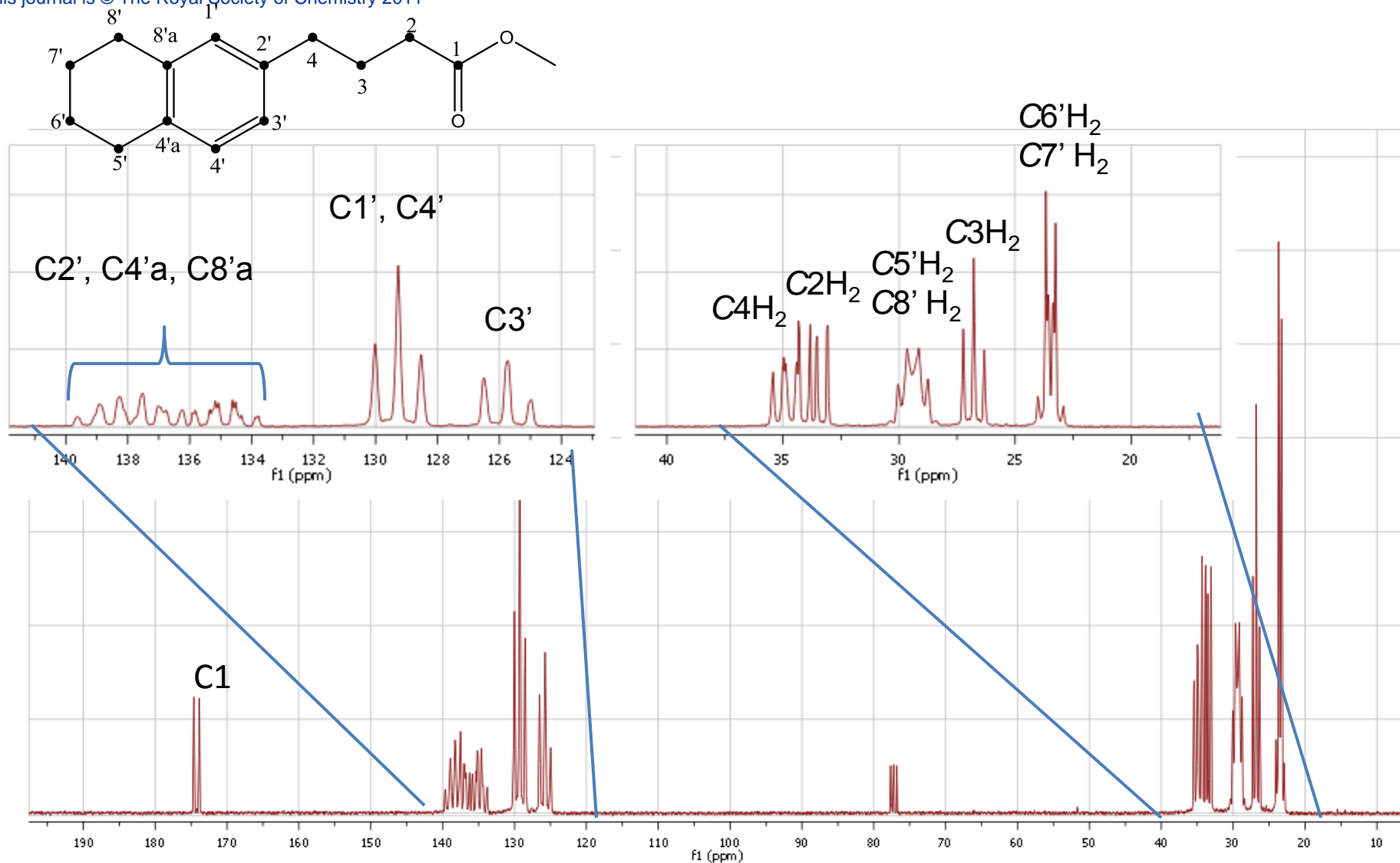
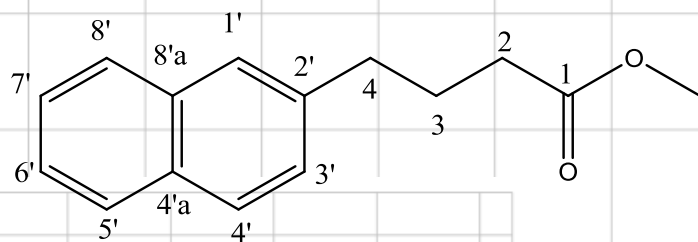


Figure S24. ¹³C NMR (75 MHz, CDCl₃) of methyl U-¹³C-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoate. (For the ¹³C NMR of the corresponding butanoic acid see: Murphy, P. V.; Hubbard, R. E.; Manallack, D. T.; Wills, R. E.; Montanaby, J. G.; Taylor, R. J. K. *Bioorg. Med. Chem.* **1998**, *6*, 2421.)



H4', H5', H8'

H1'

H6', H7'

H3'

C4H₂

C2H₂

C3H₂

CH₃

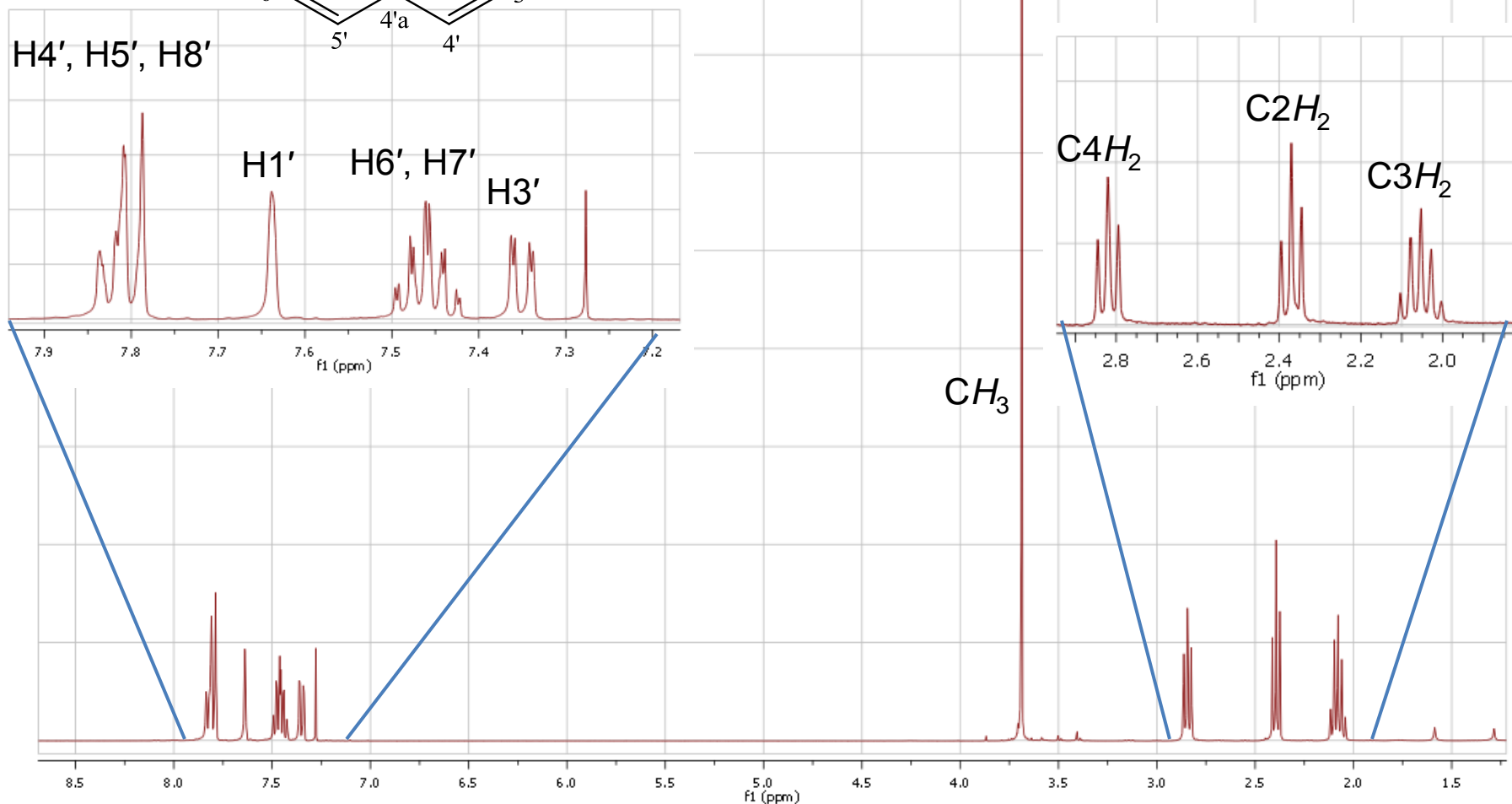


Figure S25. ¹H NMR (400 MHz, CDCl₃) of methyl NA-4-(naphthalen-2-yl)butanoate. (Assignments based on 2-methyl naphthalene: Salman, S. R. *Org. Magn. Reson.* **1982**, 19, 181.)

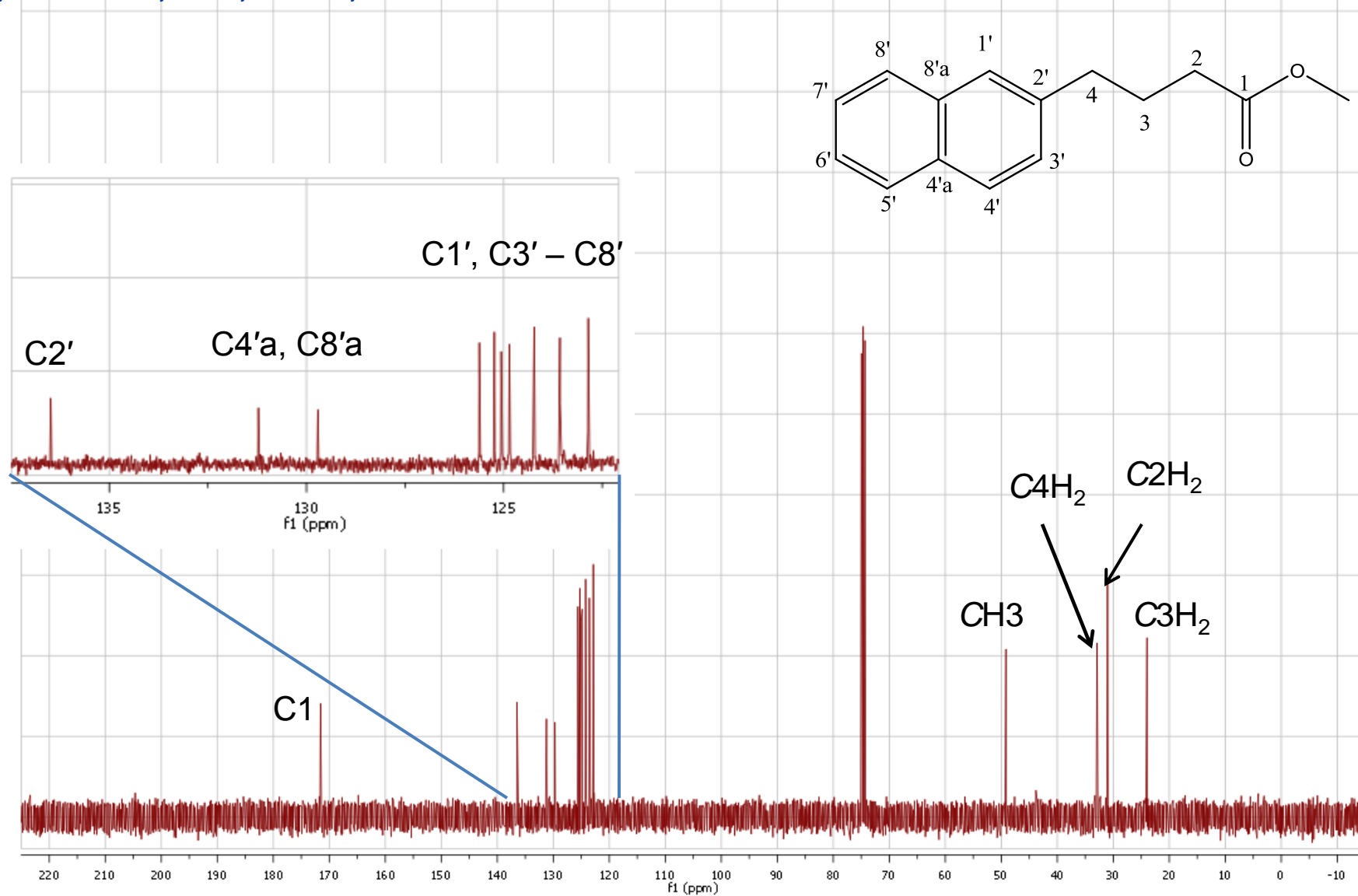


Figure S26. ^{13}C NMR (75 MHz, CDCl_3) of methyl NA-4-(naphthalen-2-yl)butanoate. (Assignments based on 2-methyl naphthalene: Salman, S. R. *Org. Magn. Reson.* **1982**, 19, 181.)

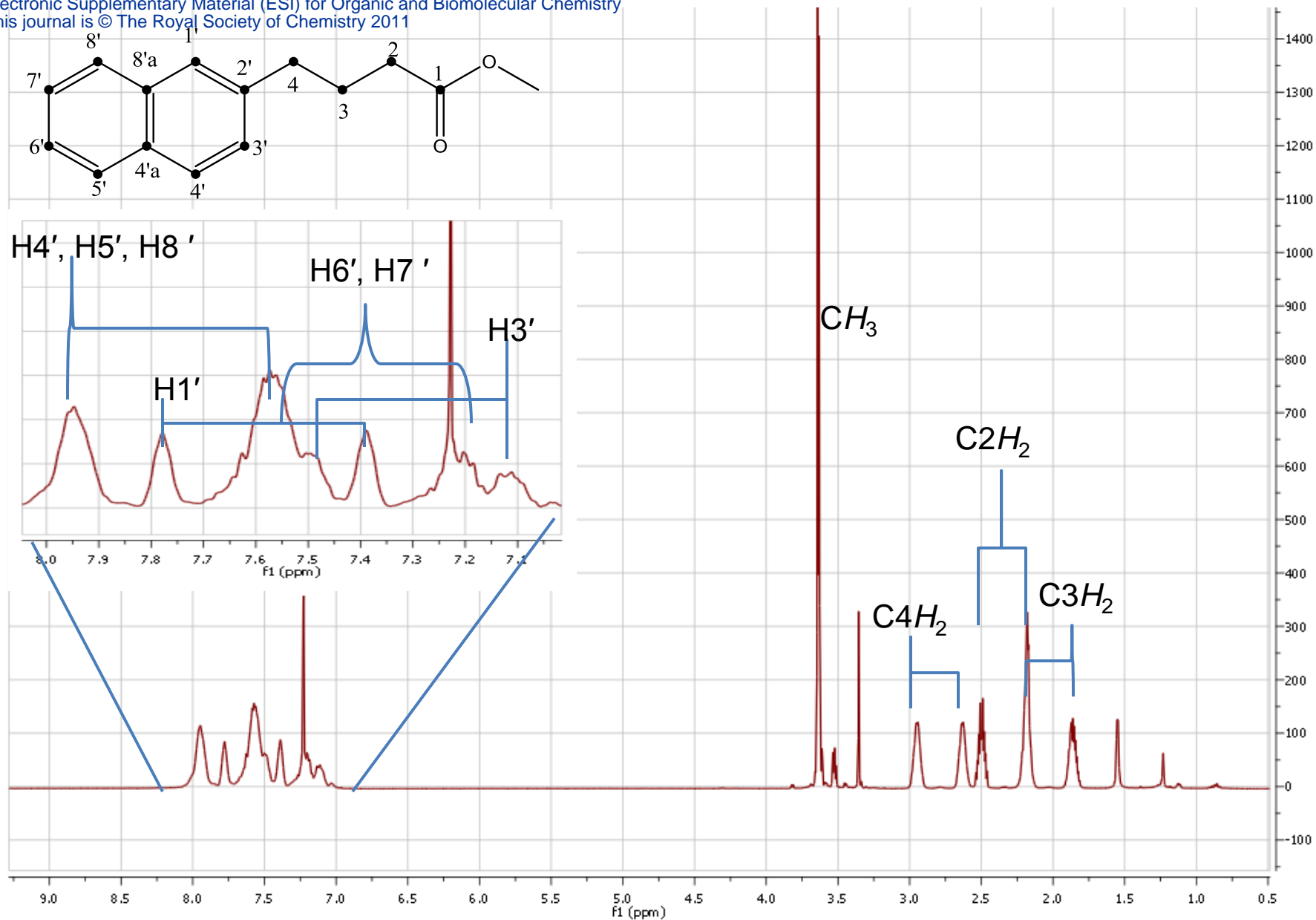


Figure S27. ¹H NMR (300 MHz, CDCl₃) of methyl U-¹³C-4-(naphthalen-2-yl)butanoate (**10**). (Assignments based on 2-methyl naphthalene: Salman, S. R. *Org. Magn. Reson.* **1982**, *19*, 181.)

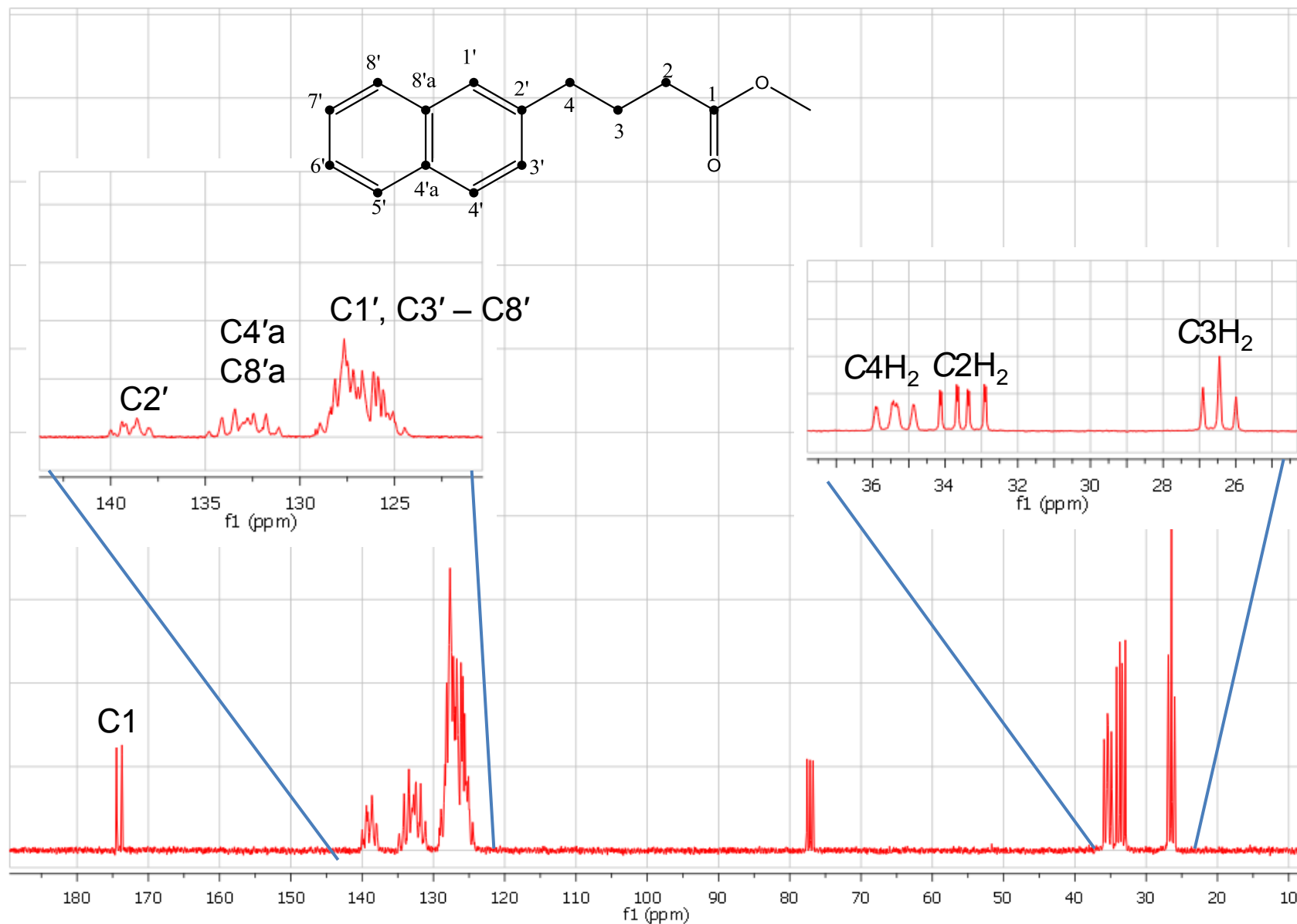


Figure S28. ^{13}C NMR (75 MHz, CDCl_3) of methyl U- ^{13}C -4-(naphthalen-2-yl)butanoate (**10**). (Assignments based on 2-methyl naphthalene: Salman, S. R. *Org. Magn. Reson.* **1982**, *19*, 181.)

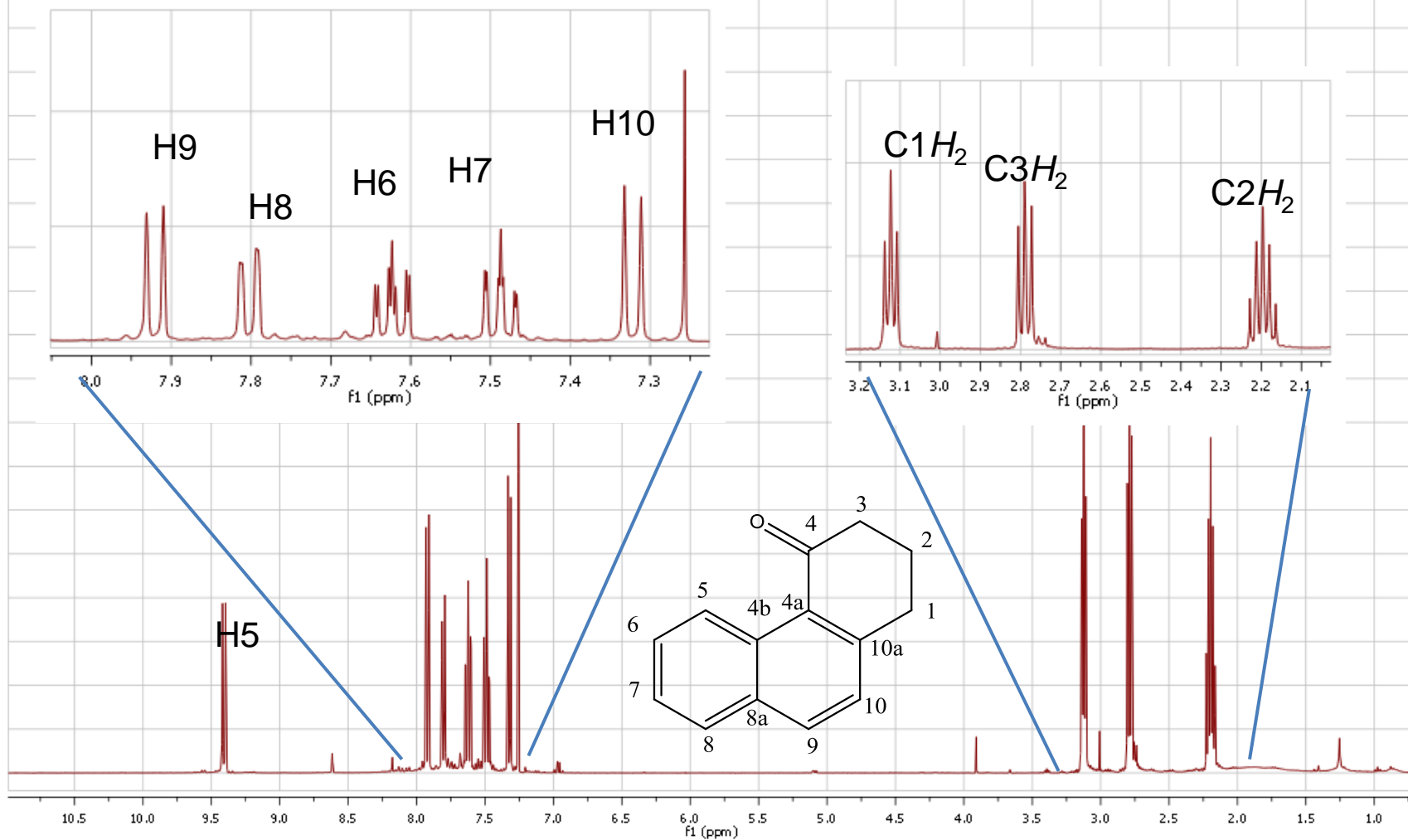


Figure S29. ^1H NMR (400 MHz, CDCl_3) of NA-2,3-dihydrophenanthren-4(1H)-one. (Chemical shifts identical to report with assignments: Ernst, L.; Stolle, R. *Magn. Reson. Chem.* **1989**, 27, 796.)

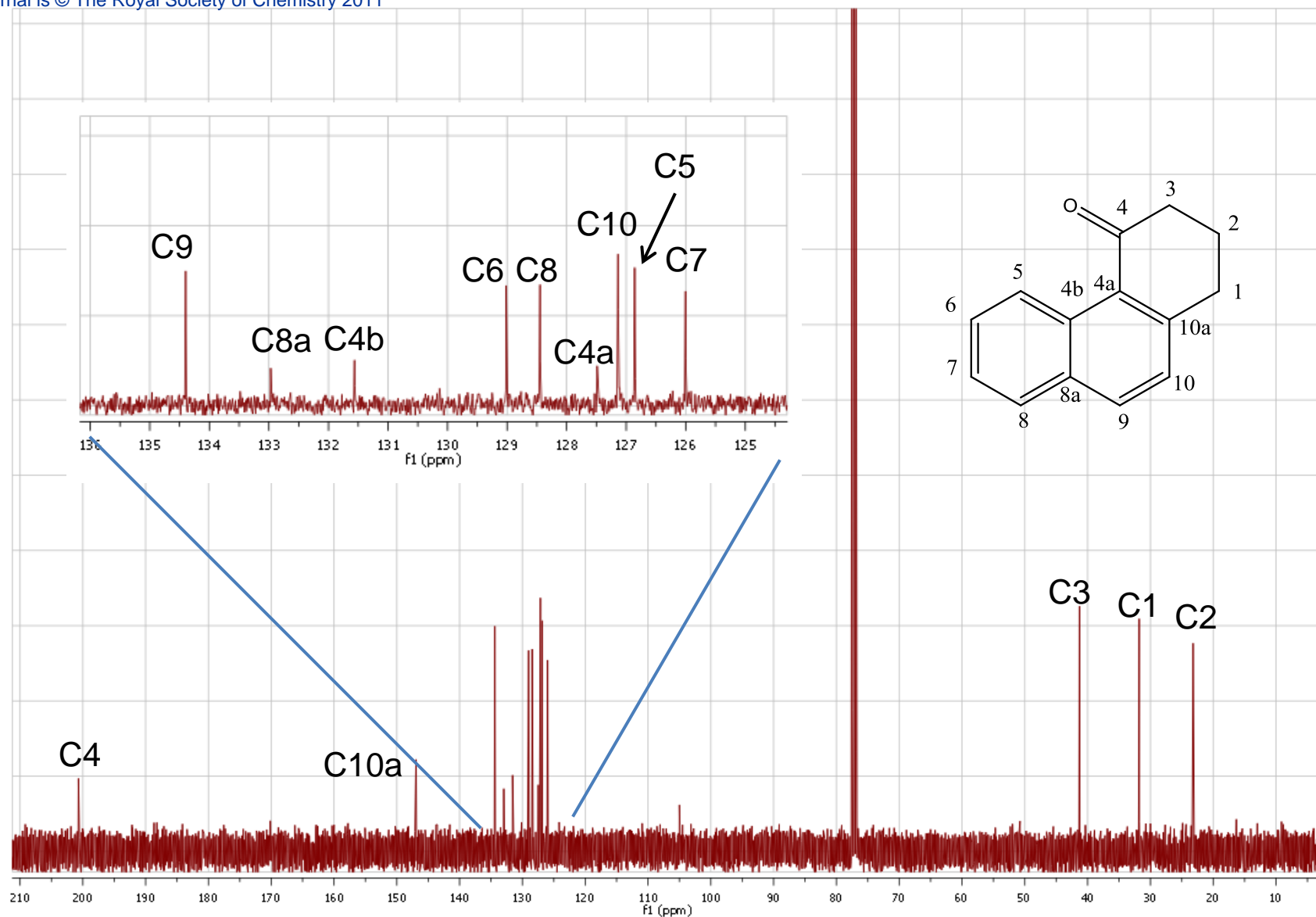


Figure S30. ^{13}C NMR (100 MHz, CDCl_3) of NA-2,3-dihydrophenanthren-4(1H)-one. (Chemical shifts identical to report with assignments: Ernst, L.; Stolle, R. *Magn. Reson. Chem.* **1989**, 27, 796.)

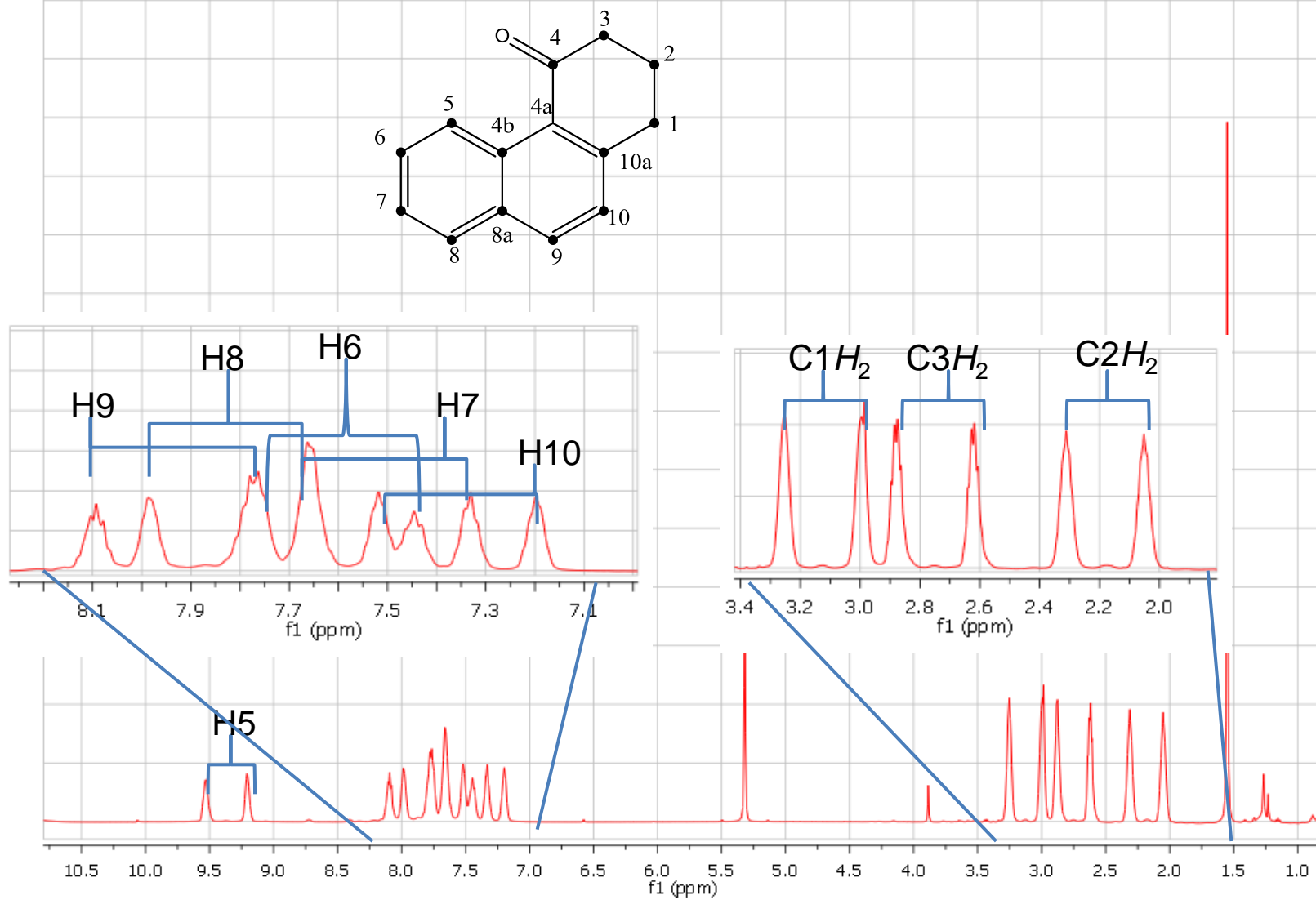


Figure S31. ¹H NMR (500 MHz, CDCl₃) of U-¹³C- 2,3-dihydrophenanthren-4(1H)-one (**8**).

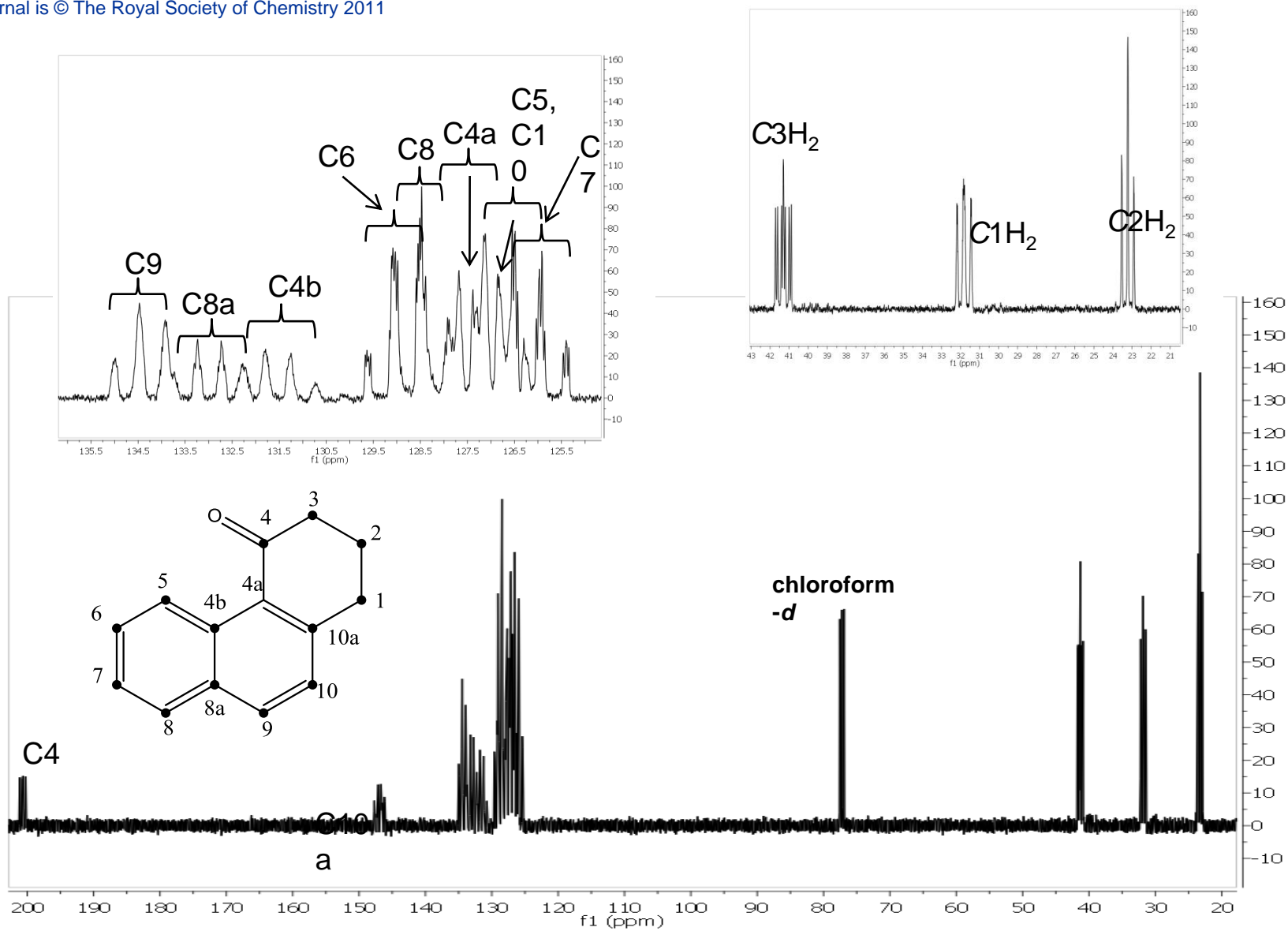


Figure S32. ^{13}C NMR (100 MHz, CDCl_3) of U- ^{13}C -2,3-dihydrophenanthren-4(1H)-one (**8**).

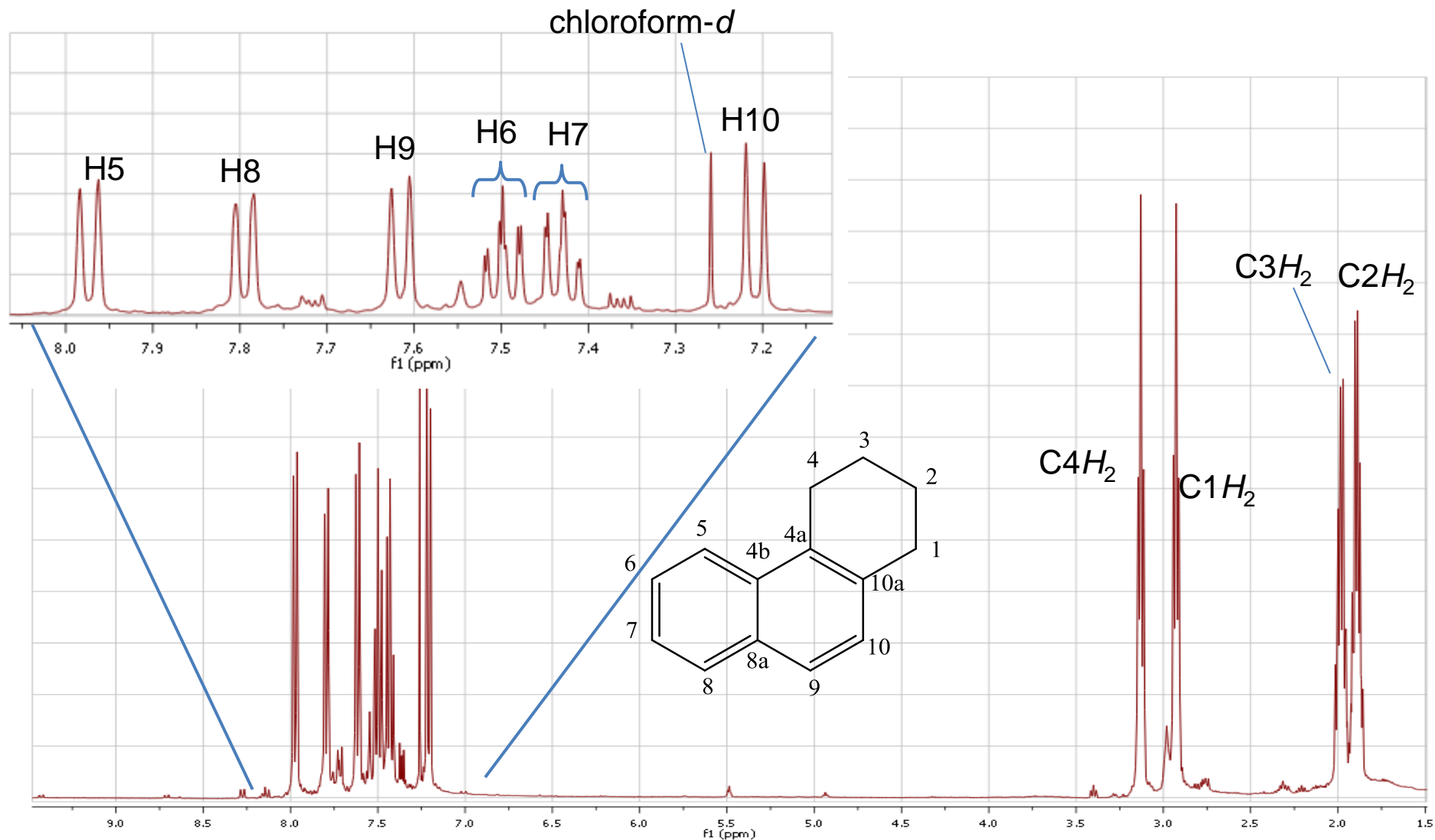


Figure S33. ^1H NMR (400 MHz, CDCl_3) of NA-1,2,3,4-tetrahydrophenanthrene. (Chemical shifts identical to report with assignments: Ernst, L.; Stolle, R. *Magn. Reson. Chem.* **1989**, 27, 796.)

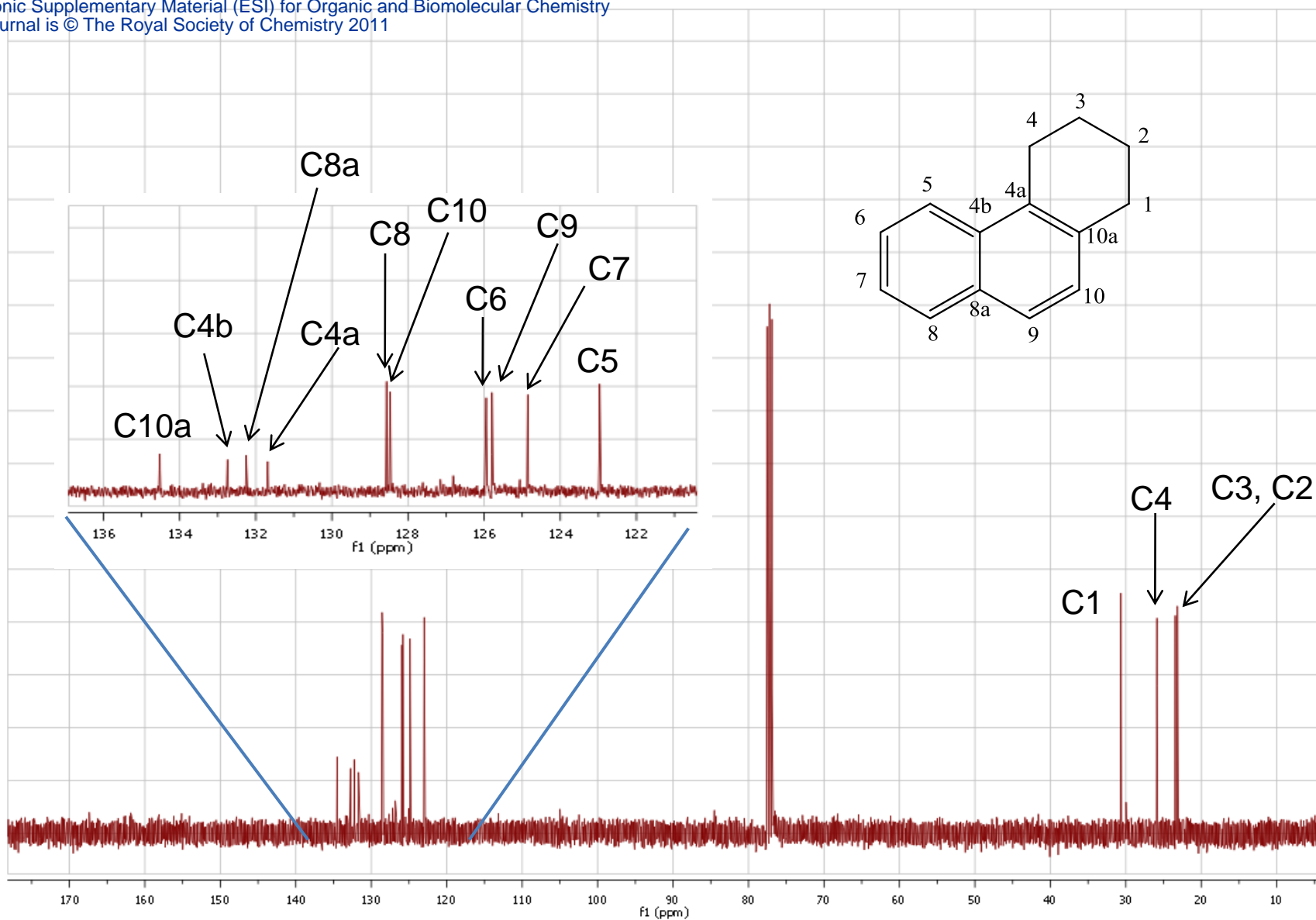


Figure S34. ^{13}C NMR (100 MHz, CDCl_3) of NA-1,2,3,4-tetrahydrophenanthrene. (Chemical shifts identical to report with assignments: Ernst, L.; Stolle, R. *Magn. Reson. Chem.* **1989**, *27*, 796.)

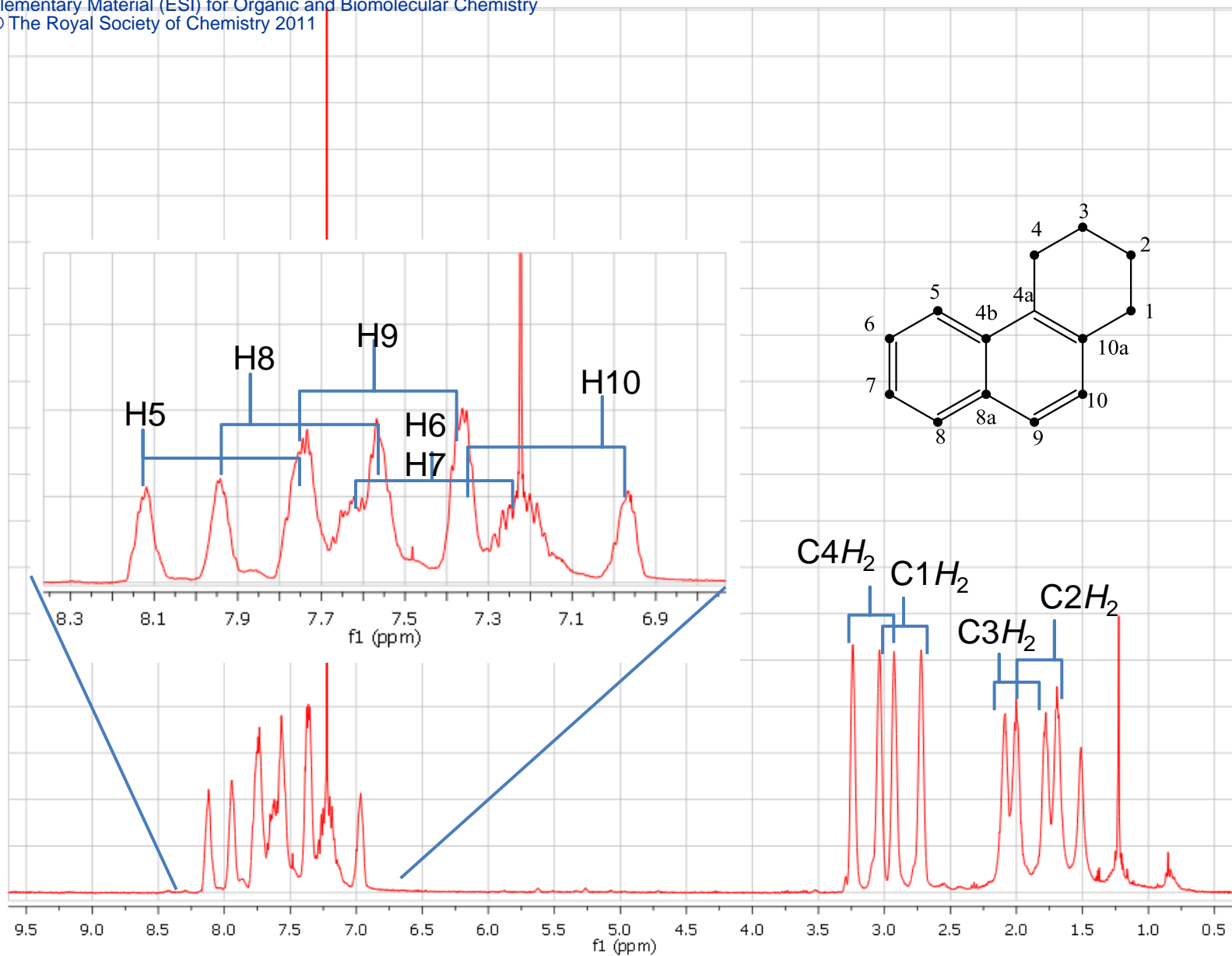


Figure S35. ^1H NMR (400 MHz, CDCl_3) of $\text{U-}^{13}\text{C}$ -1,2,3,4-tetrahydrophenanthrene.

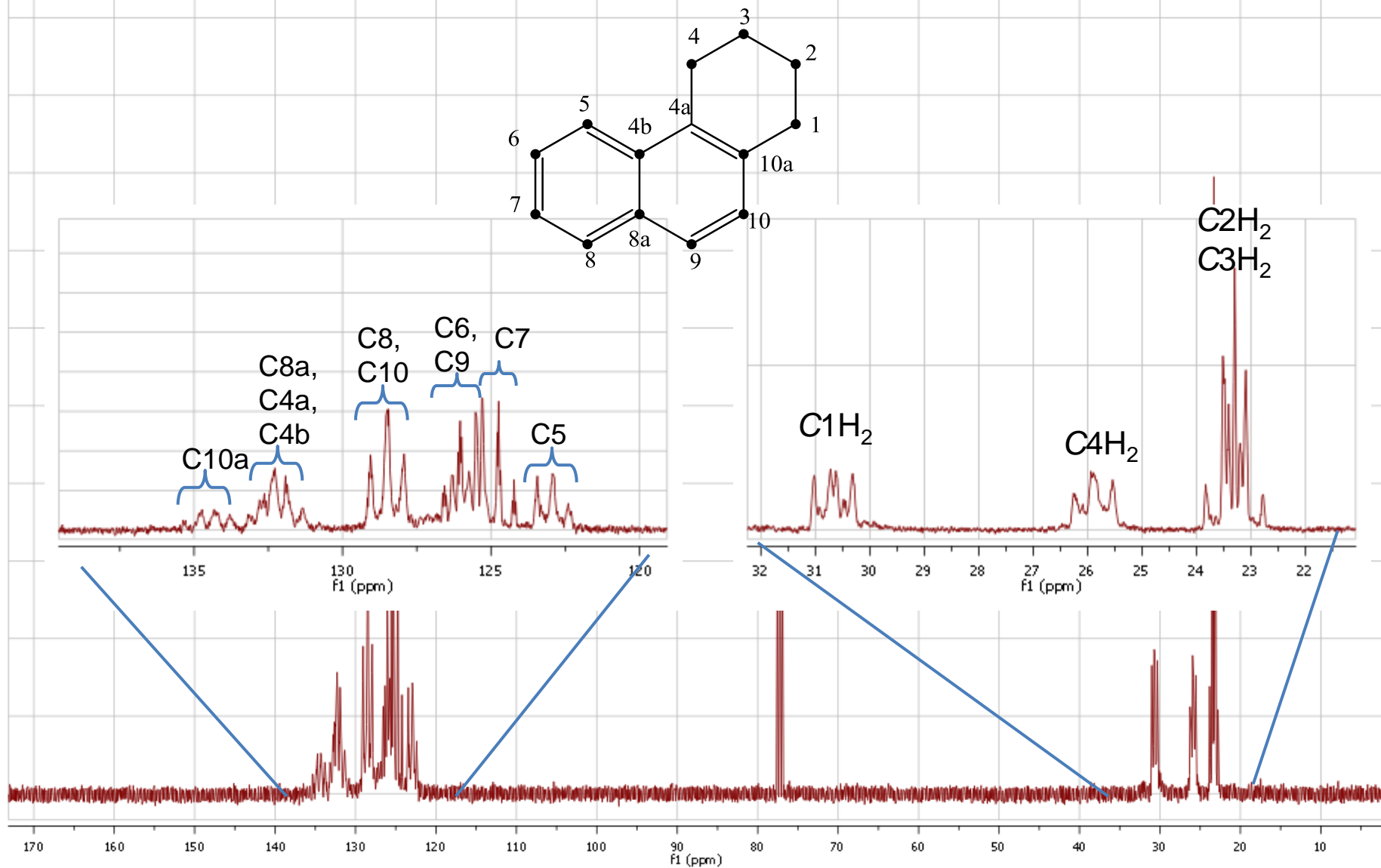


Figure S36. ^{13}C NMR (100 MHz, CDCl_3) of U- ^{13}C -1,2,3,4-tetrahydrophenanthrene.

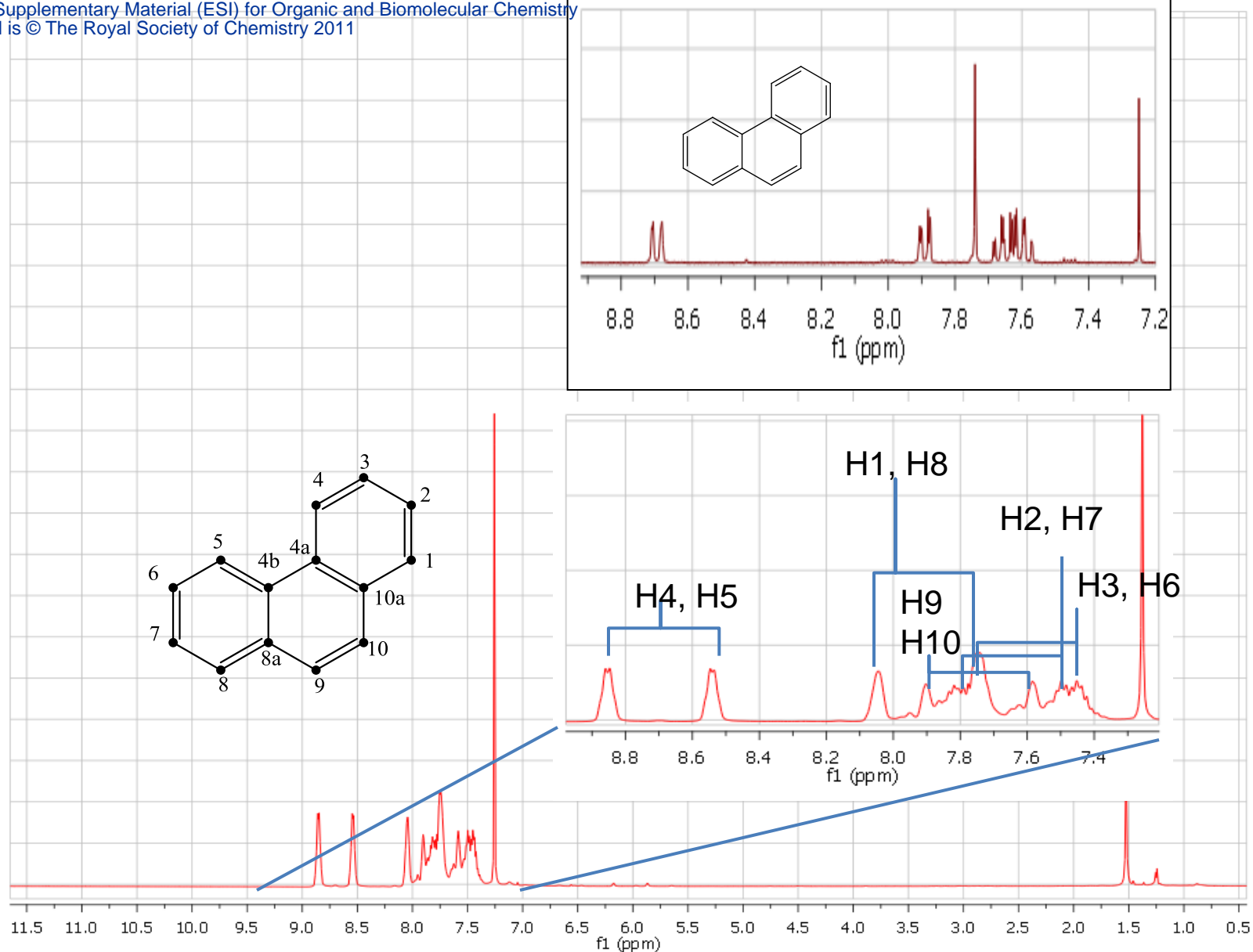


Figure S37. ¹H NMR (500 MHz, CDCl₃) of U-¹³C-phenanthrene (**11**). (Inset: ¹H NMR of NA-phenanthrene. Chemical shifts identical to report with assignments: Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem.* **2005**, *43*, 588.)

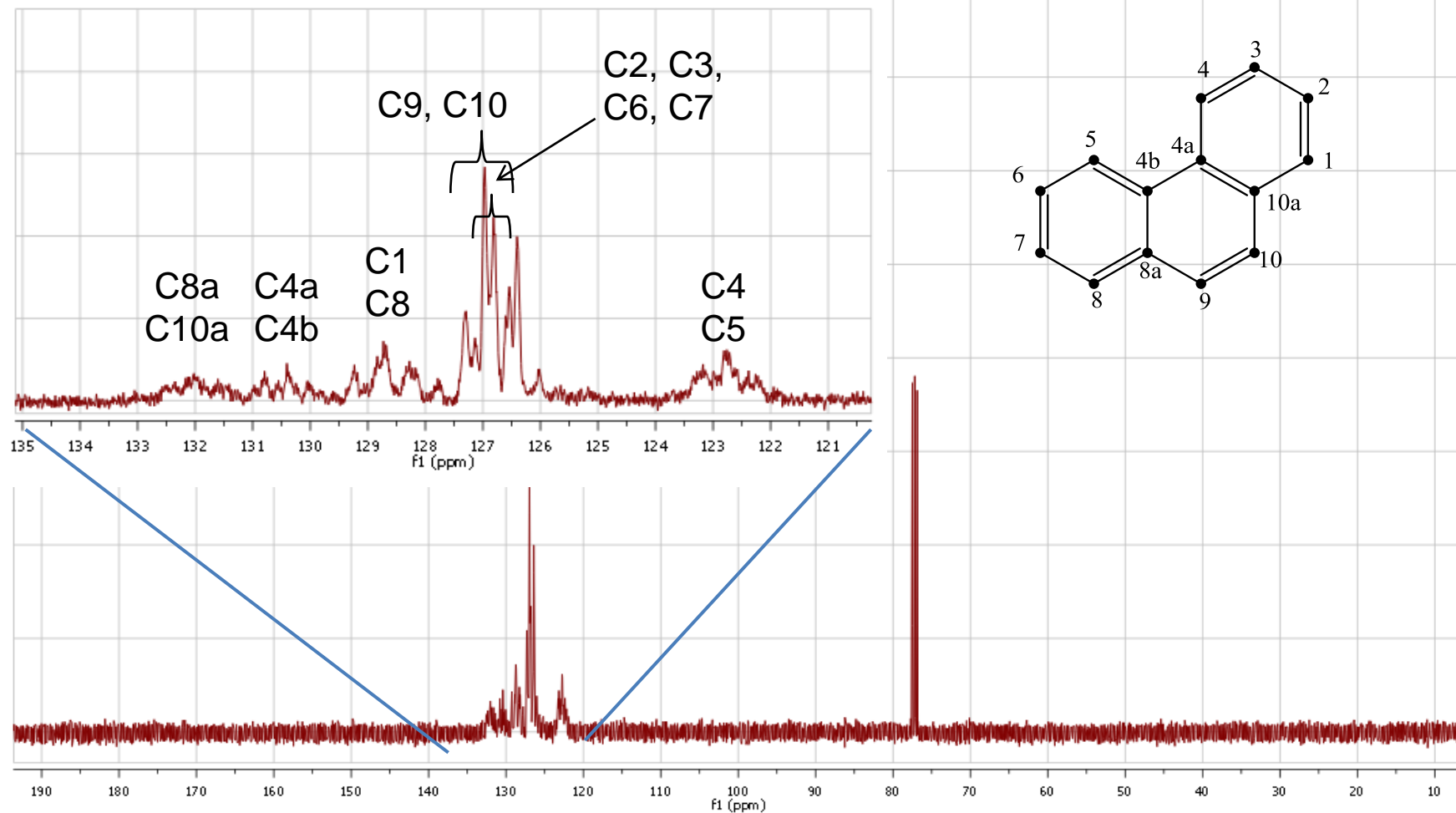


Figure S38. ^{13}C NMR (100 MHz, CDCl_3) of U- ^{13}C -phenanthrene (**11**). (For ^{13}C NMR assignments of NA-phenanthrene see: Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem.* **2005**, *43*, 588.)

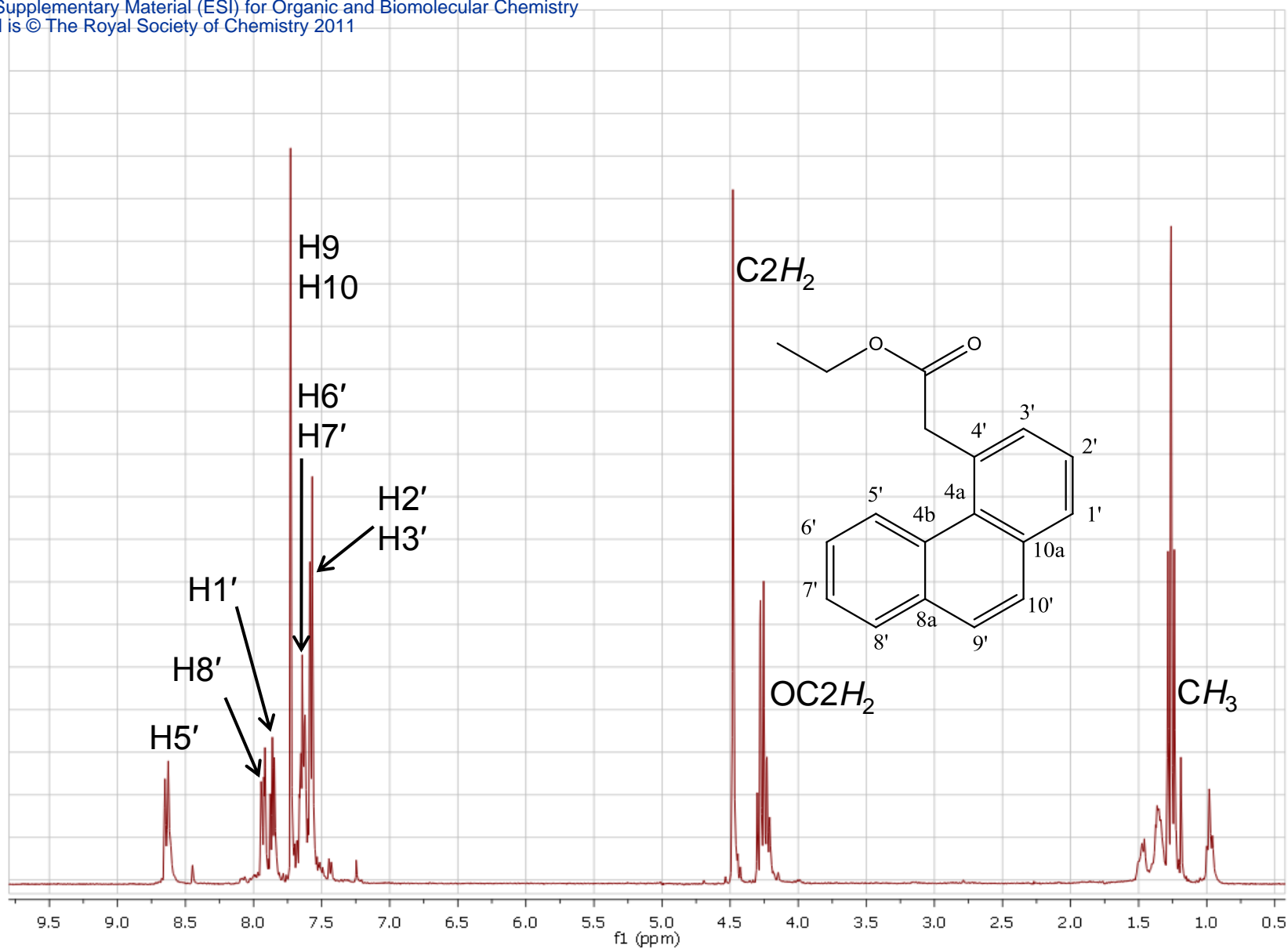


Figure S39. ^1H NMR (300 MHz, CDCl_3) of NA-ethyl 2-(phenanthren-4-yl)acetate. (Assignments based on 4-Me-phenanthrene: Ibrom, K.; Kohn, G. W.; Boeckmann, K.U.; Kraft, R.; Holba-Schulz, P.; Ernst, L. *Org. Lett.* **2000**, 2, 4111.)

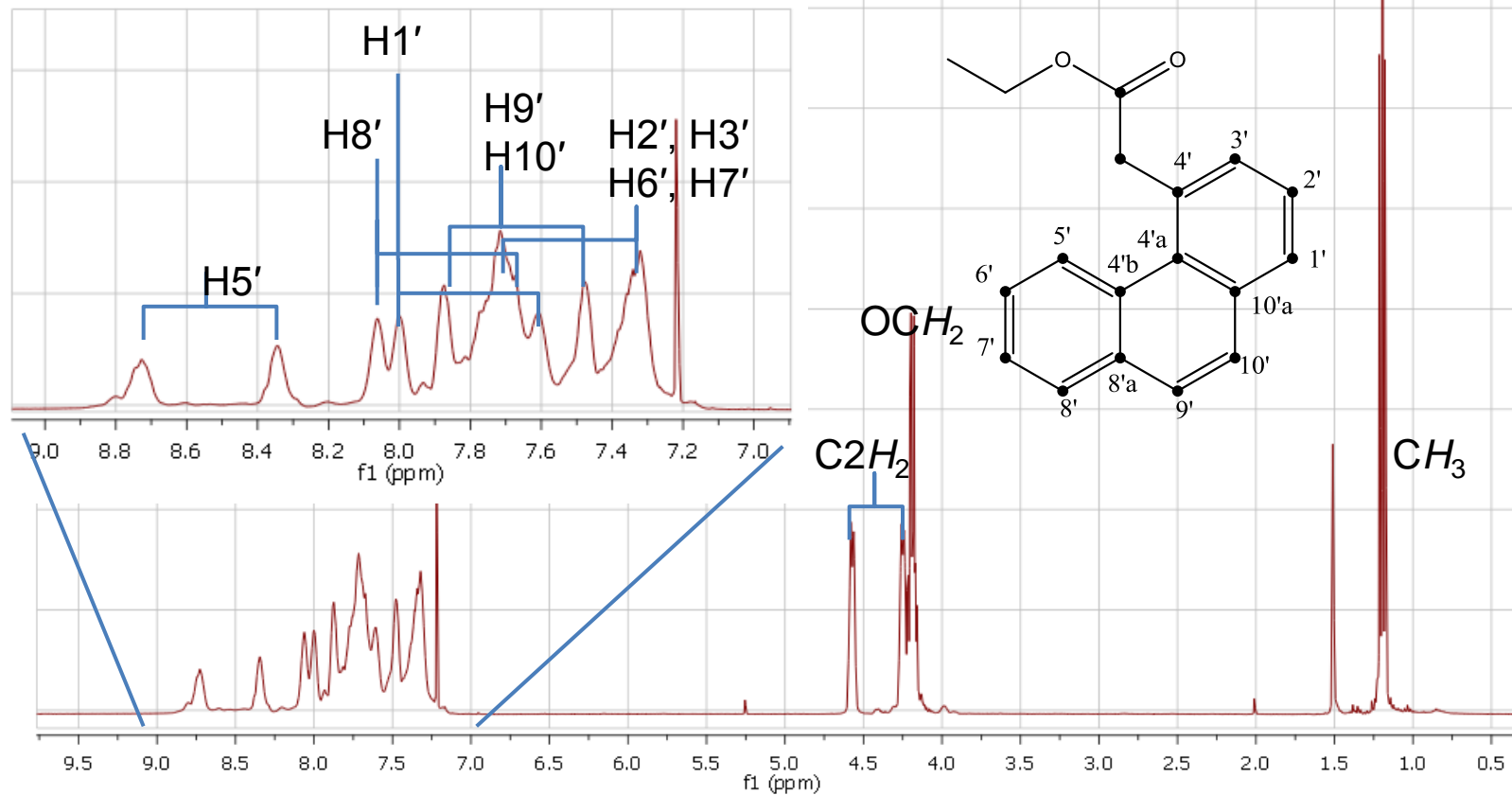


Figure S40. ^1H NMR (400 MHz, CDCl_3) of ethyl U- ^{13}C -2-(phenanthren-4-yl)acetate (**13**).

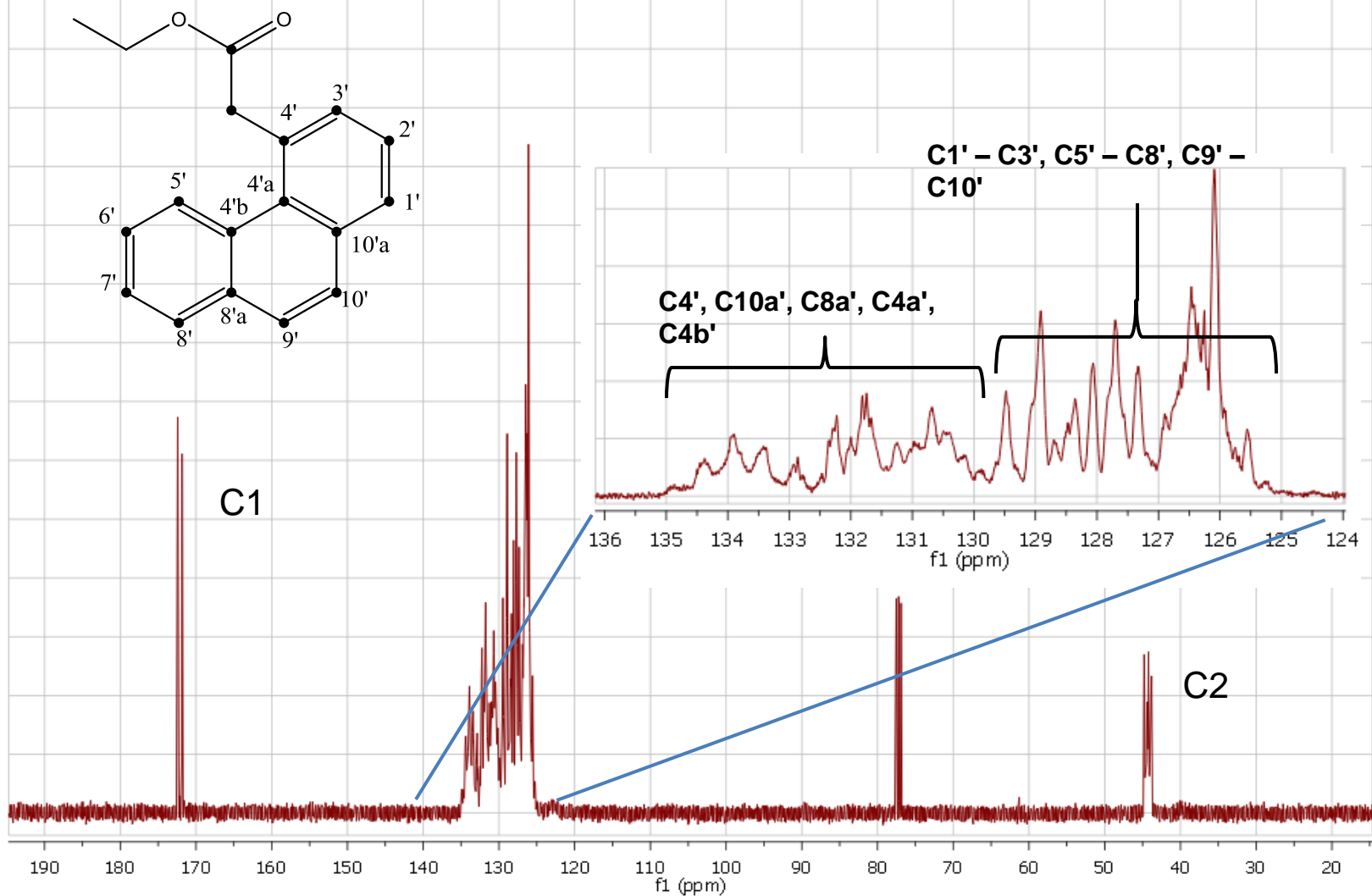


Figure S41. ^{13}C NMR (100 MHz, CDCl_3) of ethyl U- ^{13}C -2-(phenanthren-4-yl)acetate (**13**). (Assignments based on 4-Me-phenanthrene: Ibrom, K.; Kohn, G. W.; Boeckmann, K.U.; Kraft, R.; Holba-Schulz, P.; Ernst, L. *Org. Lett.* **2000**, 2, 4111.)

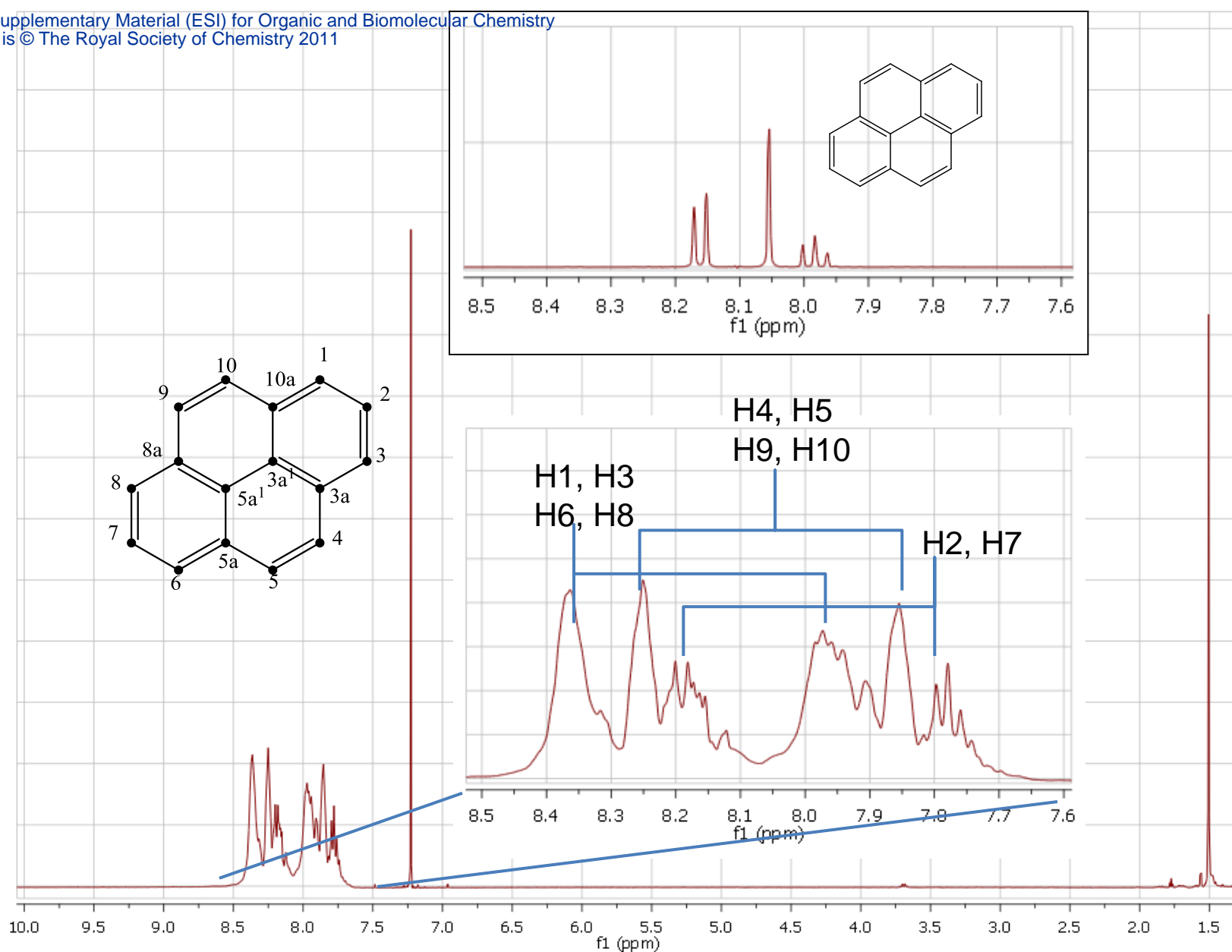


Figure S42. ^1H NMR (400 MHz, CDCl_3) of $\text{U-}^{13}\text{C}$ -pyrene (**14**). (Inset: ^1H NMR of NA-pyrene. Chemical shifts identical to report with assignments: Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem.* **2005**, *43*, 588.)

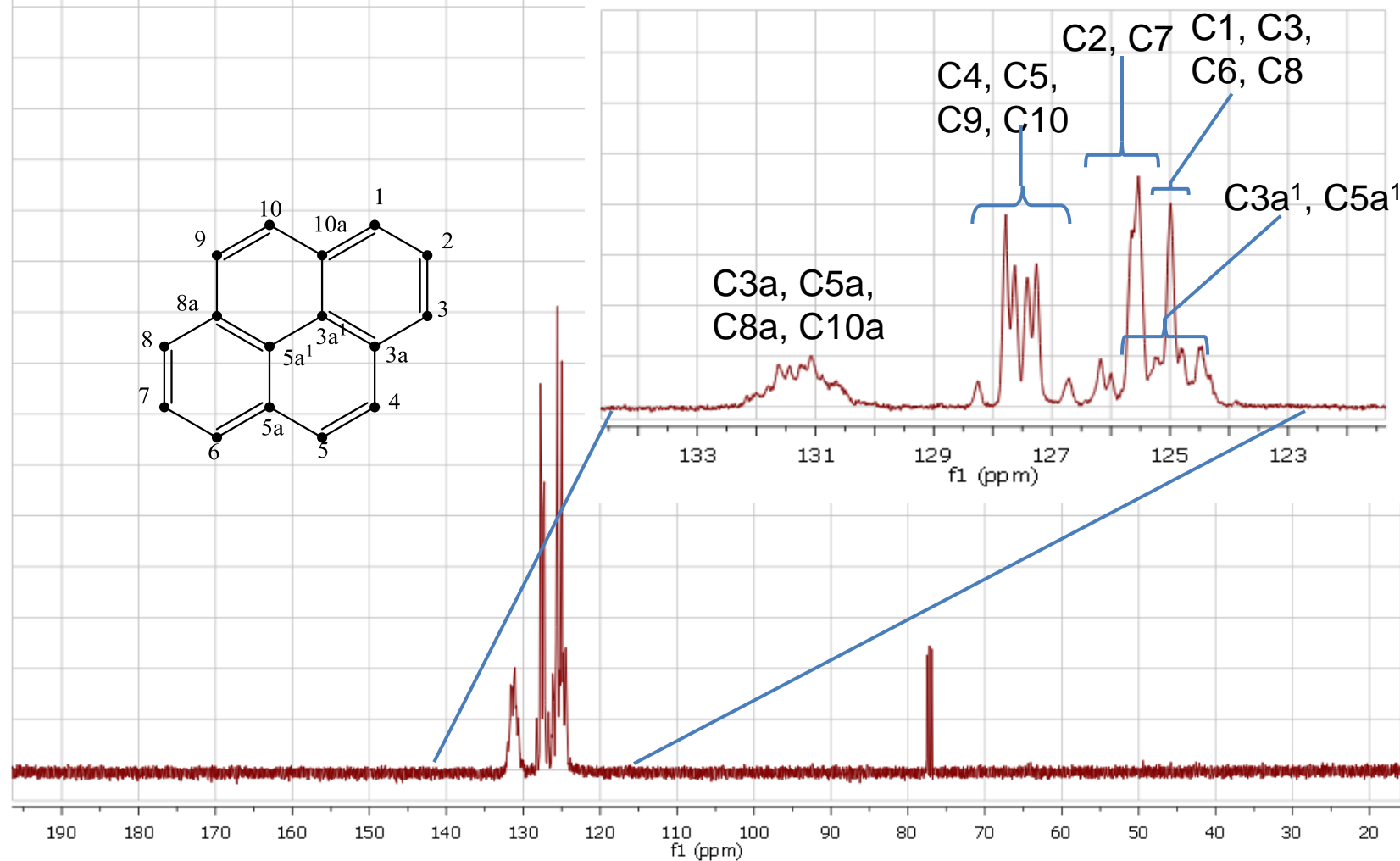


Figure S43. ¹³C NMR (100 MHz, CDCl₃) of U-¹³C-pyrene (**14**). (For ¹³C NMR assignments of NA-pyrene see: Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem.* **2005**, *43*, 588.)

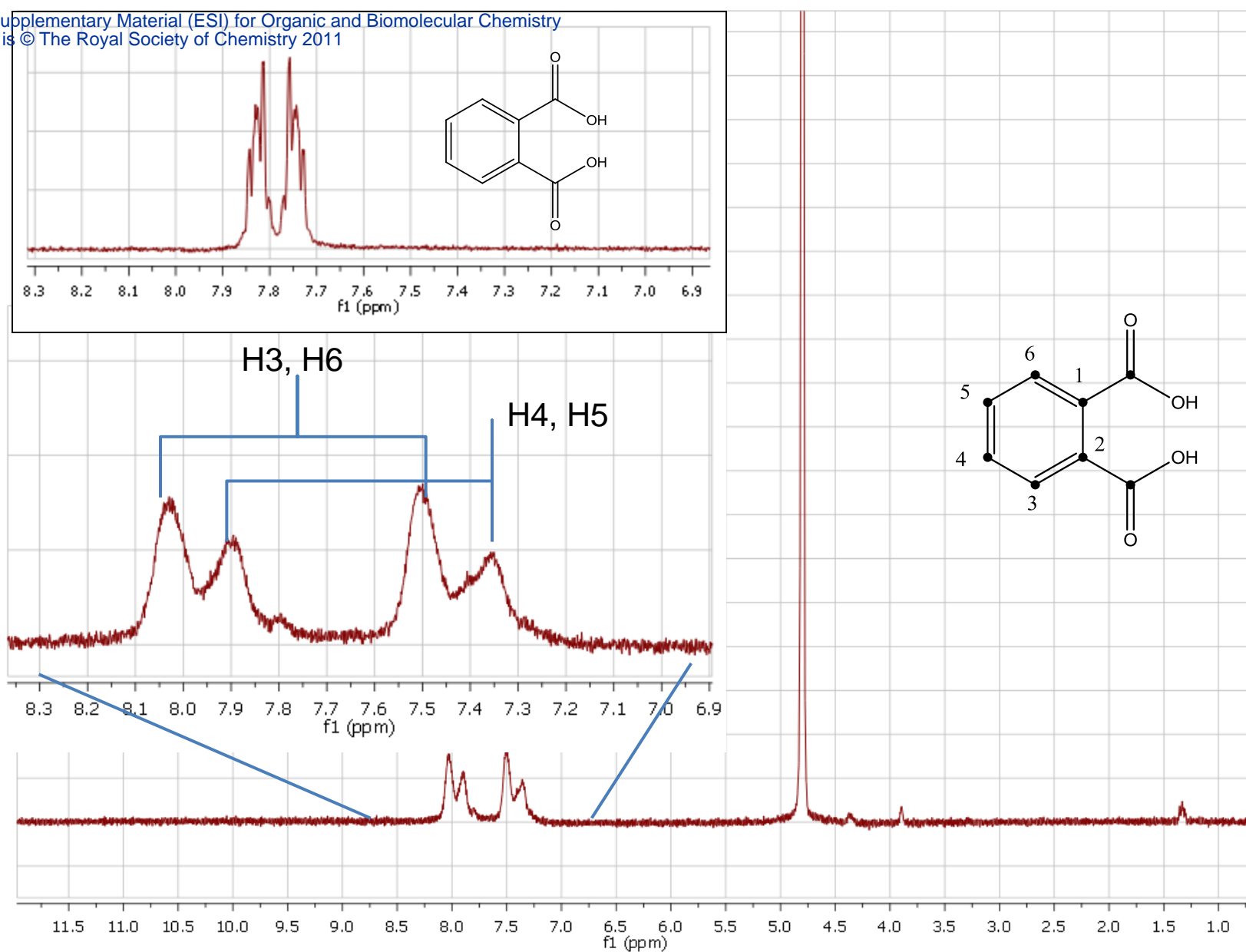


Figure S44. ¹H NMR (300 MHz, DMSO-*d*₆) of U-¹³C-phthalic acid (**15**). (inset: ¹H NMR of NA-phthalic acid. Chemical shifts identical to report: AIST: Integrated Spectral Database System of Organic Compounds.)

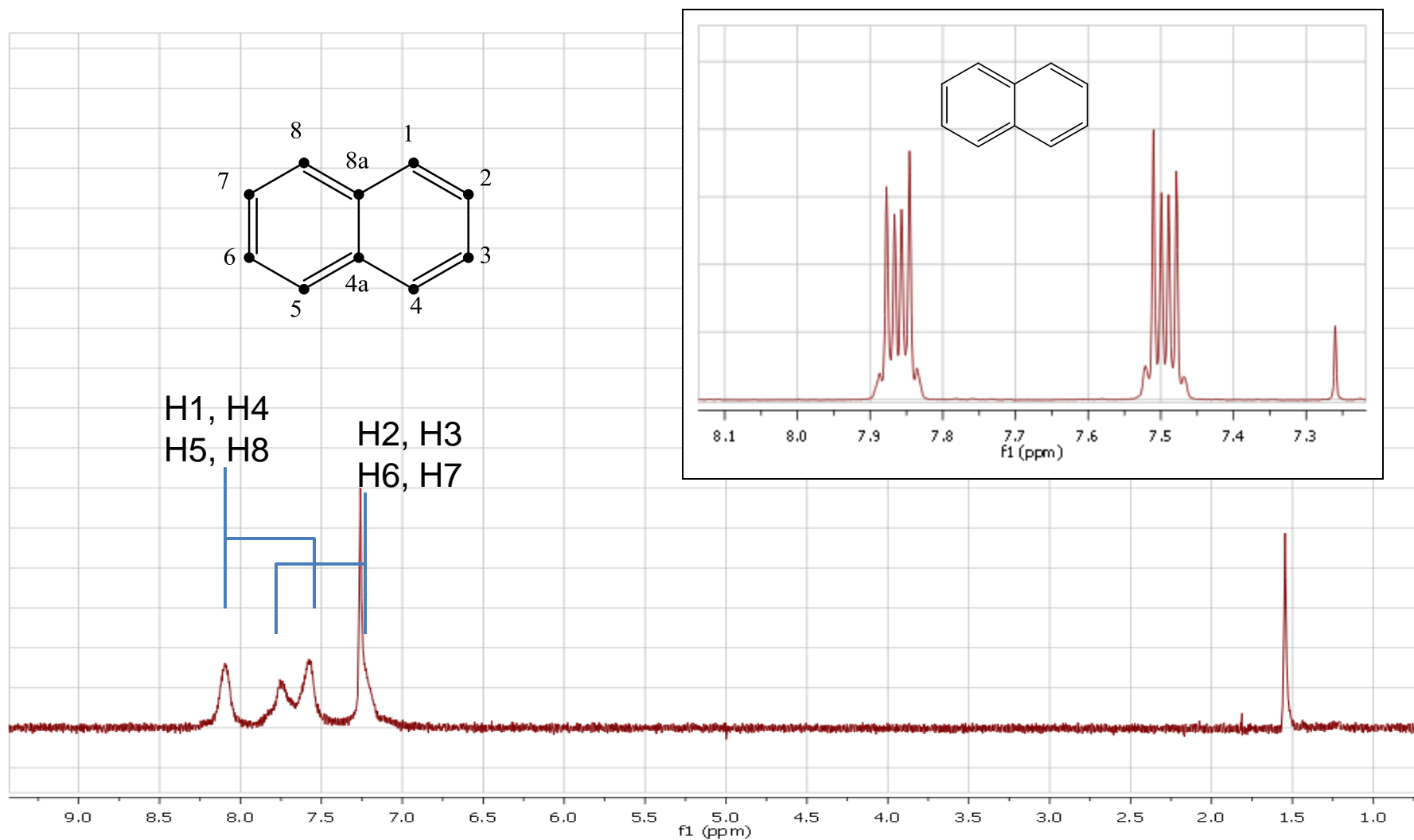


Figure S45. ¹H NMR (300 MHz, CDCl₃) of U-¹³C-naphthalene (**7**). (Inset: ¹H NMR of NA-naphthalene. Chemical Shift identical to report with assignments: Pouchert, C.J.; Behnke, J. (eds). *The Aldrich Library of ¹³C and ¹H FT NMR Spectra*. Aldrich Chemical: Milwaukee, WI, 1993.

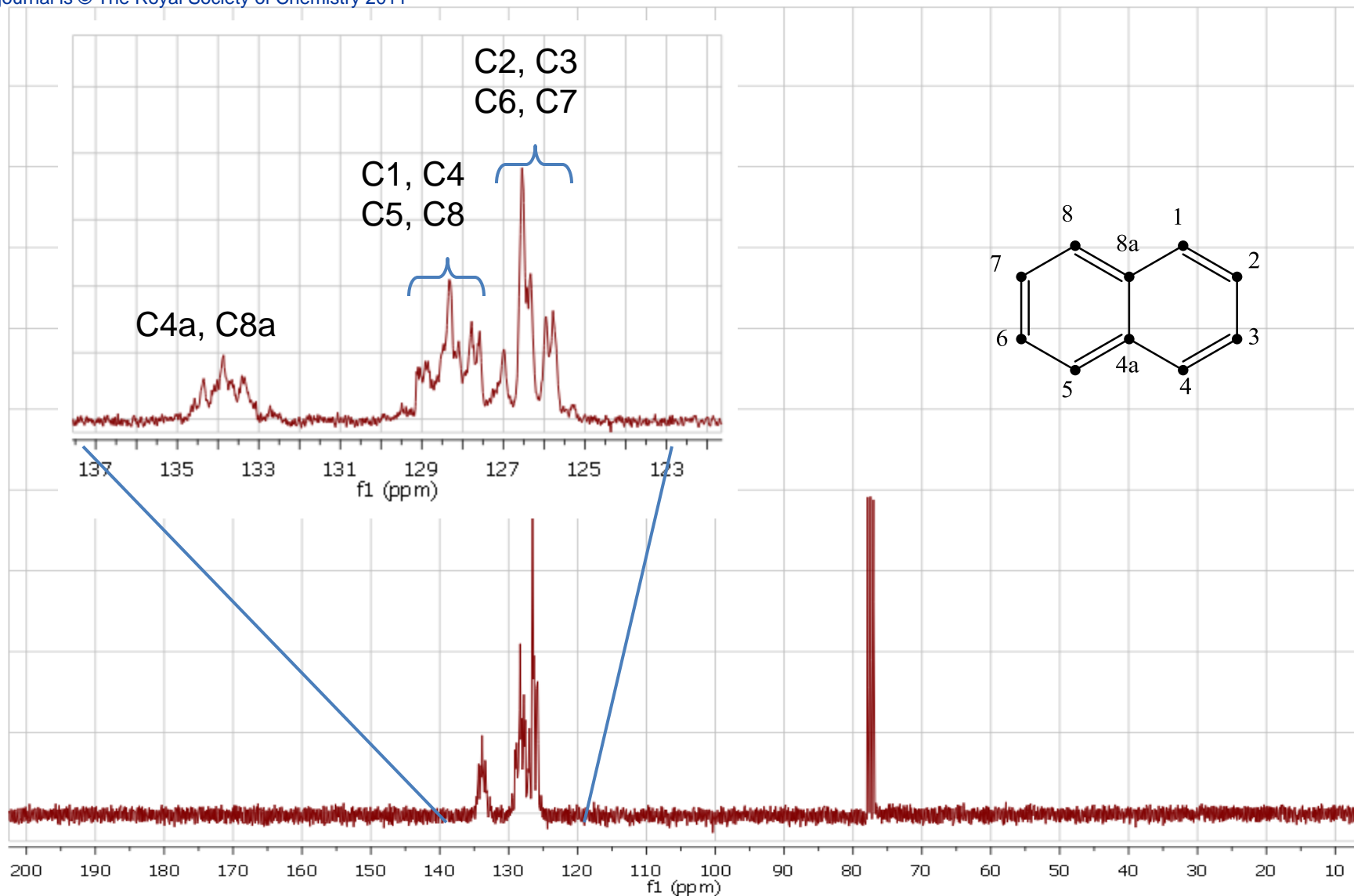


Figure S46. ^{13}C NMR (75 MHz, CDCl_3) of U- ^{13}C -naphthalene (7). (For ^{13}C NMR assignments of NA-naphthalene see: Kitching, W.; Bullpitt, M.; Doddrell, D.; Adcock, W. *Org. Magn. Reson.* **1974**, *6*, 289. Seita, J.; Sandstrom, J.; Drakenberg, T. *Org. Magn. Reson.* **1978**, *11*, 239.)

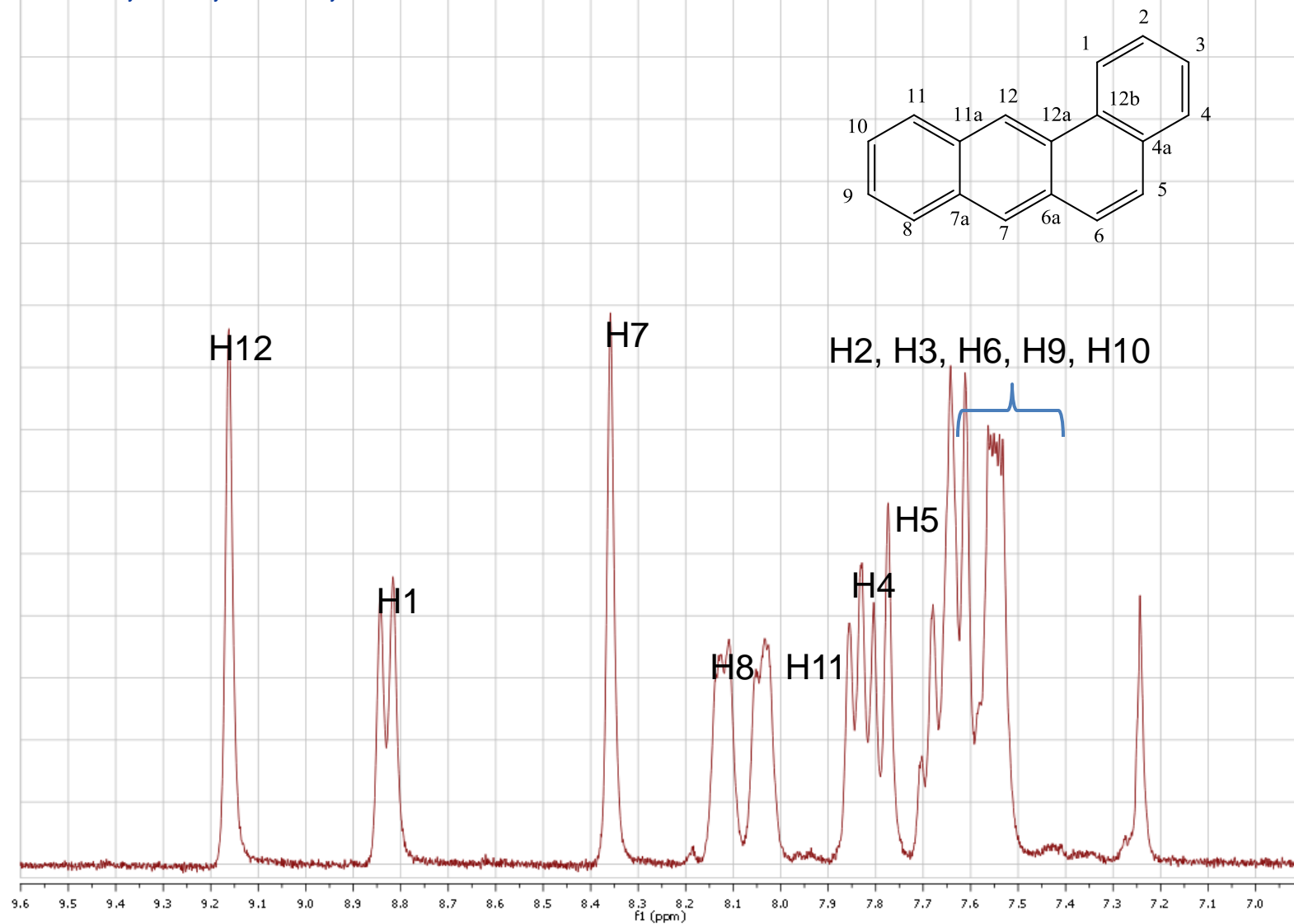


Figure S47. ¹H NMR (500 MHz, CDCl₃) of NA-benz[a]anthracene. (Chemical Shifts identical to report with assignment: Moody, J. D.; Freeman, J. P.; Cerniglia, C. E *Biodegradation* **2005**, *16*, 513.)

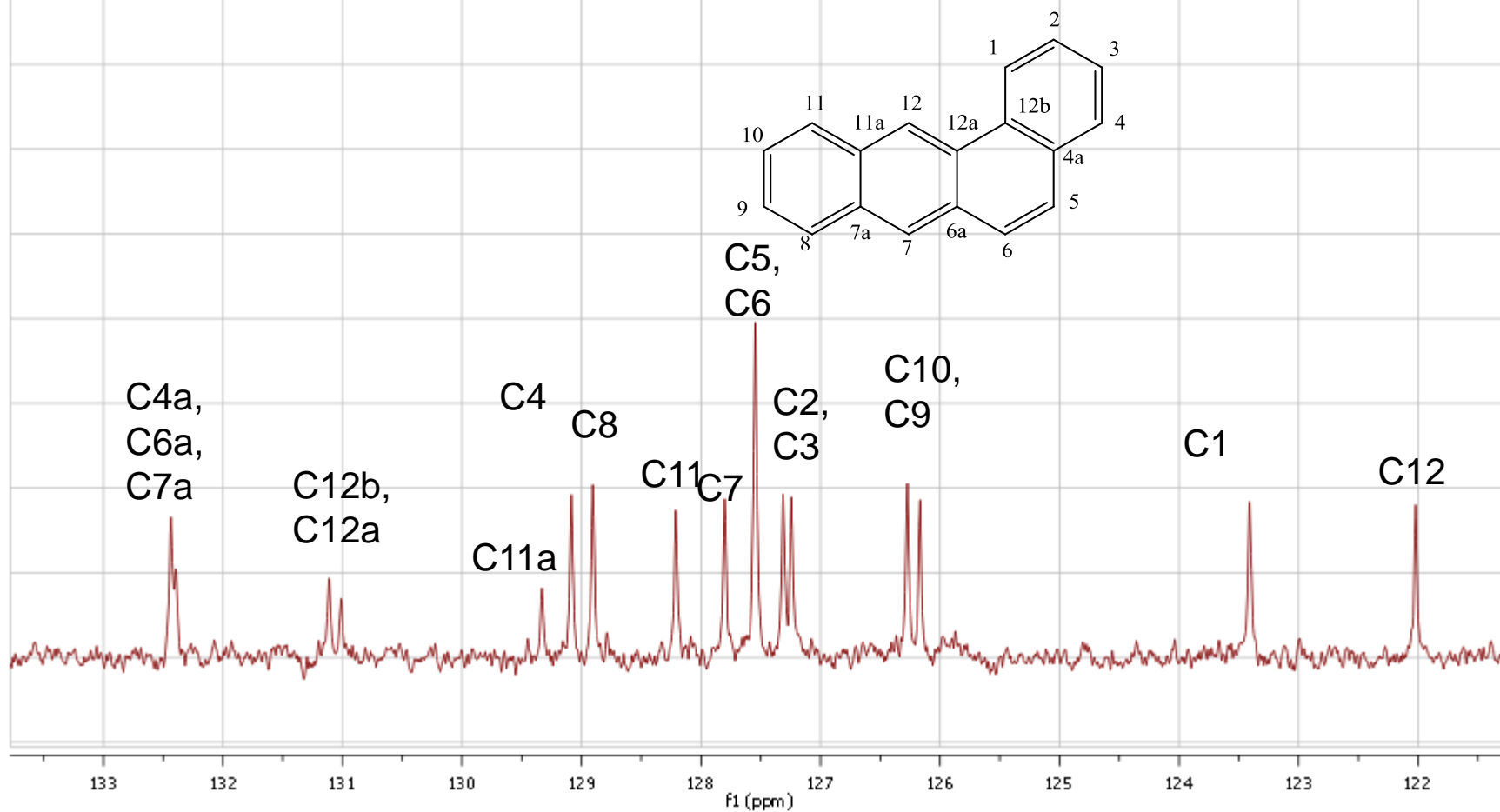


Figure S48. ¹³C NMR (75 MHz, CDCl₃) of NA-benz[a]anthracene. (Chemical shifts identical to report with assignments: Cox, R. H.; Levy, L. A.; *Org. Magn. Reson.* **1983**, *21*, 173.)

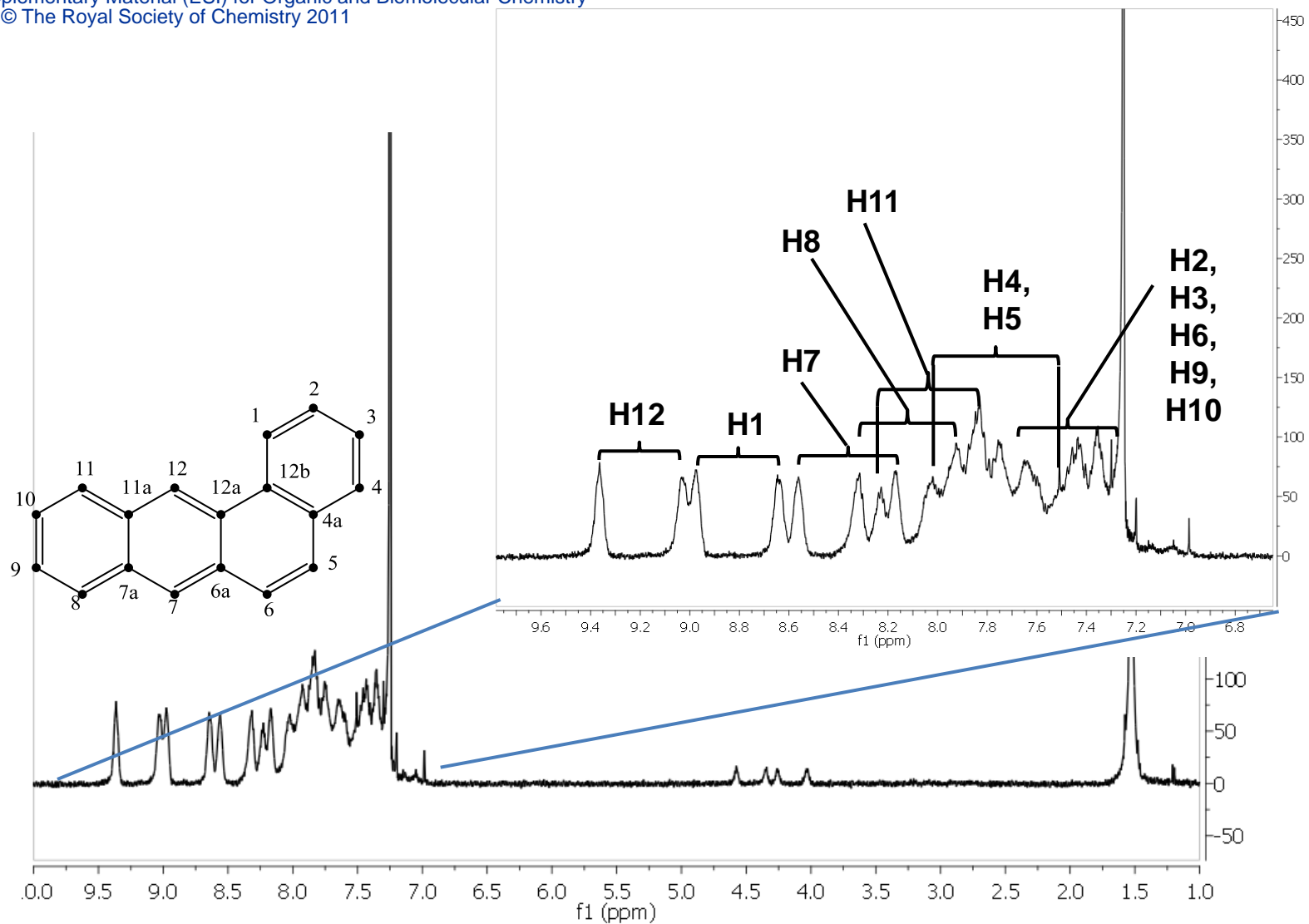


Figure S49. ^1H NMR (500 MHz, CDCl_3) of $\text{U-}^{13}\text{C}$ -benz[*a*]anthracene (17).

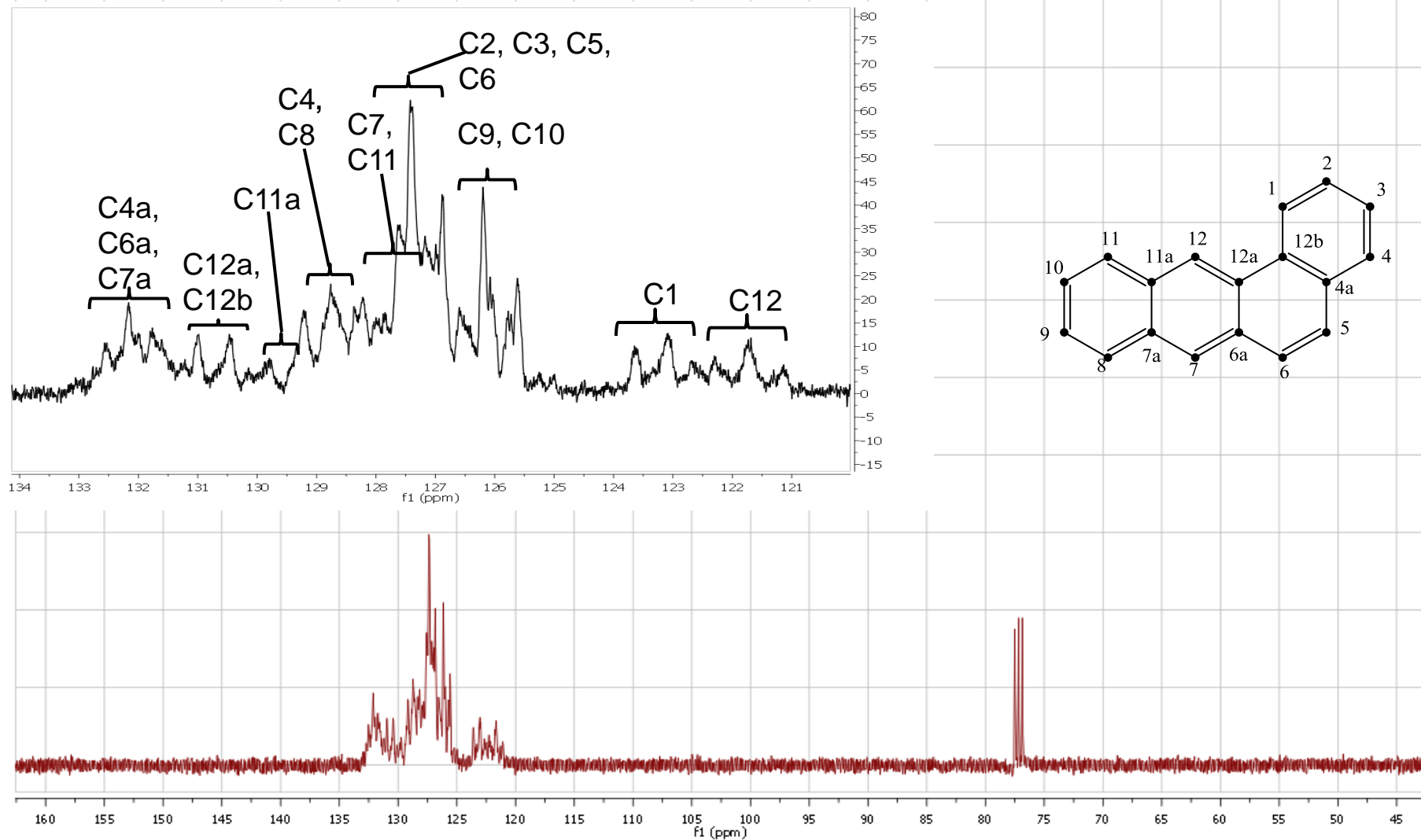


Figure S50. ^{13}C NMR (75 MHz, CDCl_3) of U- ^{13}C -benz[a]anthracene (17).

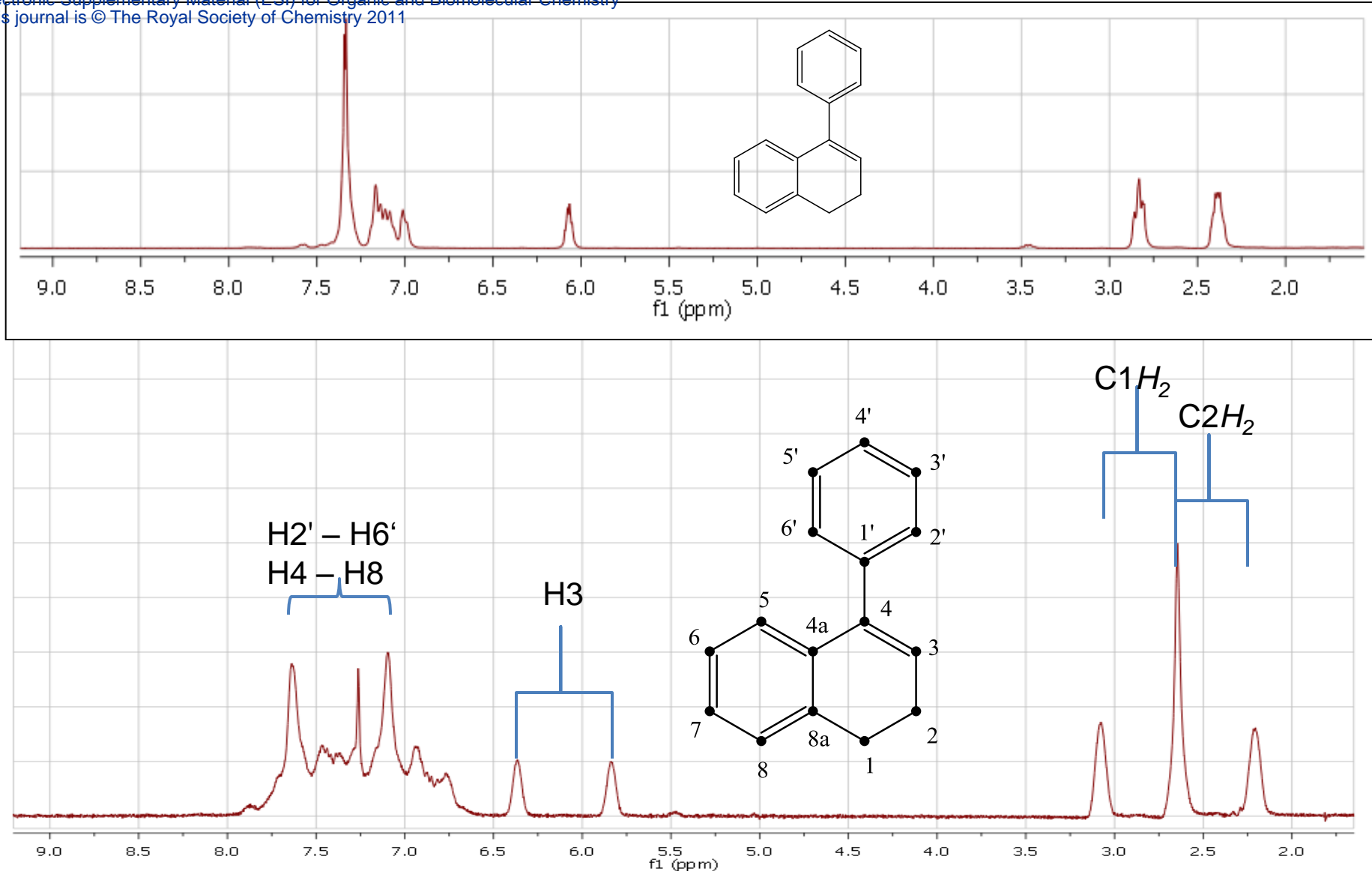


Figure S51. ¹H NMR (300 MHz, CDCl₃) of U-¹³C-4-phenyl-1,2-dihydronaphthalene (**21**). (Inset: ¹H NMR of NA-4-phenyl-1,2-dihydronaphthalene. Chemical shifts identical report: Cook, M. J.; Katritzky, A. R.; Pennington, F. C.; Semple, B. M. *J. Chem. Soc. B.* **1969**, 523.)

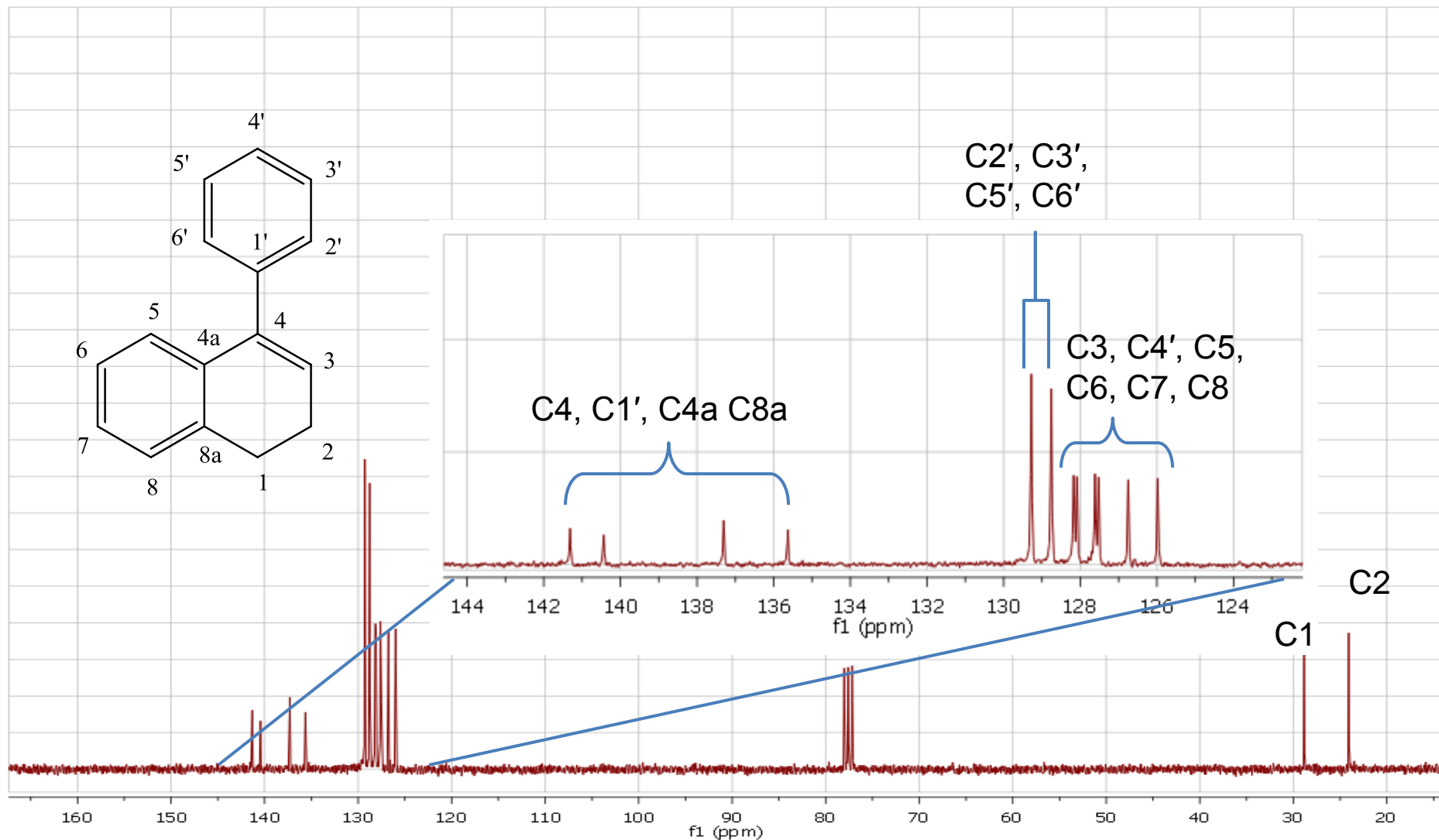


Figure S52. ^{13}C NMR (75 MHz, CDCl_3) of NA-4-phenyl-1,2-dihydronaphthalene. (Chemical shifts identical to report: Rao, M. L. N.; Jadhav, Deepak N.; Venkatesh, Varadhachari *Eur. J. Org. Chem.* **2009**, 4300.)

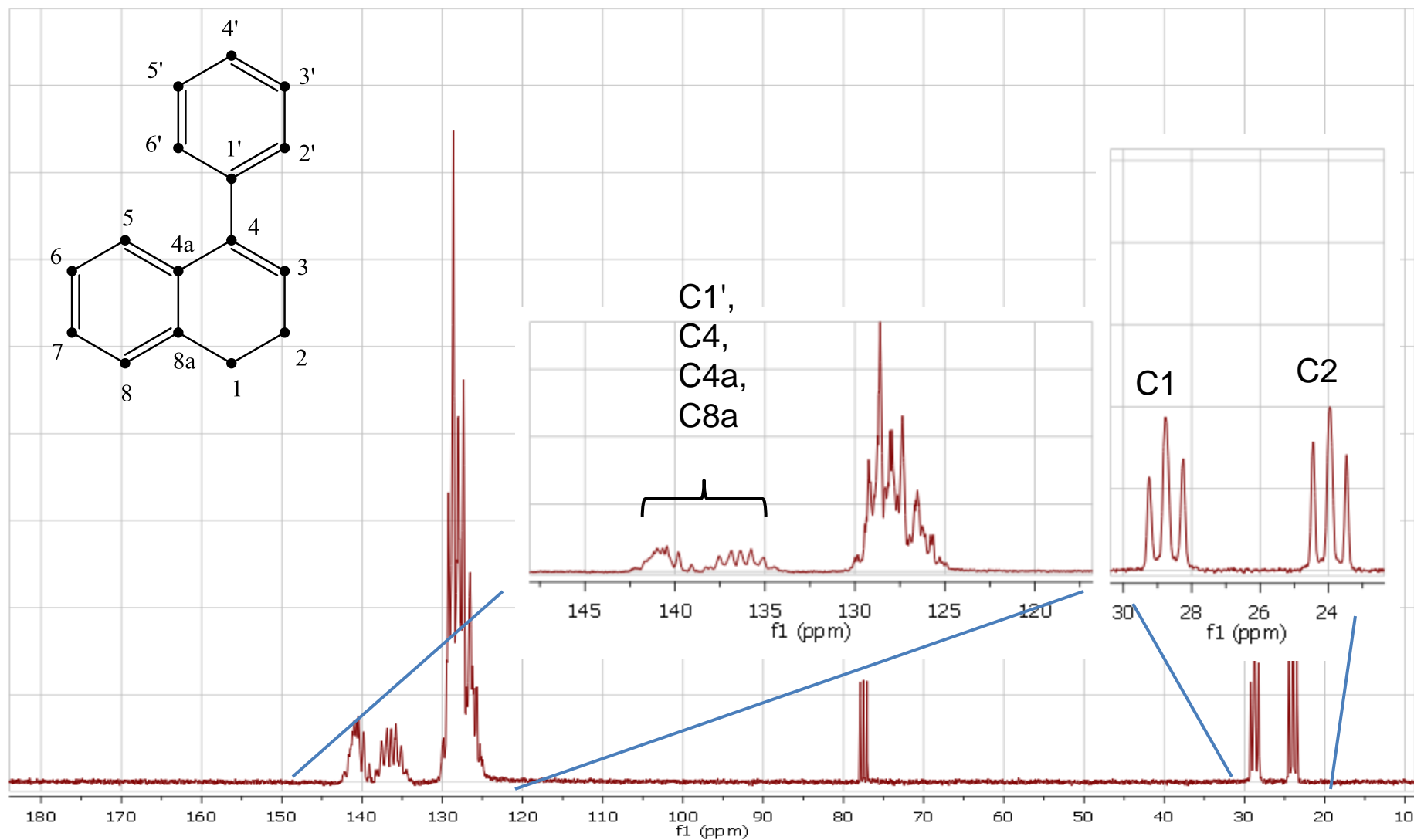


Figure S53. ^{13}C NMR (75 MHz, CDCl_3) of U- ^{13}C -4-phenyl-1,2-dihydronaphthalene (**21**).

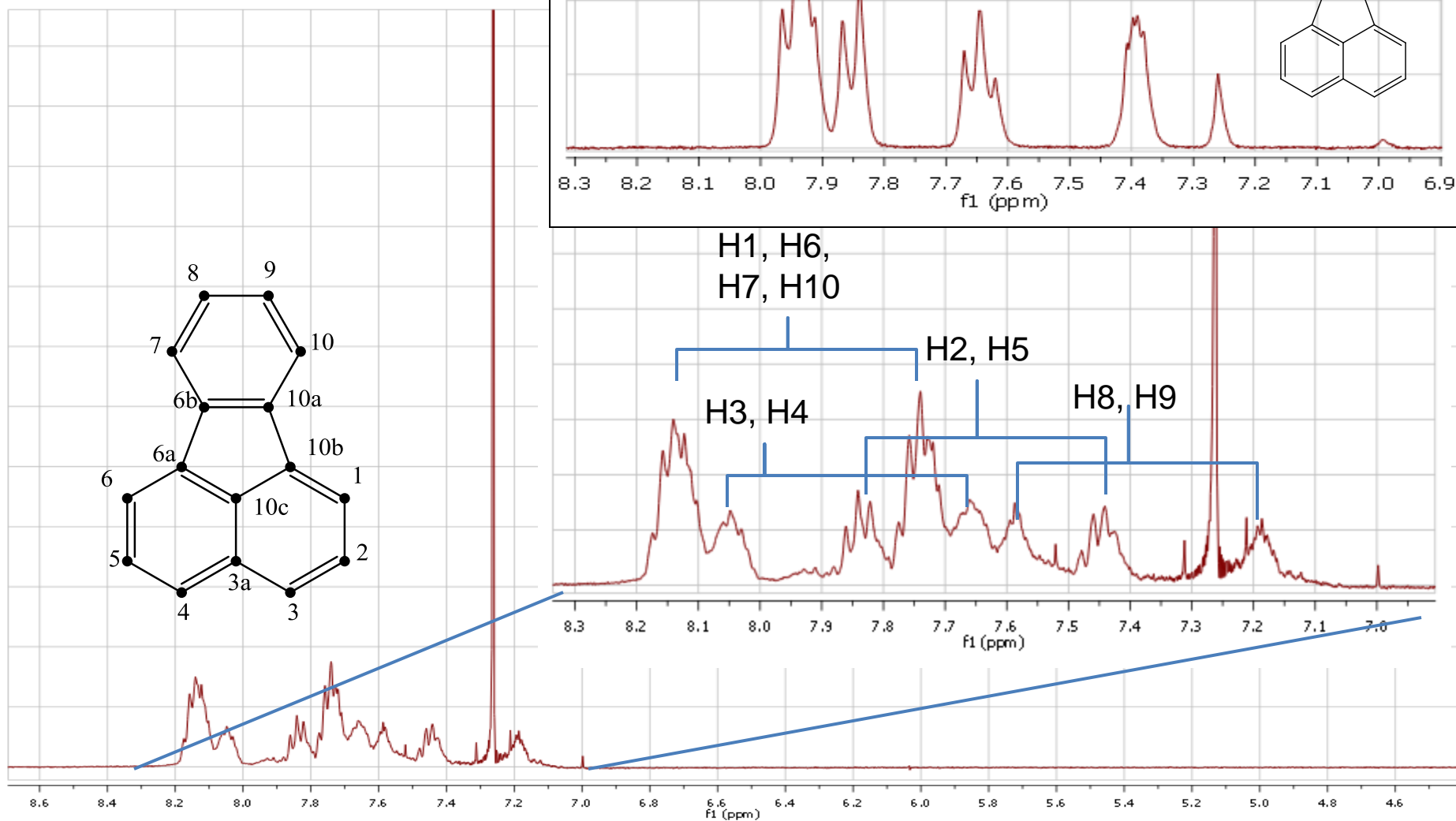


Figure S55. ¹H NMR (300 MHz, CDCl₃) of U-¹³C-fluoranthene (**23**). (Inset: ¹H NMR of NA-fluoranthene. Chemical shifts identical to report with assignments : Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem.* **2005**, *43*, 588.)

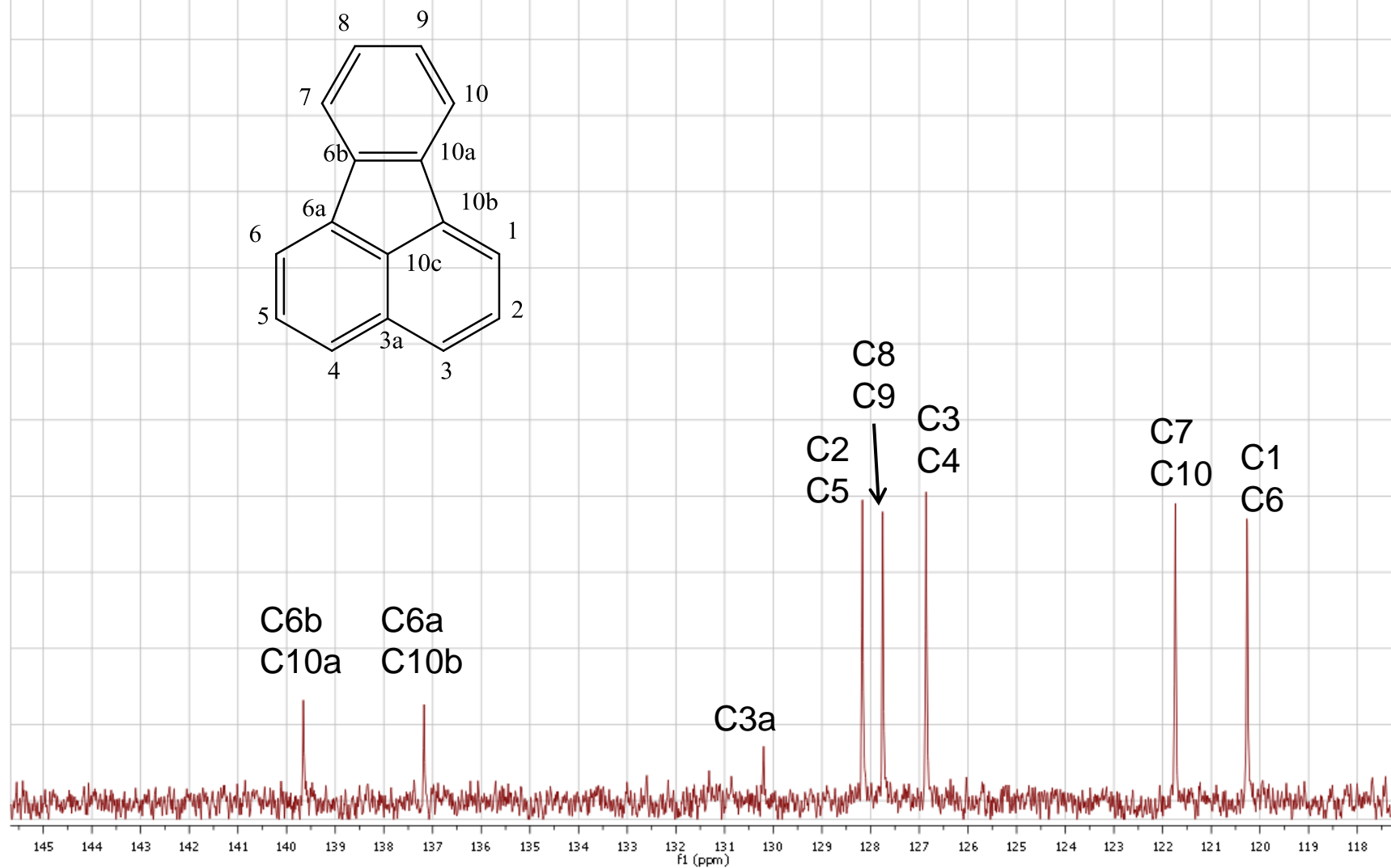


Figure S54. ¹³C NMR (100 MHz, CDCl₃) of NA-fluoranthene. (Chemical shifts identical report with assignments : Lutnaes, B. F.; Luthe, G. Brinkman, U. A. T.; Johansen, J. E.; Krane, J. *Magn. Reson. Chem.* **2005**, *43*, 588.)

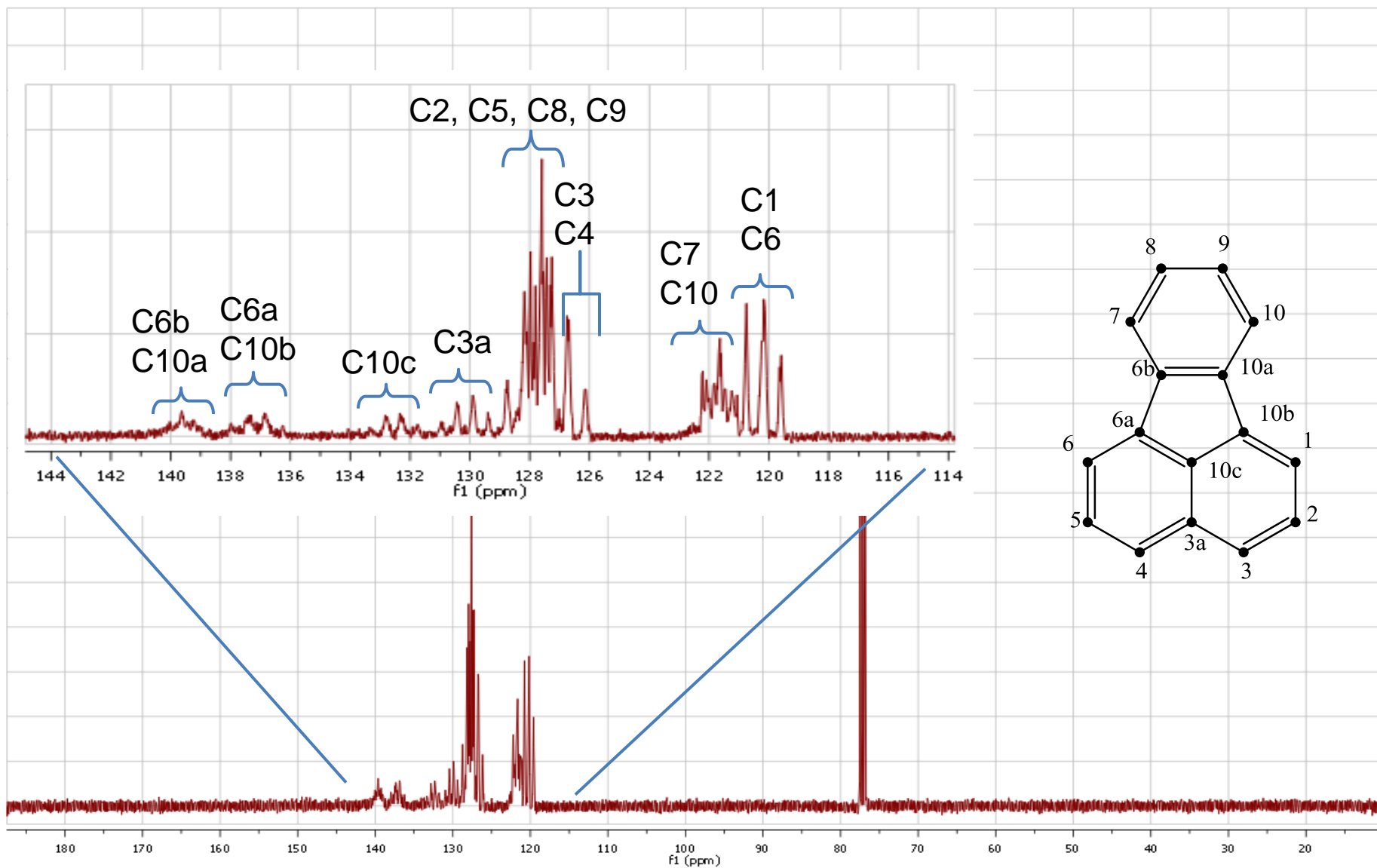


Figure S56. ^{13}C NMR (75 MHz, CDCl_3) of U- ^{13}C -fluoranthene (23).