Supplementary Information for

Bis-cyclometallated Ir(III) complexes containing 2-(1*H*-pyrazol-3-yl)pyridine ligands; influence of substituents and cyclometallating ligands on response to changes in pH.

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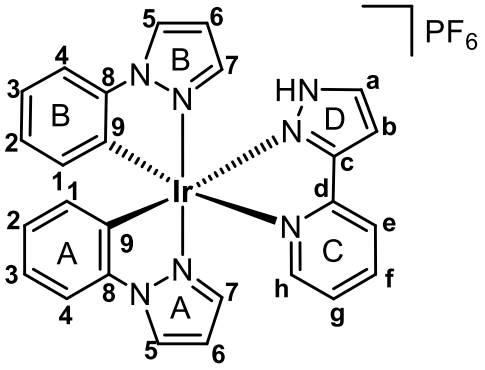
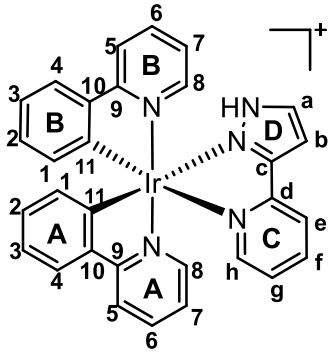
NMR labelling for iridium complexes





(i)

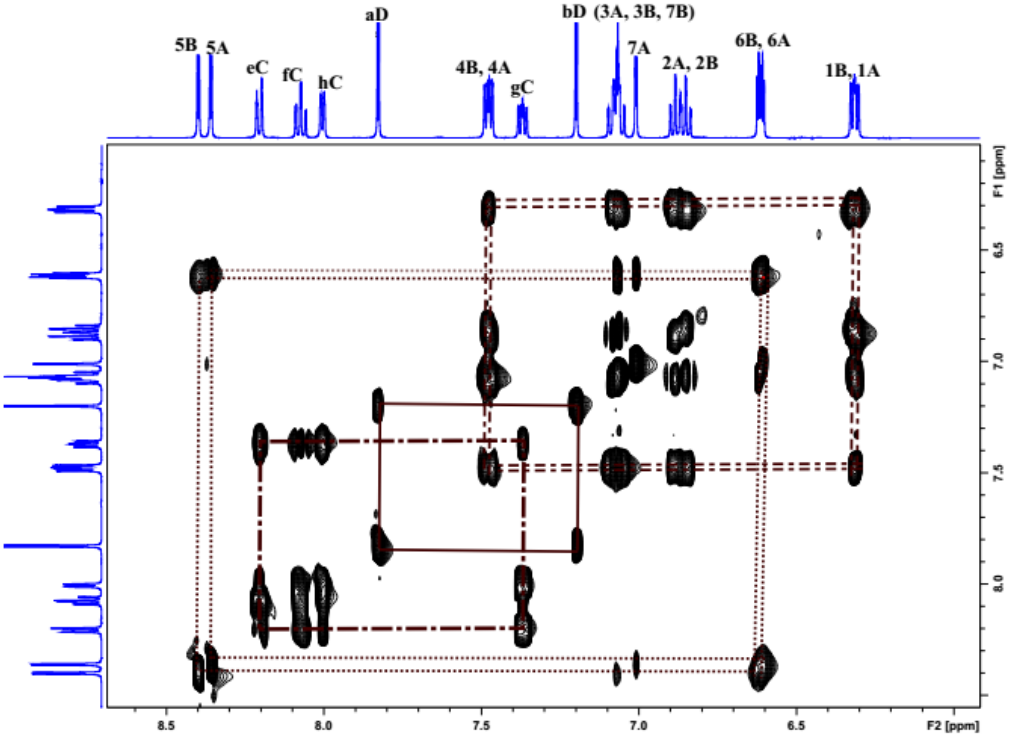


Fig. S1: Region of the TOCSY spectrum of **2aHL1** showing identification of two cyclometallated phenyls A, B **(– – – – –)**, two pyrazoles A, B (·····), one pyridine C (**- — — - —**) and the other pyrazole D (**———**) ring, (500 MHz, 298 K in CD_3CN).

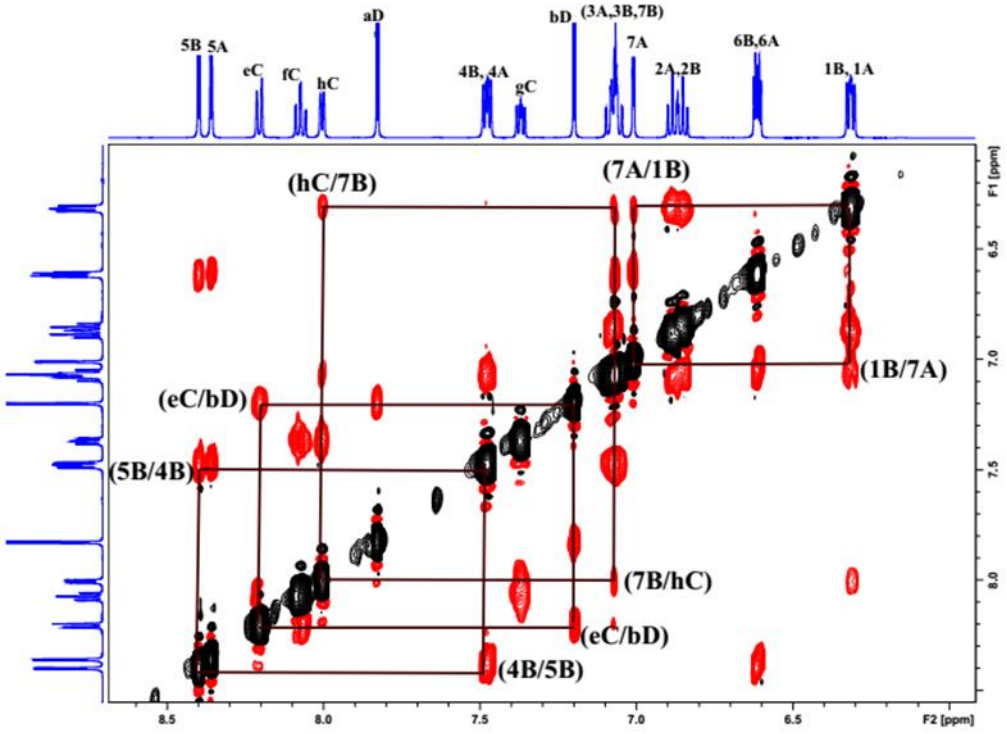


Fig. S2: NOESY spectrum of **2aHL$_1$** showing some key NOEs (500 MHz, 298 K in CD_3CN).

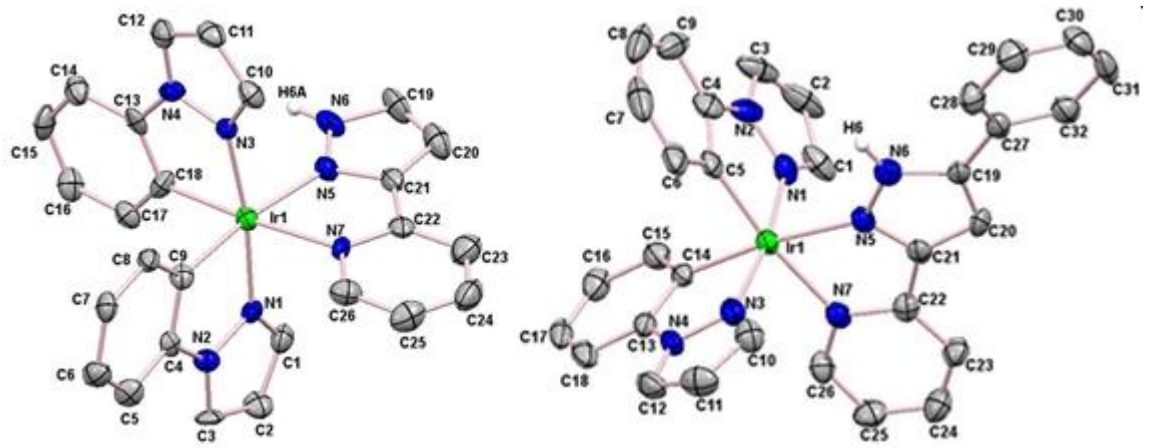


Fig S3 X-ray crystal structure of the cations of **2aHL$_1$** and **2aHL$_3$** showing 50% ellipsoids, the hydrogens (except NH) have been omitted for clarity.

Table S1: Selected bond lengths (**Å**) and angles (°) for **2aHL$_1$**, **2aHL$_2$, 2aHL$_3$** and **3aL$_2$**.

(Å)	**2aHL$_1$**	**2aHL$_2$**	**2aHL$_3$**	**3aL$_2$**
Ir(1)—N(1)	2.036(7)	2.033(10)	2.014(5)[a]	2.025(6)
Ir(1)—N(3)	2.031(6)	2.033(9)	2.005(6)[b]	2.015(7)
Ir(1)—N(5)	2.116(7)	2.175(8)	2.121(5)	2.122(6)
Ir(1)—N(7)	2.148(6)	2.160(8)	2.148(5)	2.148(6)
Ir(1)—C(9)	2.024(8)	1.989(10)	2.015(6)[c]	2.022(7)
Ir(1)—C(18)	2.039(8)	2.005(10)	2.008(7)[d]	1.992(8)
(°)				
N(1)—Ir(1)—C(9)	80.2(3)	79.8(4)	80.1(2)[e]	80.9(3)
N(3)—Ir(1)—C(18)	80.3(3)	79.6(4)	80.3(3)[f]	80.0(3)
N(5)—Ir(1)—N(7)	75.1(3)	75.7(3)	75.7(2)	76.8(2)

Note the labelling for **2aHL$_3$** is different the bond lengths shown are those that correspond to the ones in the other structures the actual labels are [a] labelled Ir(1)—N(3), [b] labelled Ir(1)—N(1), [c] labelled Ir(1)—C(14), [d] labelled Ir(1)—C(5), [e] labelled N(3)—Ir(1)—C(14), and [f] labelled N(1)—Ir(1)—C(5)

Fig. S4 X-Ray crystal structures for the cations of **2cHL$_{1\text{-}2}$** showing 50% ellipsoids. All hydrogen atoms (except NH) have been omitted for clarity.

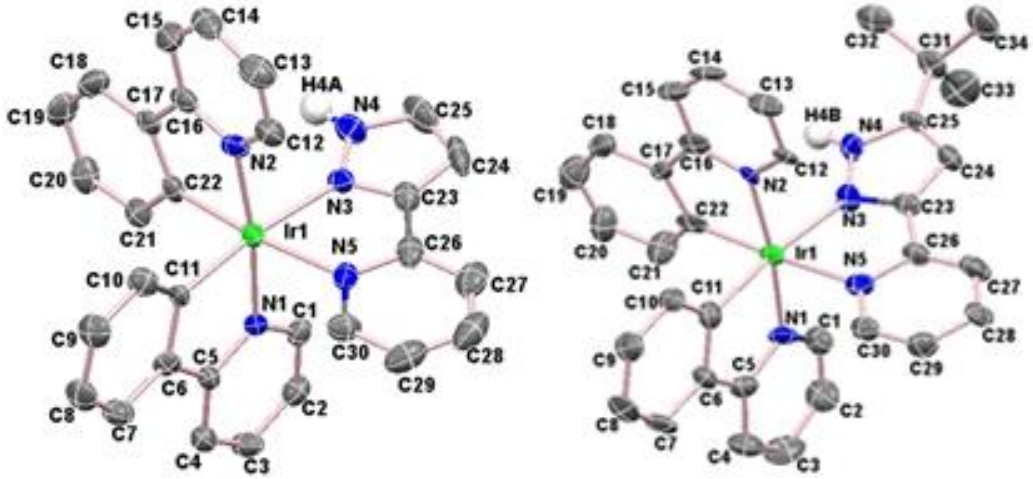


Table S2: Selected bond lengths (**Å**) and angles (°) for **2cHL$_1$**, **2cHL$_2$**, **2cHL$_3$** and **3cL$_3$**.

(Å)	**2cHL$_1$**	**2cHL$_2$[a]**	**2cHL$_3$**	**3cL$_3$**
Ir(1)—N(1)	2.052(5)	2.031(10)	2.056(4)	2.022(7)
Ir(1)—N(2)	2.036(5)	2.050(10)	2.062(4)	2.033(7)
Ir(1)—N(3)	2.119(5)	2.150(9)	2.149(4)	2.088(7)
Ir(1)—N(5)	2.159(6)	2.173(10)	2.170(4)	2.147(8)
Ir(1)—C(11)	2.009(6)	2.026(12)	2.014(5)	2.021(10)
Ir(1)—C(22)	2.006(7)	2.002(13)	2.010(5)	1.999(9)
(°)				
N(1)—Ir(1)—C(11)	80.2(2)	80.5(4)	81.0(2)	81.5(4)
N(2)—Ir(1)—C(22)	80.5(3)	80.7(4)	81.2(2)	81.6(4)
N(3)—Ir(1)—N(5)	75.1(2)	75.7(4)	74.8(2)	75.6(3)

a Average of two independent molecules in the unit cell

Photophysical properties

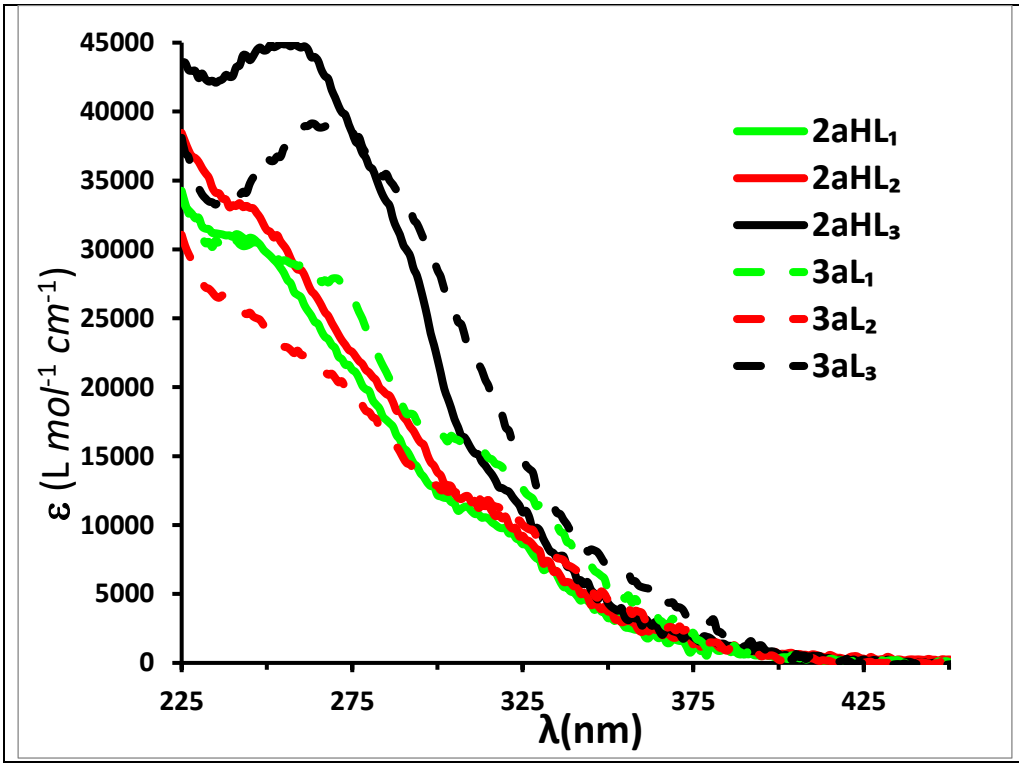


Fig. S5: Absorption spectra of cationic and neutral complexes **2aHL**$_{1\text{-}3}$ **(—)** and **3aL**$_{1\text{-}3}$**(---)**, respectively, in MeCN at 0.02 mM at room temperature.

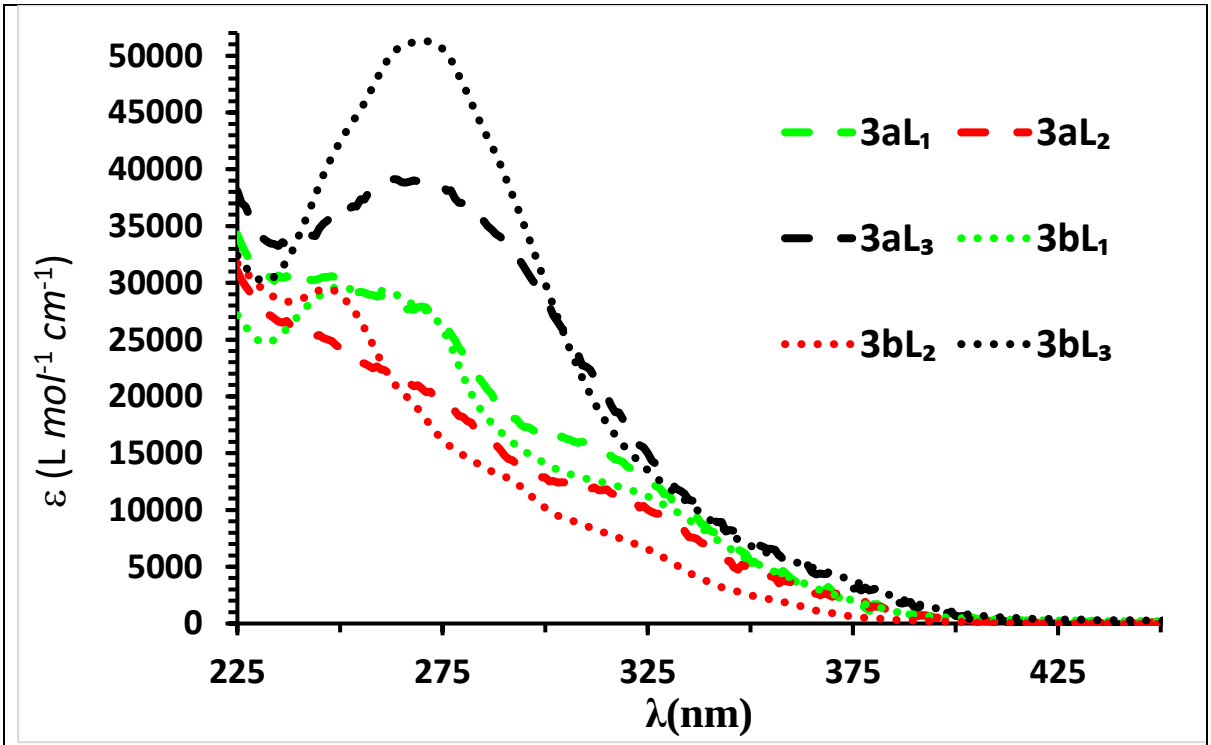


Fig. S6: Absorption spectra of neutral complexes **3aL**$_{1\text{-}3}$ **(---)** and **3bL**$_{1\text{-}3}$ **(…)** in MeCN at 0.02 mM at room temperature

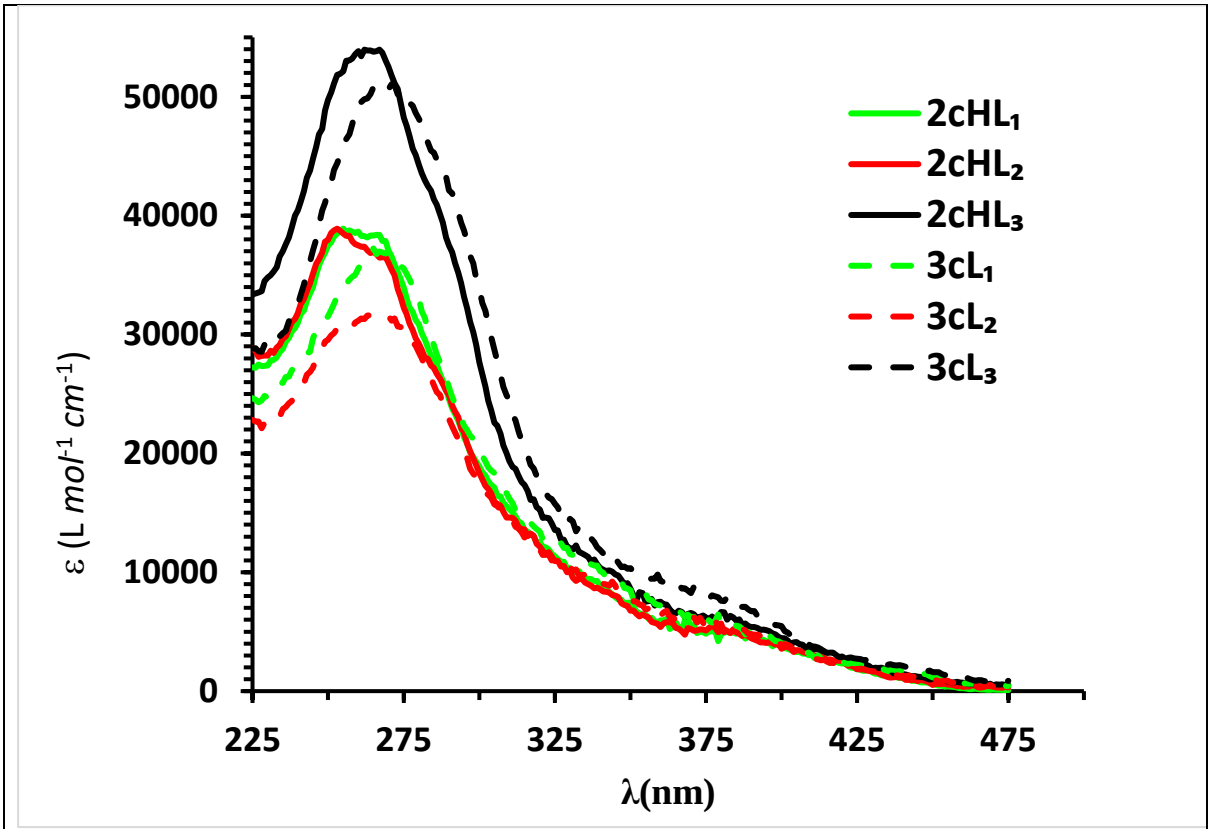


Fig. S7: Absorption spectra of cationic and neutral complexes **2cHL**$_{1\text{-}3}$ **(—)** and **3cL**$_{1\text{-}3}$ **(- - -)**, respectively, in MeCN at 0.02 mM at room temperature.

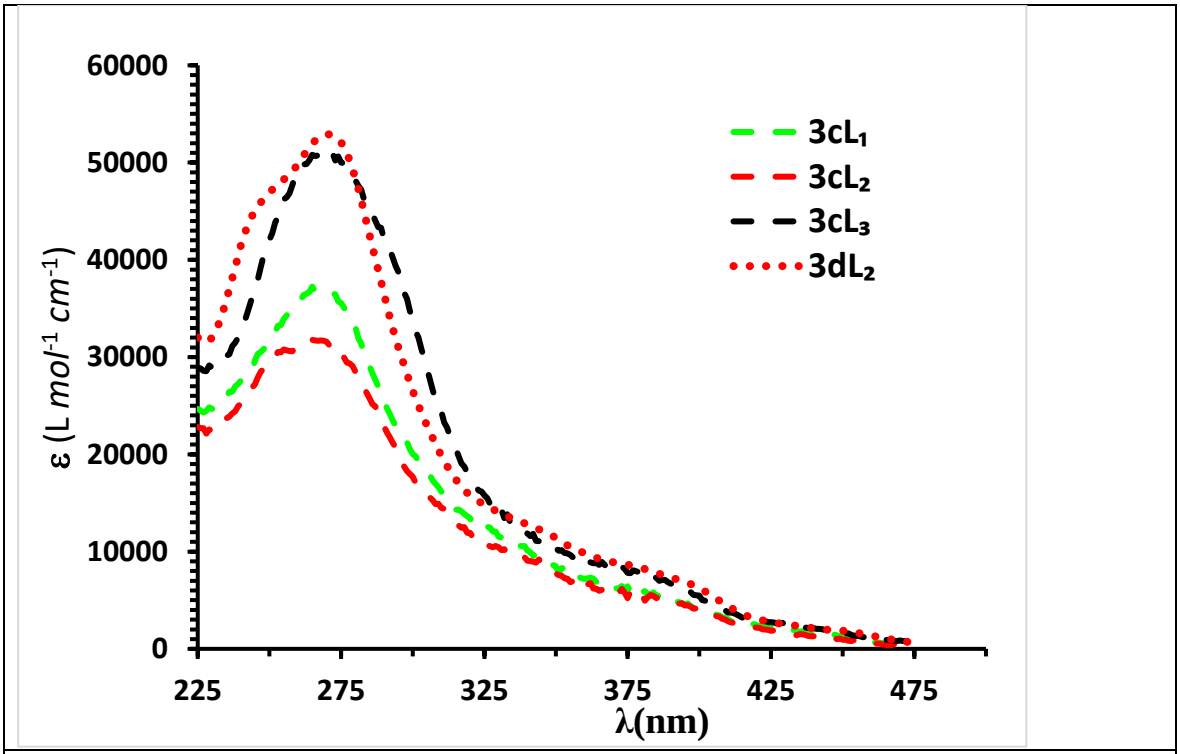


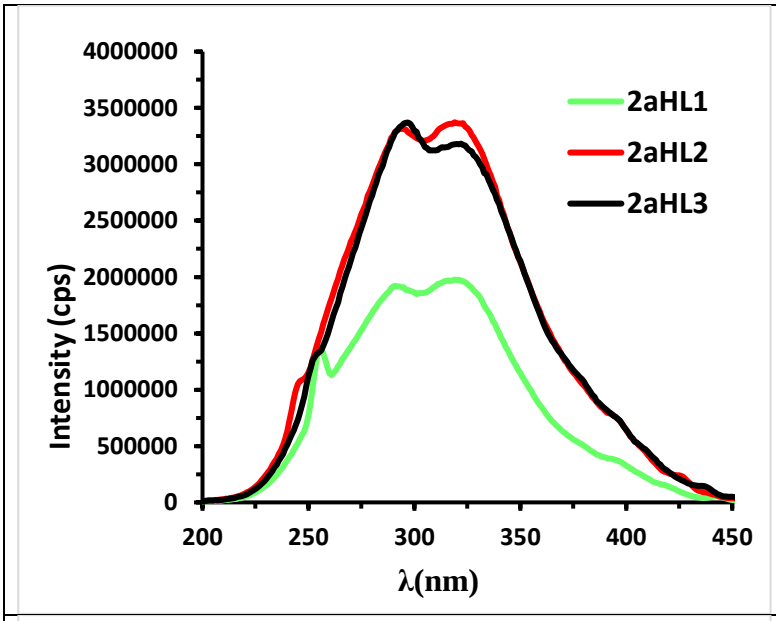
Fig. S8: Absorption spectra of neutral **3cL**$_{1\text{-}3}$ (– – –) and **3dL**$_2$ (….) respectively, in MeCN at 0.02 mM at room temperature

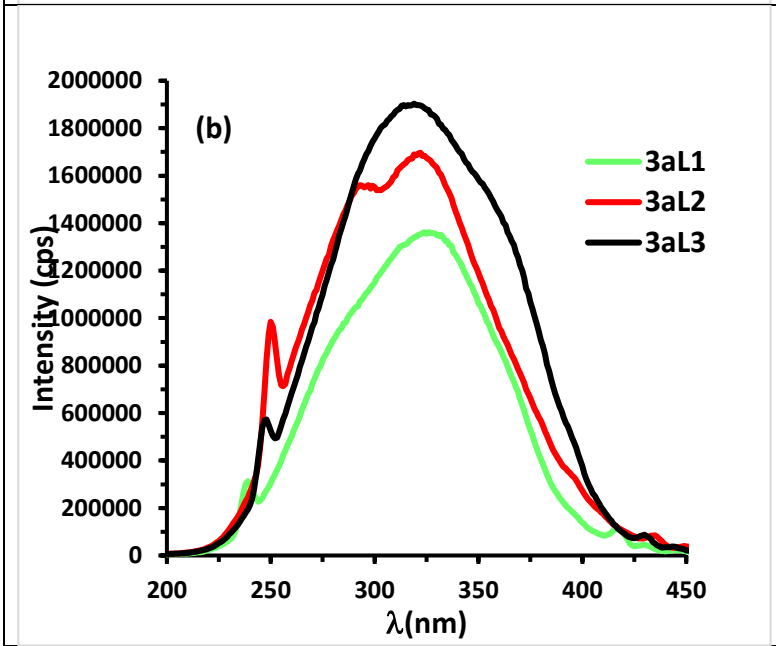
Table S3 Electronic absorption spectral data [λ_{abs} (nm), ε_{max} (L mol^{-1} cm^{-1})] of complexes **2aHL$_{1-3}$, 3aL$_{1-3}$**, **3bL$_{1-3}$**, **2cHL$_{1-3}$, 3cL$_{1-3}$** and **3dL$_2$**.

	L_1	L_2	L_3
2a	241(31100), 324(8900)	242(33400), 321(10000)	254(44900) 320(12500)
3a	270 sh(27900), 324(13100)	270 sh(27900), 321(10800)	268(39100) 320(17100)
3b	250(29700), 324(11400)	249(29300), 328(6000)	270(51300), 367(4700)
2c	255(38900), 340 sh (8870)	253(38900), 341 sh (8370)	267(54000), 368 sh (6240)
3c	267(37000), 340sh(10100)	265(31800), 341sh (9030)	271(51100), 368 sh (8650)
3d		273(52700), 399 sh (6400)	

Table S4 Emission data (λ_{max} nm) of complexes **2a/cHL$_{1-3}$**, and **3a/dL$_{1-3}$**.

	L_1	L_2	L_3
2a	502	495	508
3a	475	493	496
3b	445 sh, 467	433sh, 458	483
2c	505	484sh, 510	497, 520sh
3c	499, 517sh	506	497, 518sh
3d		502, 533sh	





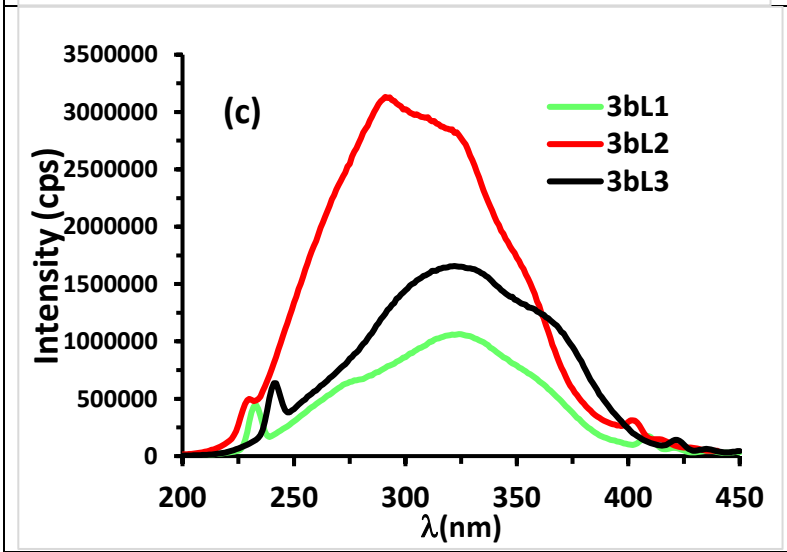
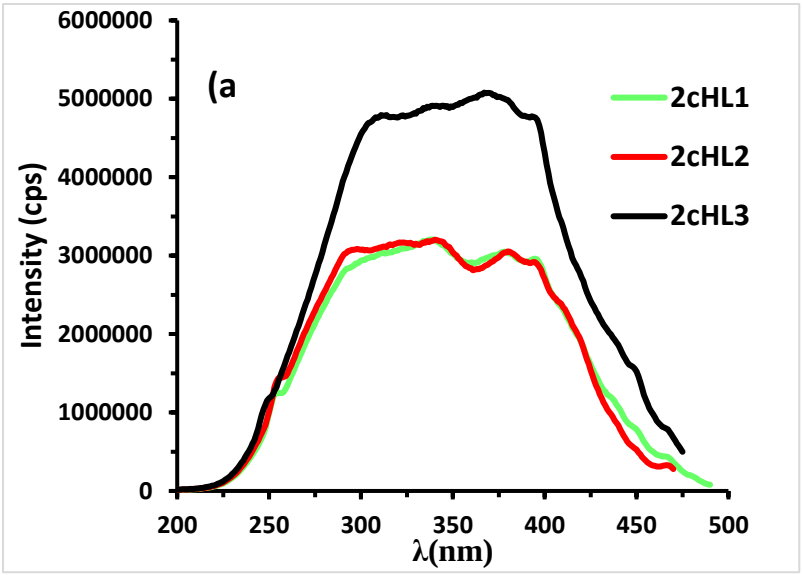
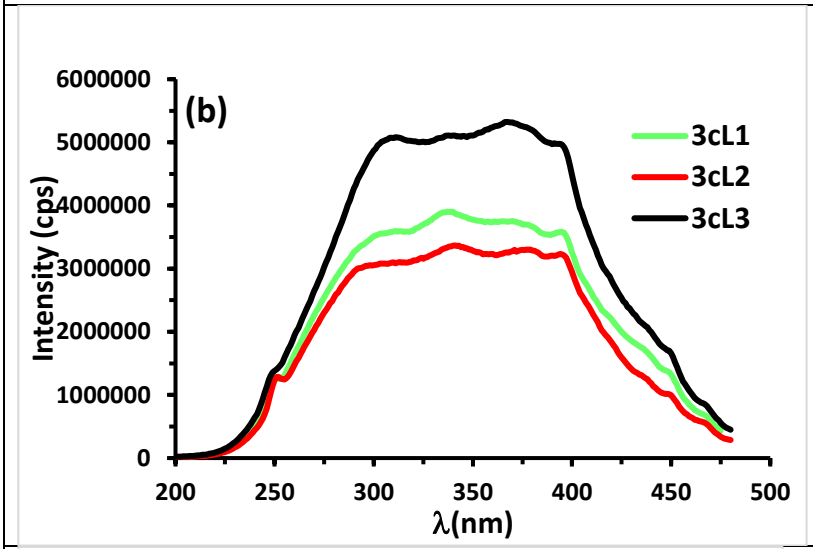


Fig. S9**:** Excitation spectra of **(a)** cationic ppz complexes **2aHL**$_{1\text{-}3}$, (**b**) neutral ppz complexes **3aL**$_{1\text{-}3}$, and **(c)** neutral ppz-CF_3 complexes **3bL**$_{1\text{-}3}$ in MeCN at 0.02 mM





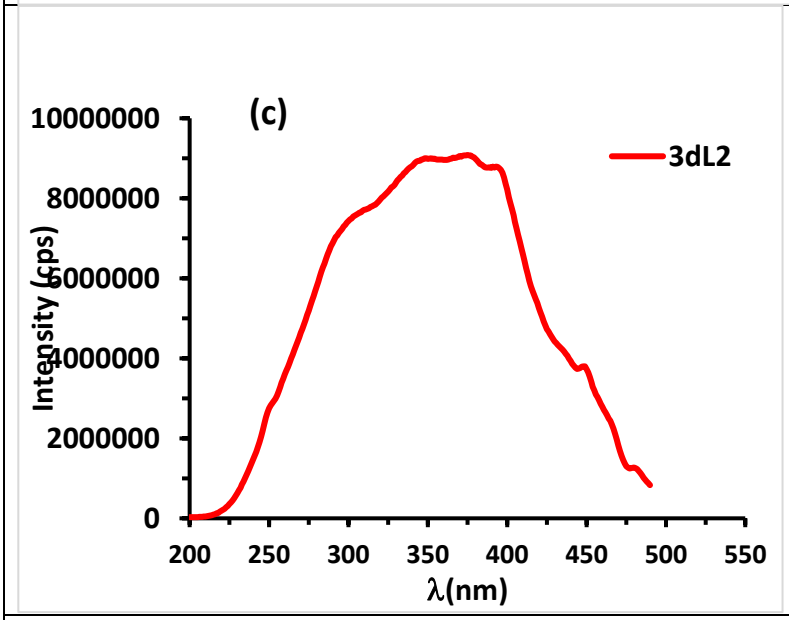


Fig. S10**:** Excitation spectra of **(a)** cationic ppy complexes **2cHL**$_{1\text{-}3}$, (**b**) neutral ppy complexes **3cL**$_{1\text{-}3}$, and **(c)** neutral ppy-CF_3 complex **3dL**$_2$ in MeCN at 0.02 mM

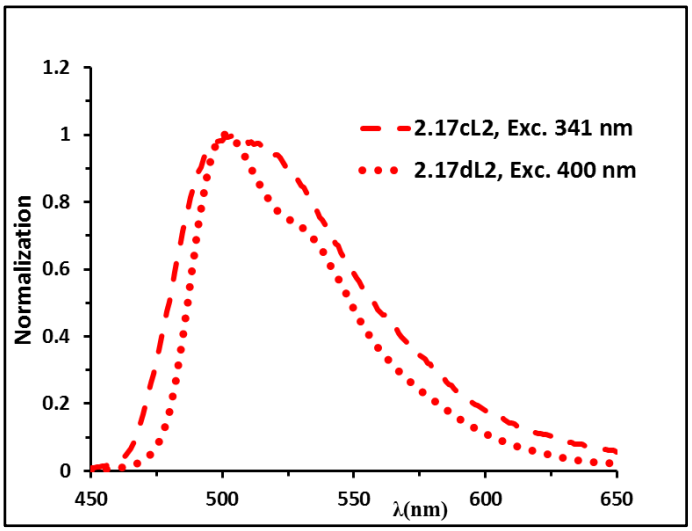


Fig. S11 Normalised emission spectra of neutral complexes **3cL$_2$ (- - -)** and **3dL$_2$ (….)**, respectively, in MeCN at 0.02 mM at room temperature in air.

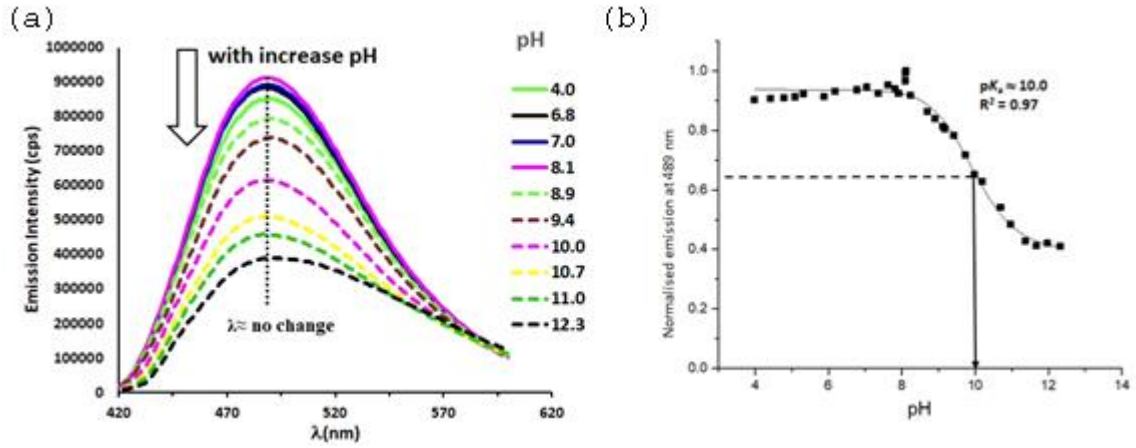


Fig S12 (a) Selected emission spectra of complexes **3aL$_2$** (0.02mM) at various pH values in MeCN/H_2O (1:9), in air with excitation at 324 nm; (b) Plot of normalised emission intensity of **3aL$_2$** against pH

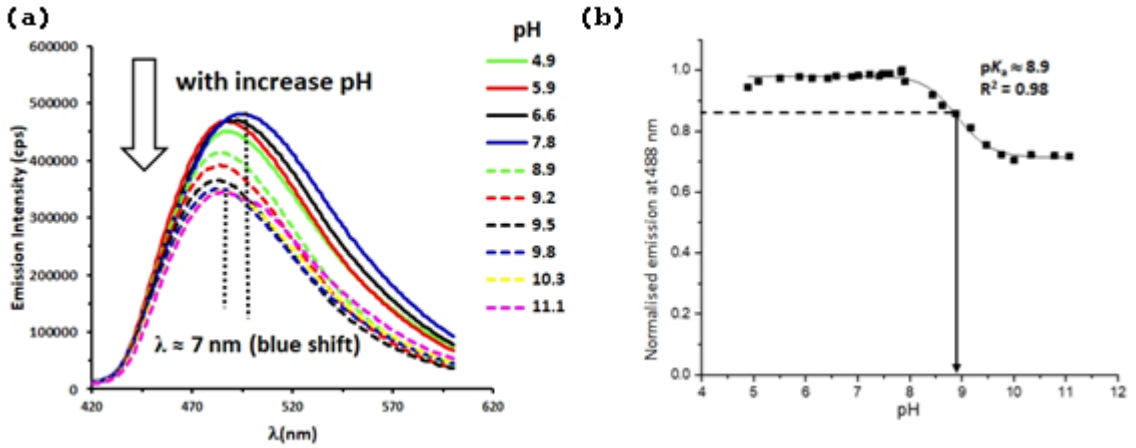


Fig S13 (a) Selected emission spectra of complexes **3aL$_3$** (0.02mM) at various pH values in MeCN/H_2O (1:9), in air with excitation at 324 nm; (b) Plot of normalised emission intensity of **3aL$_3$** against pH

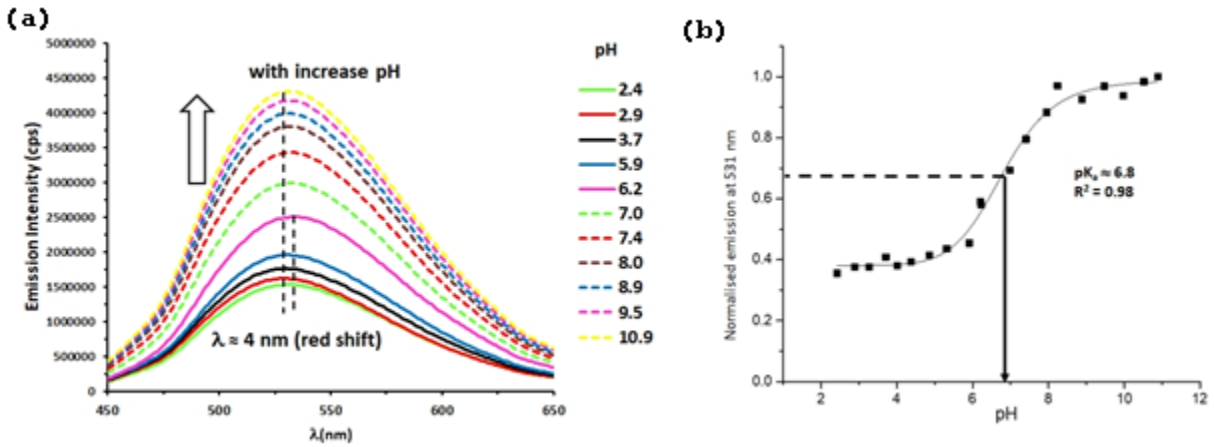


Fig S12 (a) Selected emission spectra of complexes **3bL$_1$** (0.02mM) at various pH values in MeCN/H_2O (1:9), in air with excitation at 324 nm; (b) Plot of normalised emission intensity of **3bL$_1$** against pH

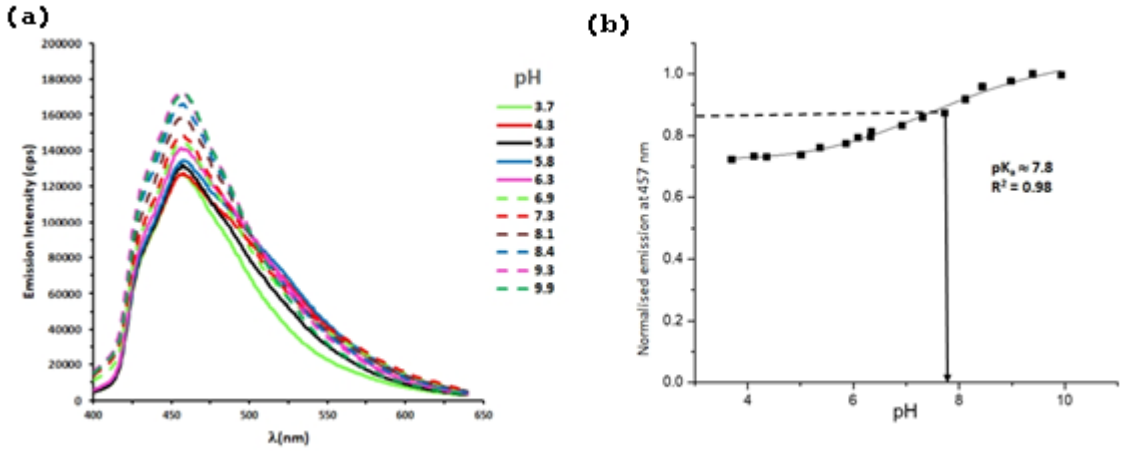


Fig S15 (a) Selected emission spectra of complexes **3bL$_2$** (0.02mM) at various pH values in MeCN/H_2O (1:9), in air with excitation at 324 nm. (b) Plot of normalised emission intensity of **3bL$_2$** against pH

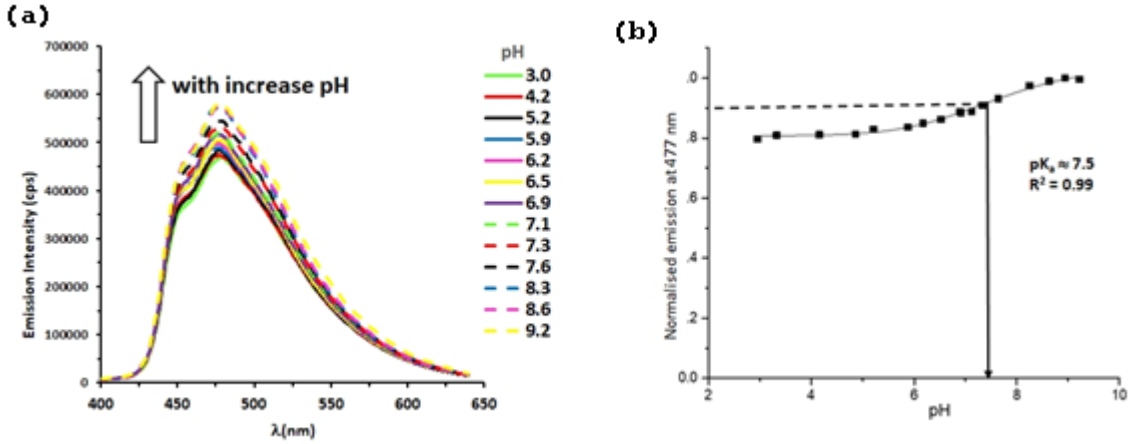


Fig S16 (a) Selected emission spectra of complexes **3bL$_3$** (0.02mM) at various pH values in MeCN/H_2O (1:9), in air with excitation at 324 nm. (b) Plot of normalised emission intensity of **3bL$_3$** against pH.

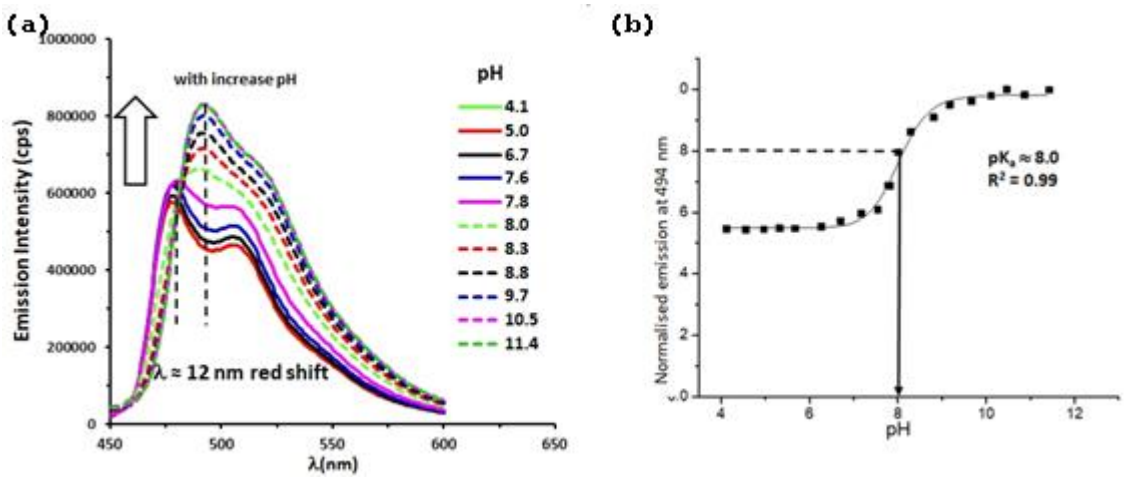


Fig S17 (a) Selected emission spectra of complexes **3cL$_2$** (0.02mM) at various pH values in MeCN/H_2O (1:9), in air with excitation at 324 nm. (b) Plot of normalised emission intensity of **3cL$_2$** against pH

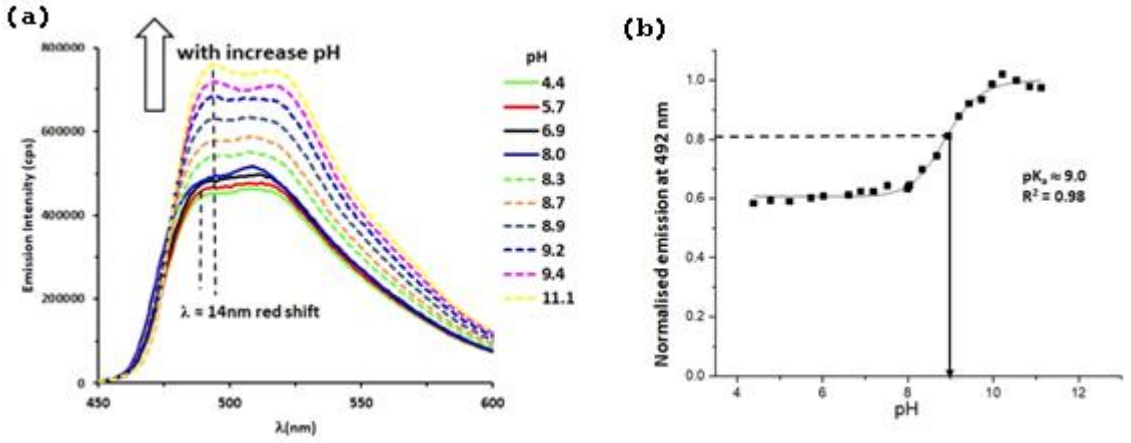


Fig S18 (a) Selected emission spectra of complexes **3cL$_3$** (0.02mM) at various pH values in MeCN/H_2O (1:9), in air with excitation at 324 nm. (b) Plot of normalised emission intensity of **3cL$_3$** against pH

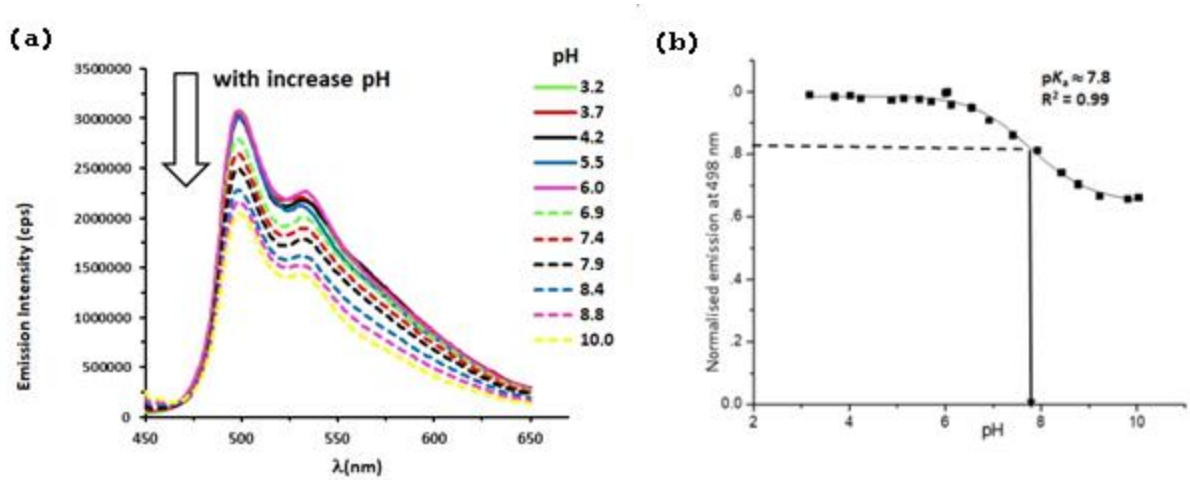
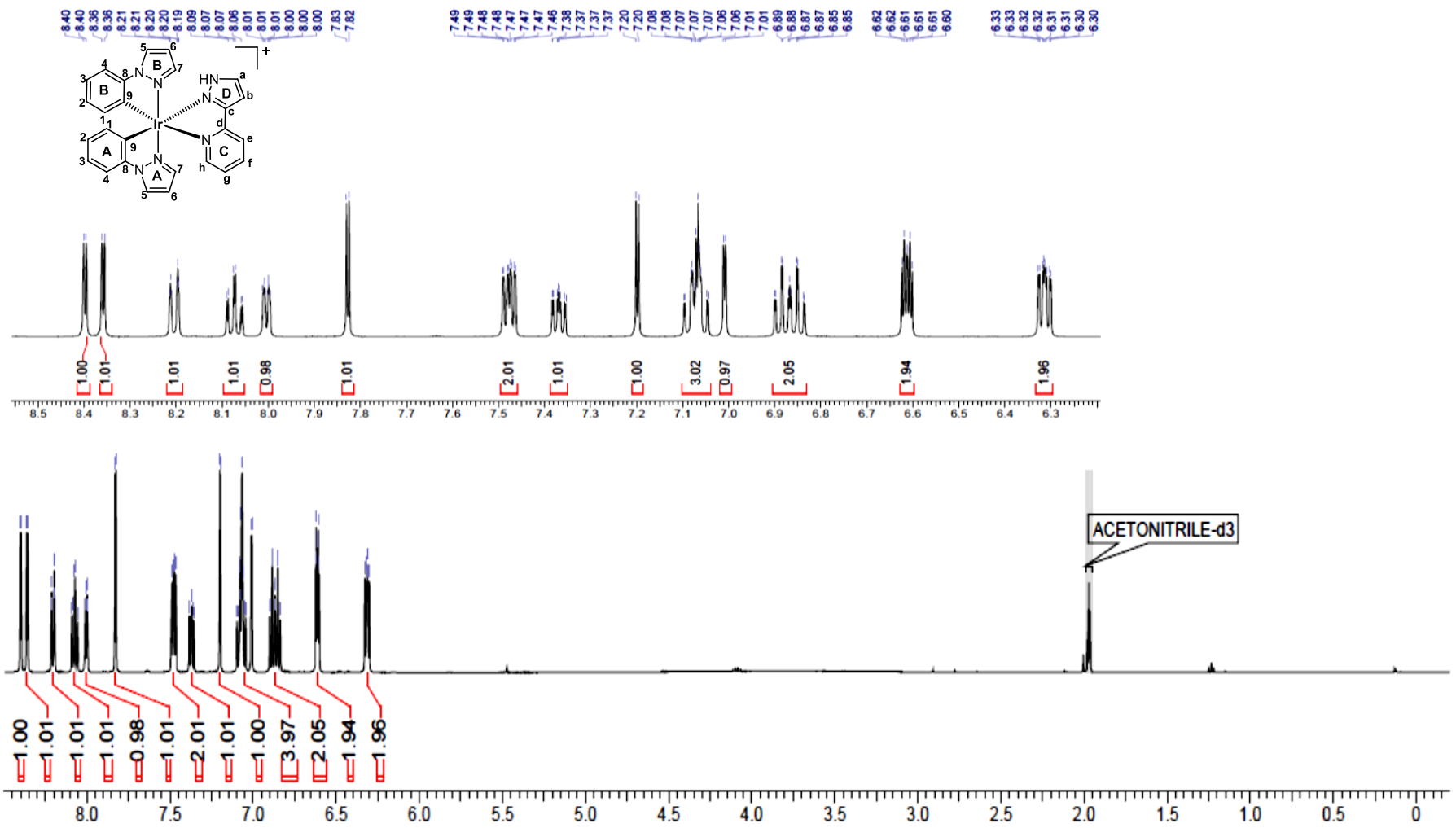
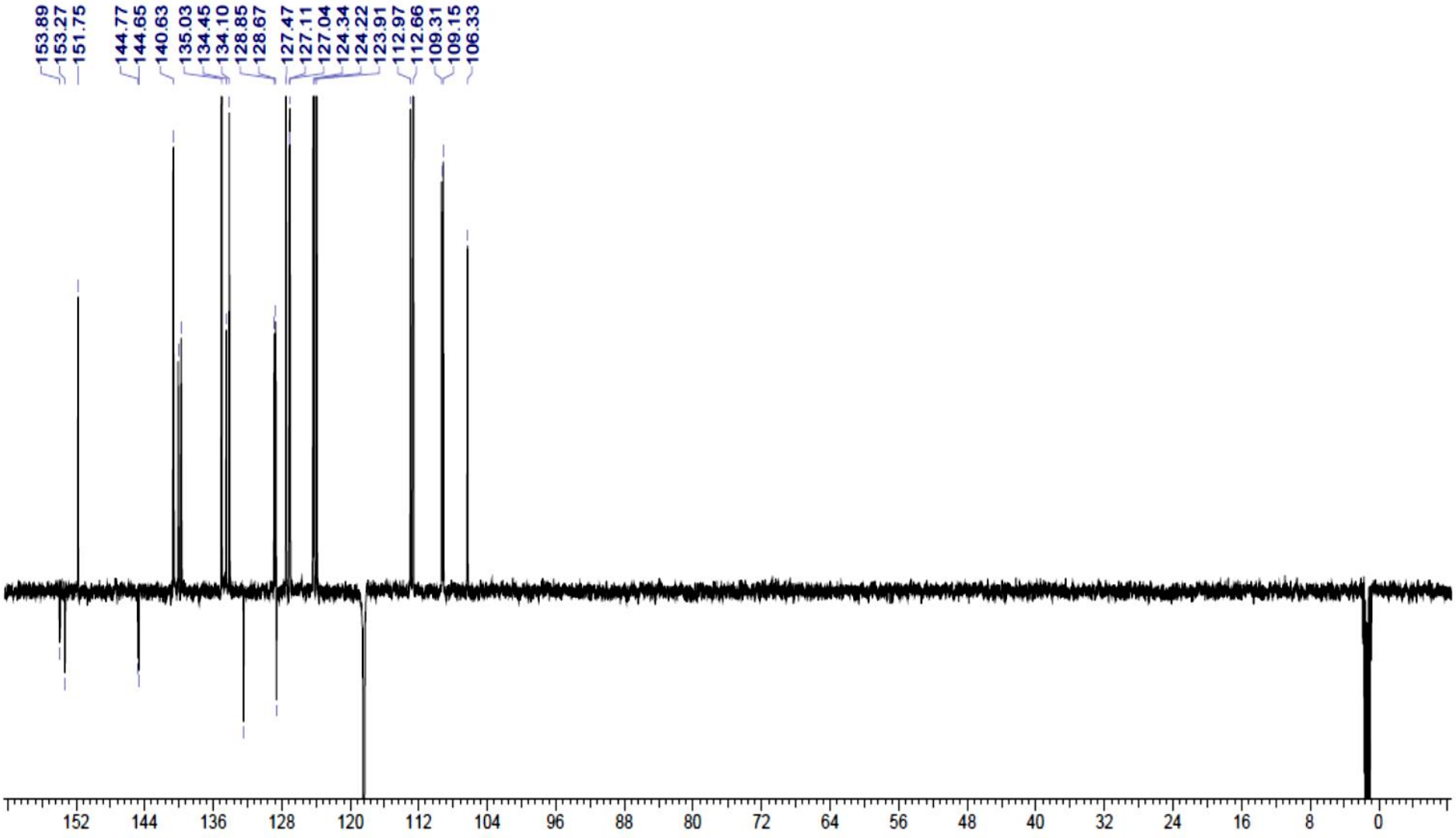


Fig S19 (a) Selected emission spectra of complexes **3dL$_2$** (0.02mM) at various pH values in MeCN/H_2O (1:9), in air with excitation at 324 nm. (b) Plot of normalised emission intensity of **3dL$_2$** against pH.

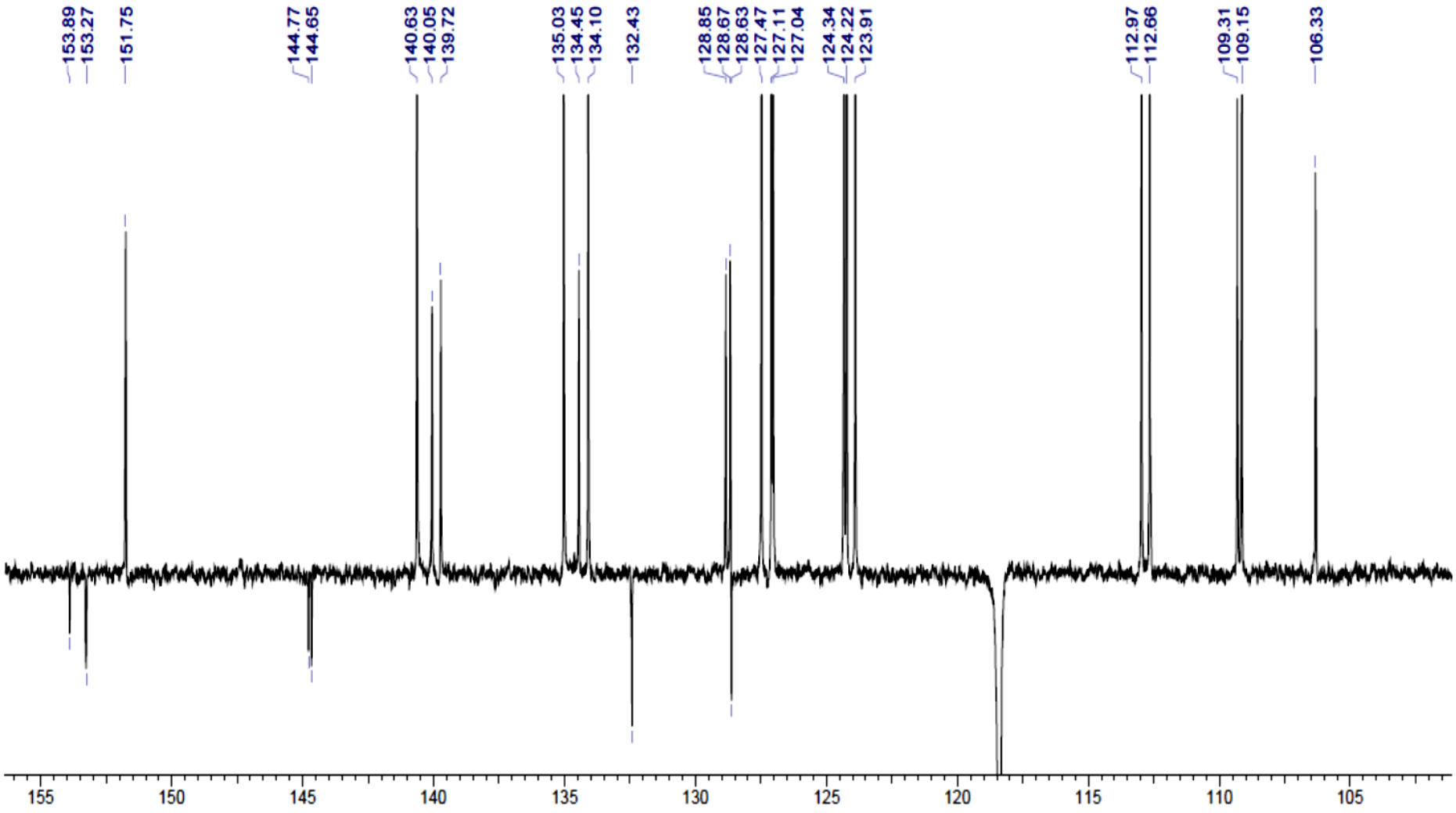
^{1}H NMR spectrum of **2aHL$_1$**



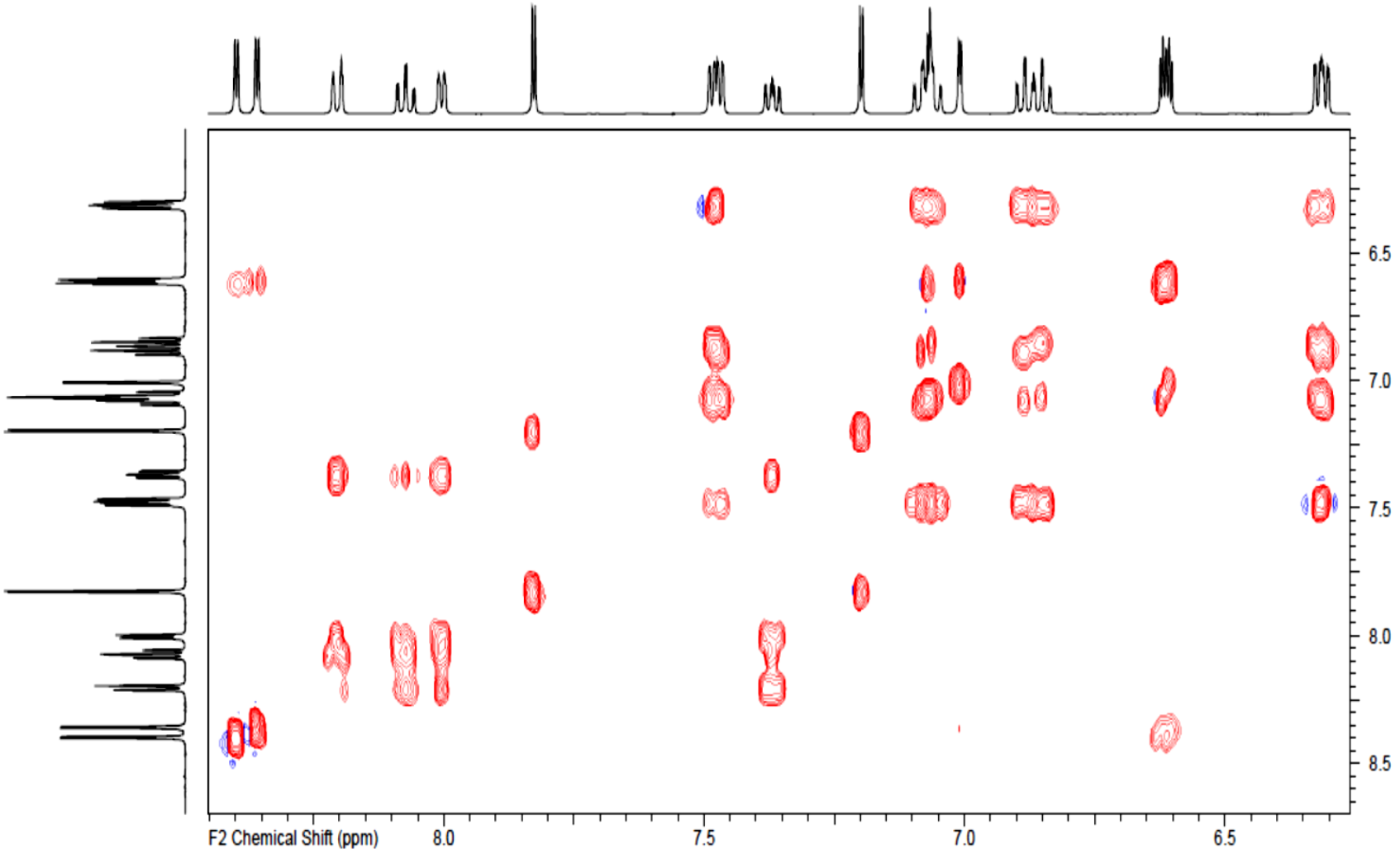
^{13}C NMR APT spectrum of **2aHL$_1$**



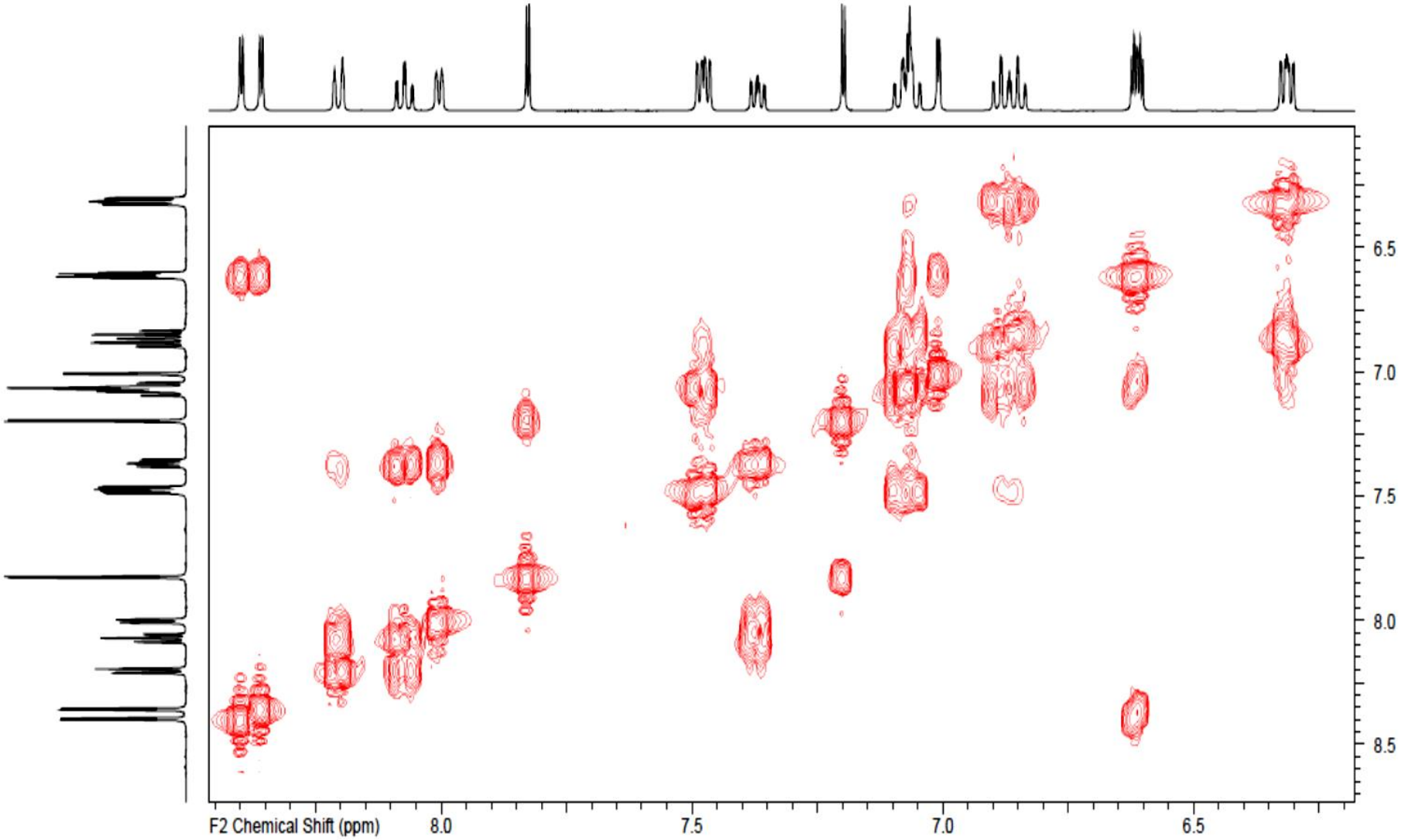
Section of the ^{13}**C APT** spectrum of **2aHL$_1$**



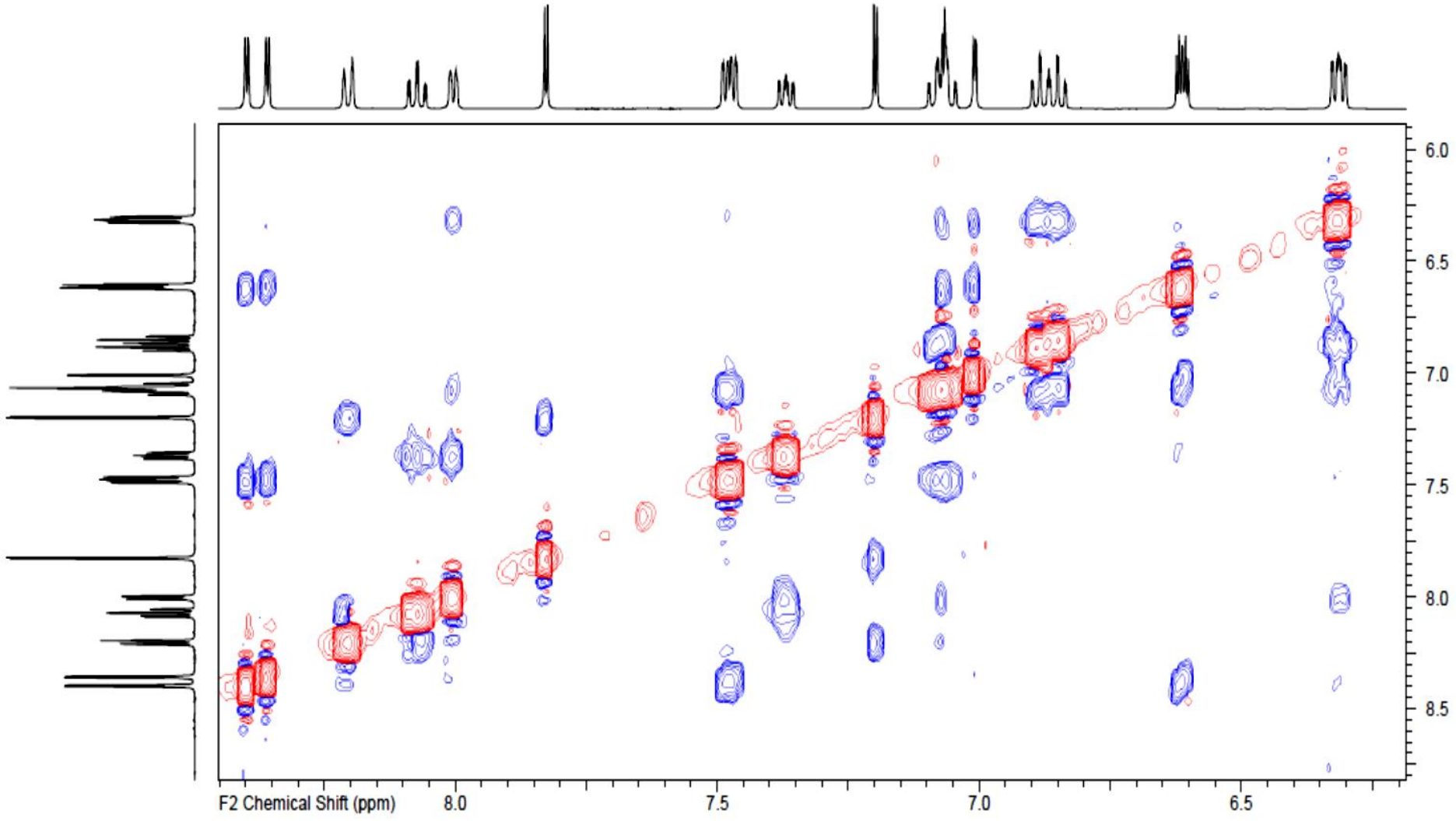
Section of the **TOCSY** spectrum of **2aHL$_1$**



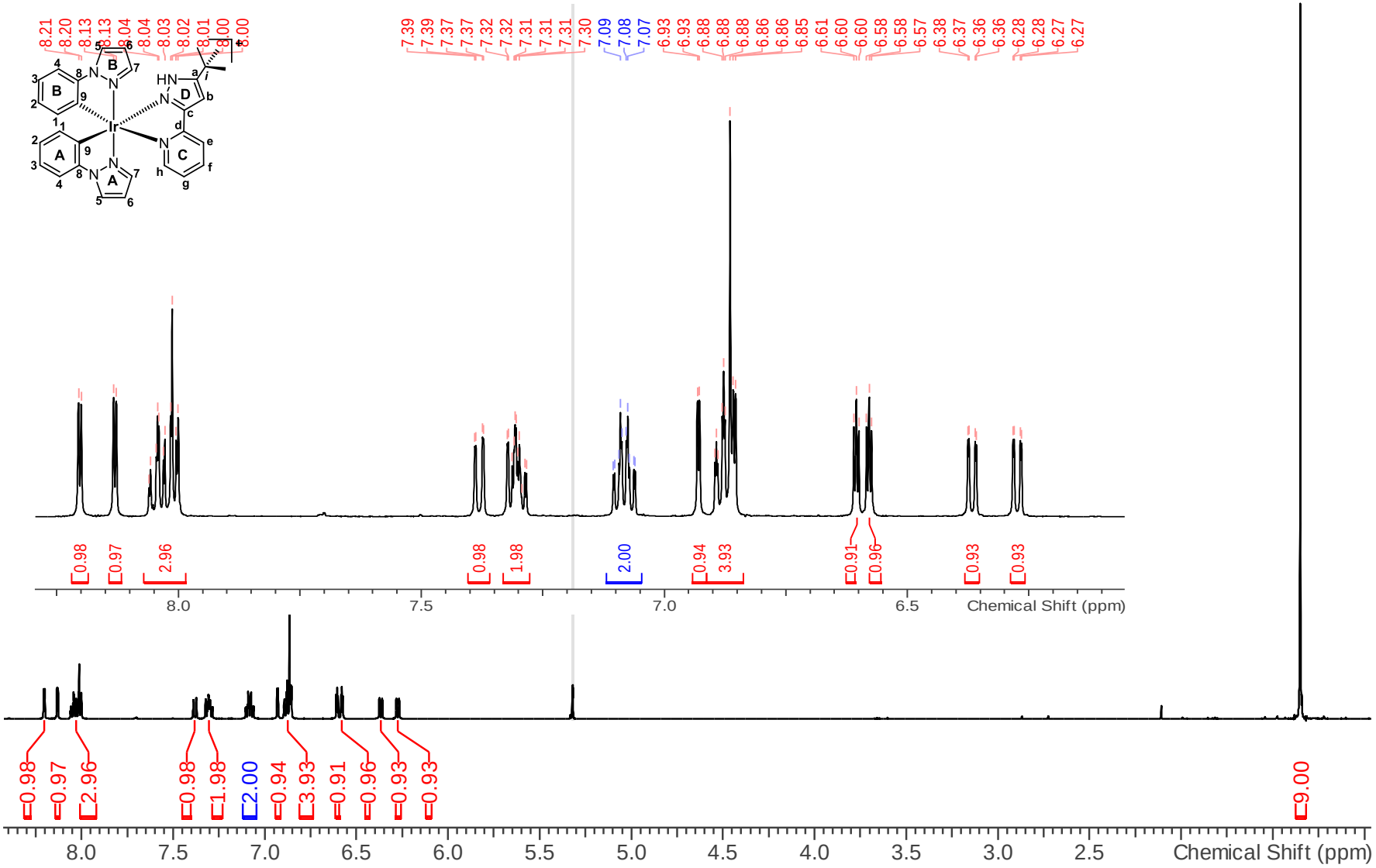
Section of the **COSY** spectrum of **2aHL$_1$**



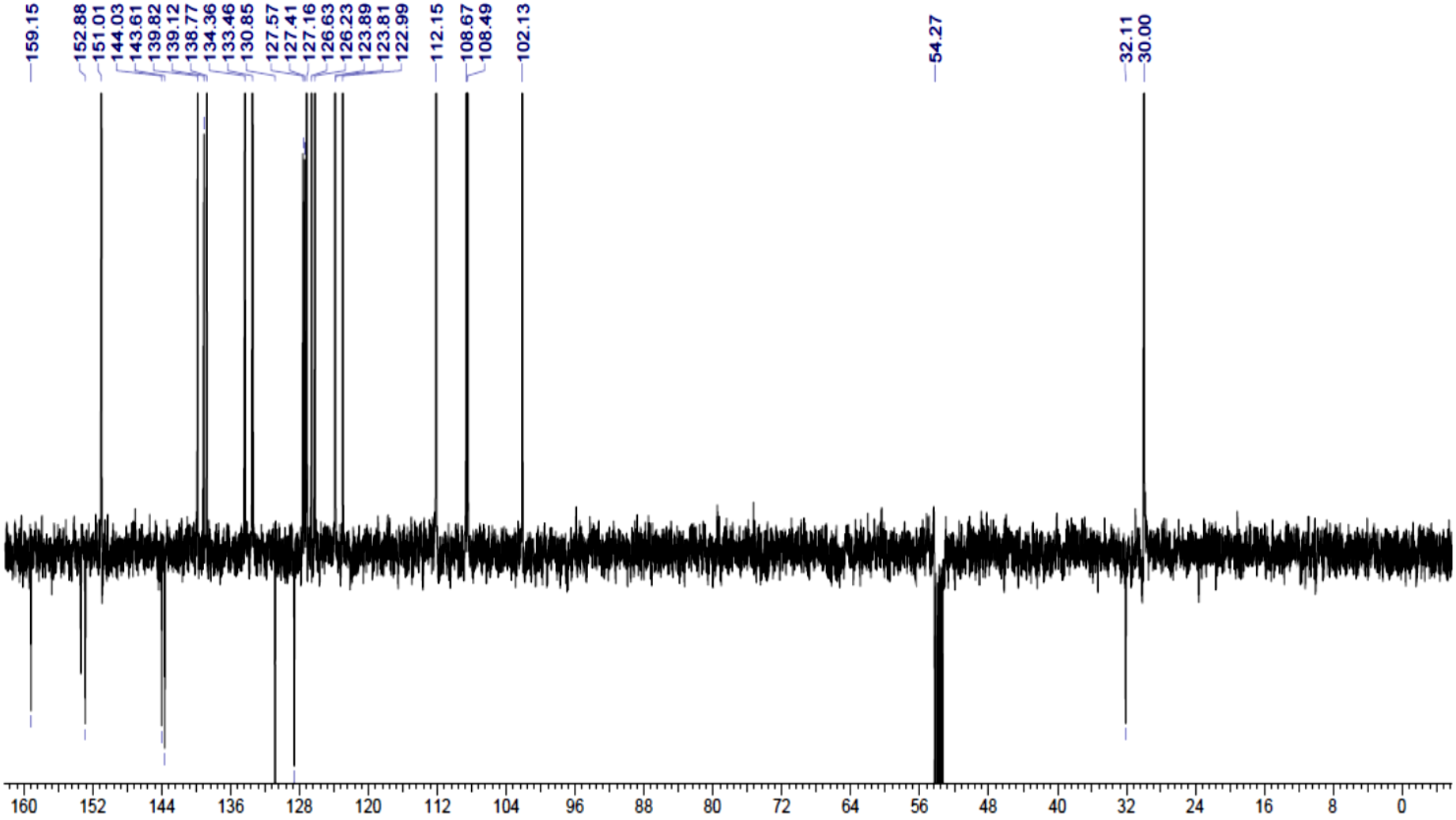
Section of the **NOESY** spectrum of **2aHL$_1$**



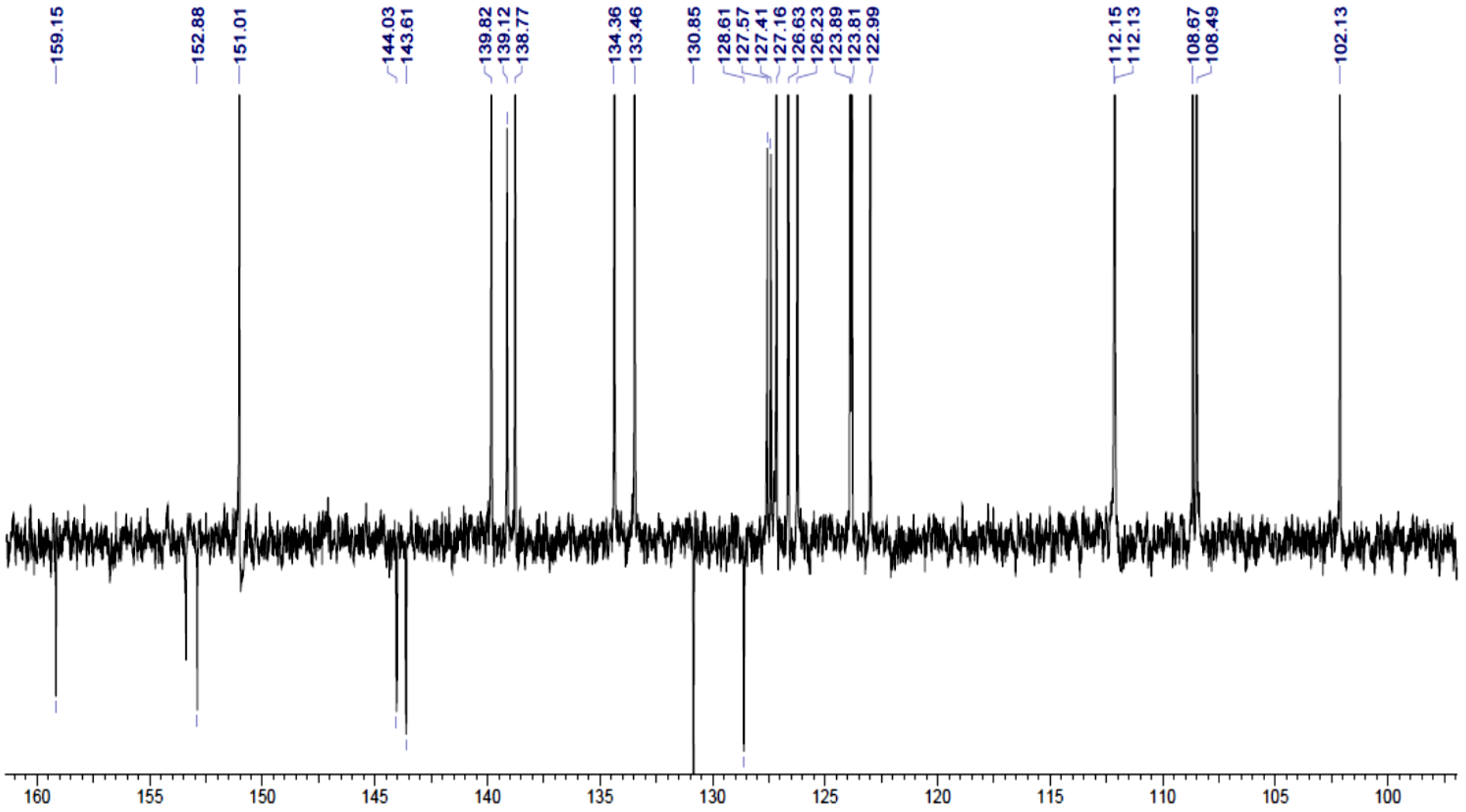
^{1}H NMR spectrum of **2aHL$_2$**



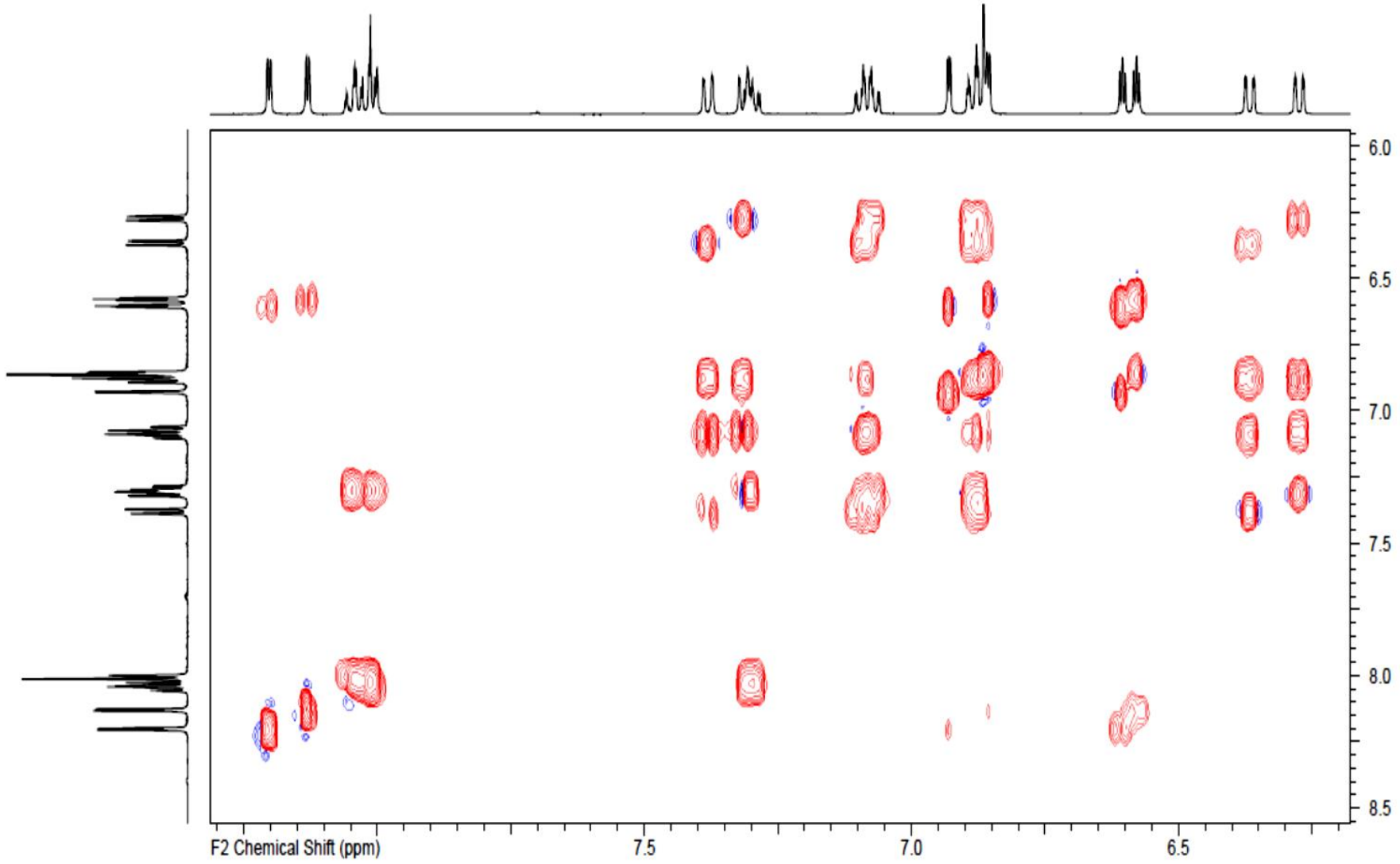
^{13}C NMR APT spectrum of **2aHL$_2$**



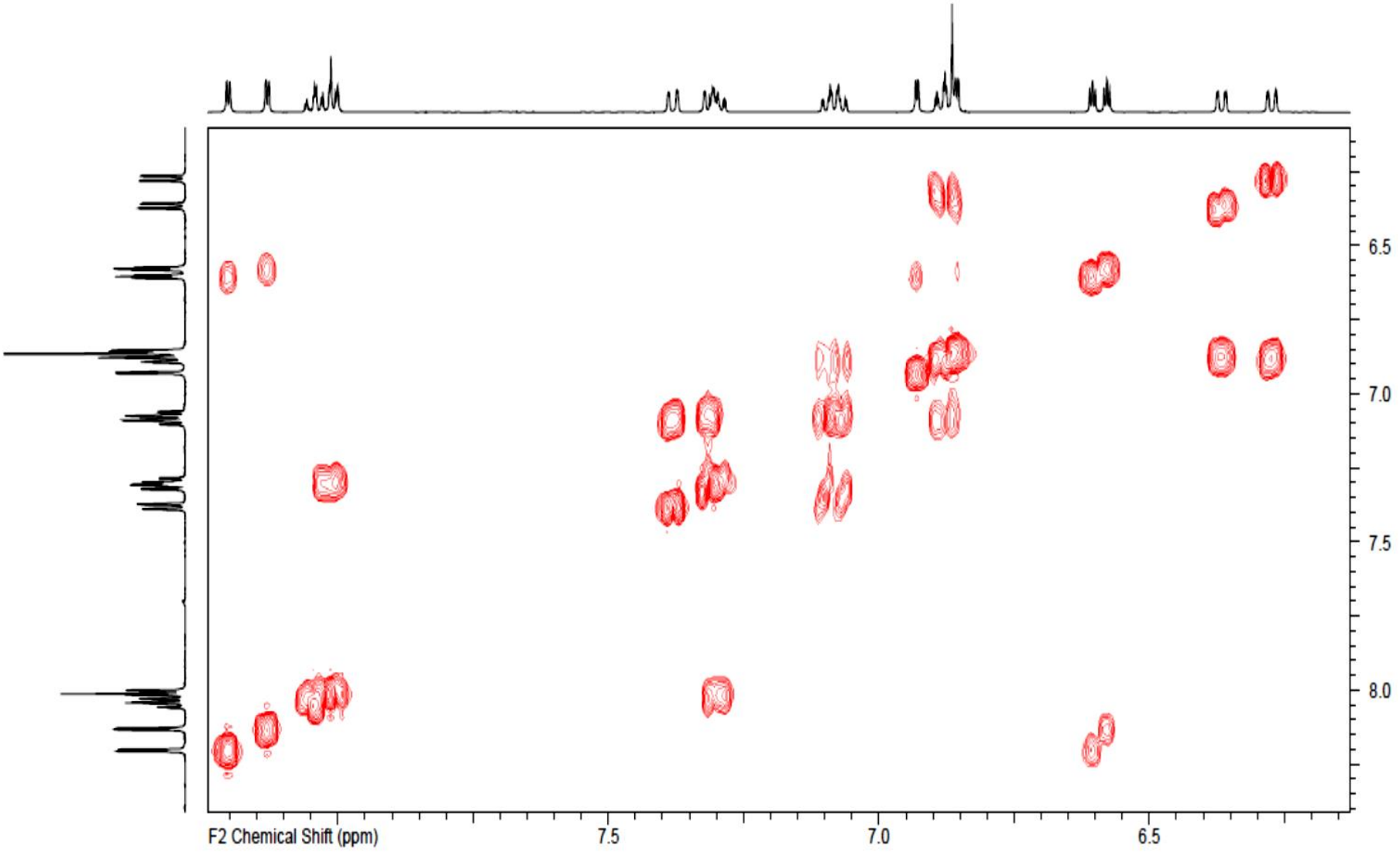
Section of ^{13}**C APT** spectrum of **2aHL**$_2$



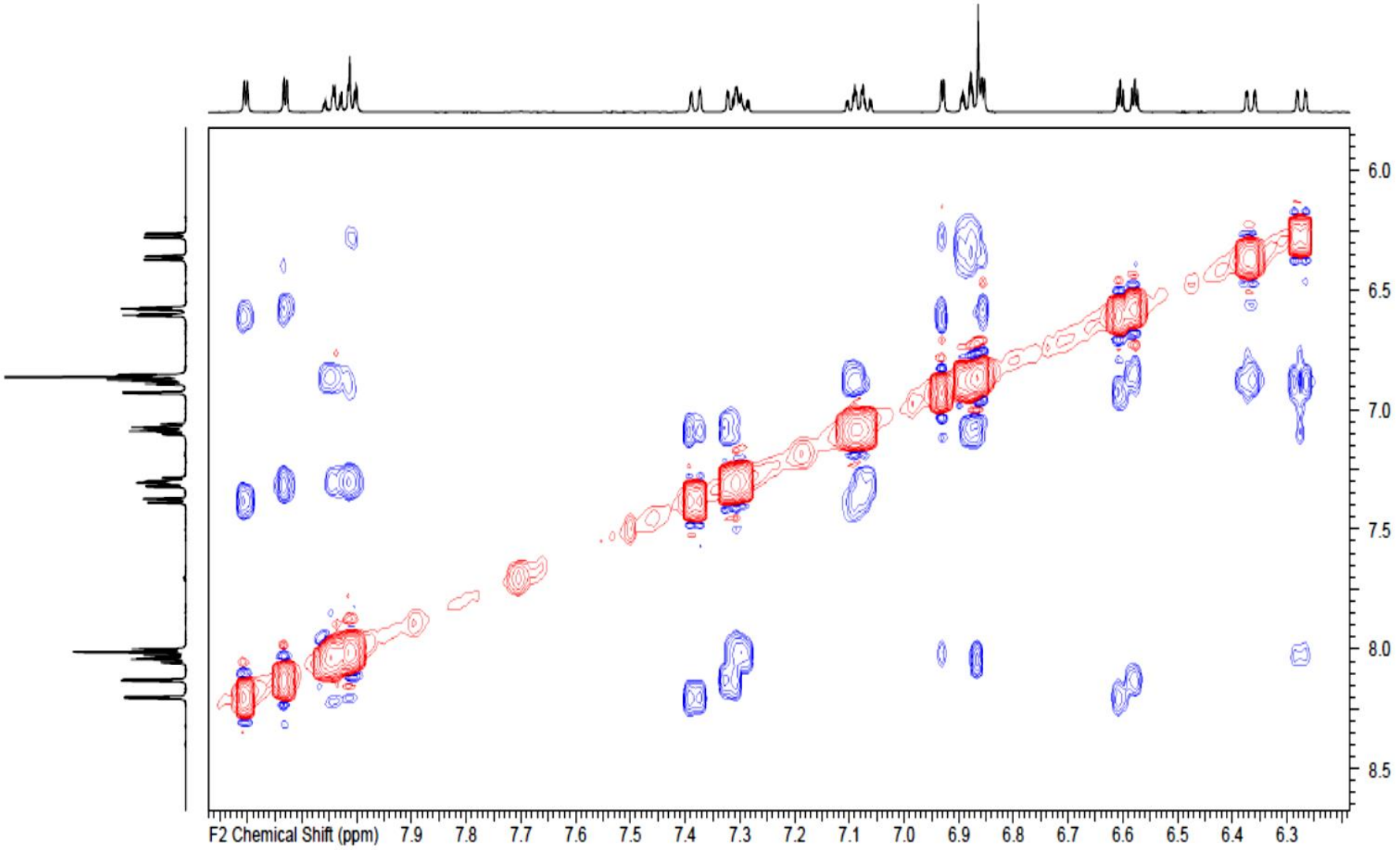
Section of the **TOCSY** spectrum of **2aHL**$_2$



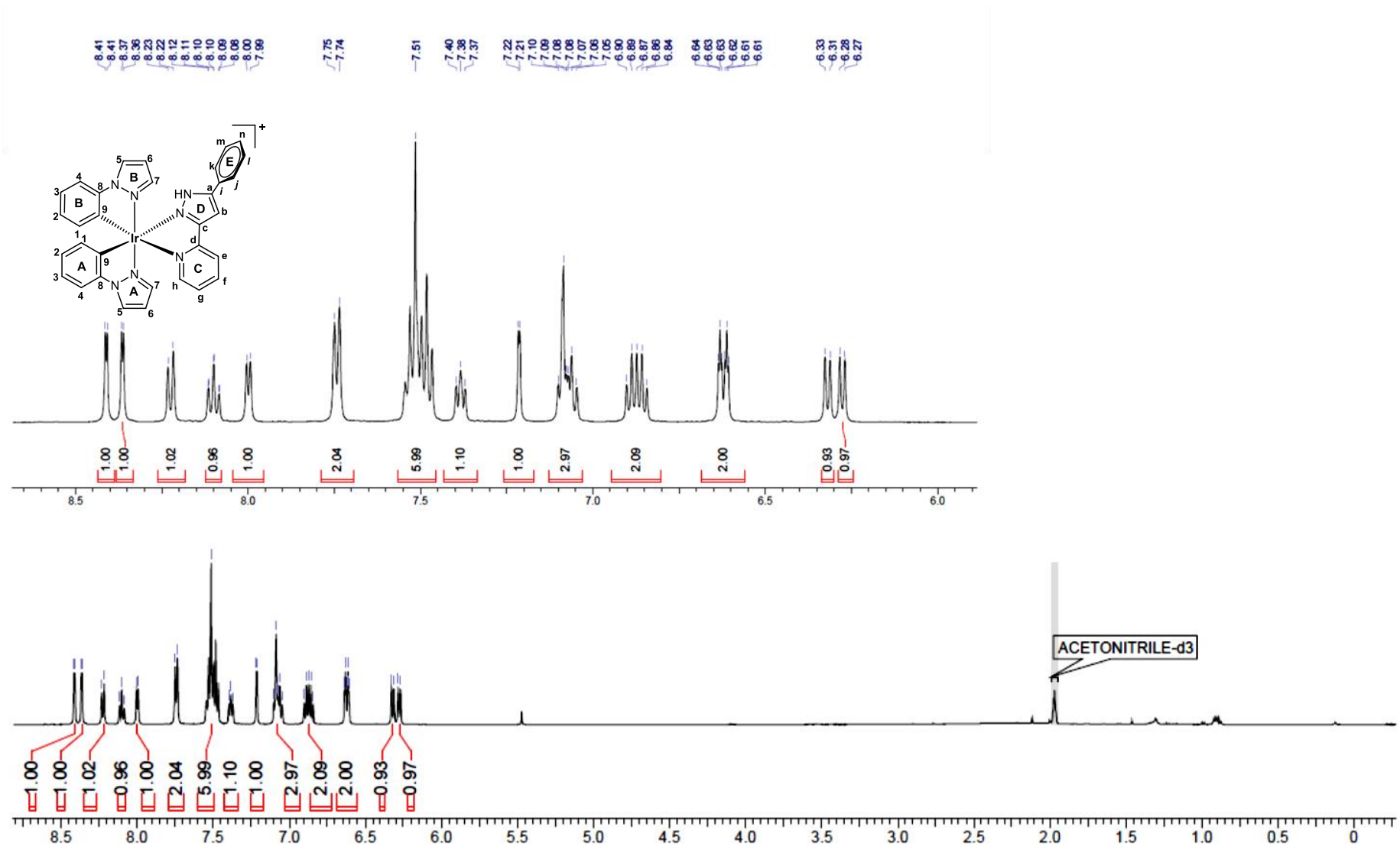
Section of the **COSY** spectrum of **2aHL$_2$**



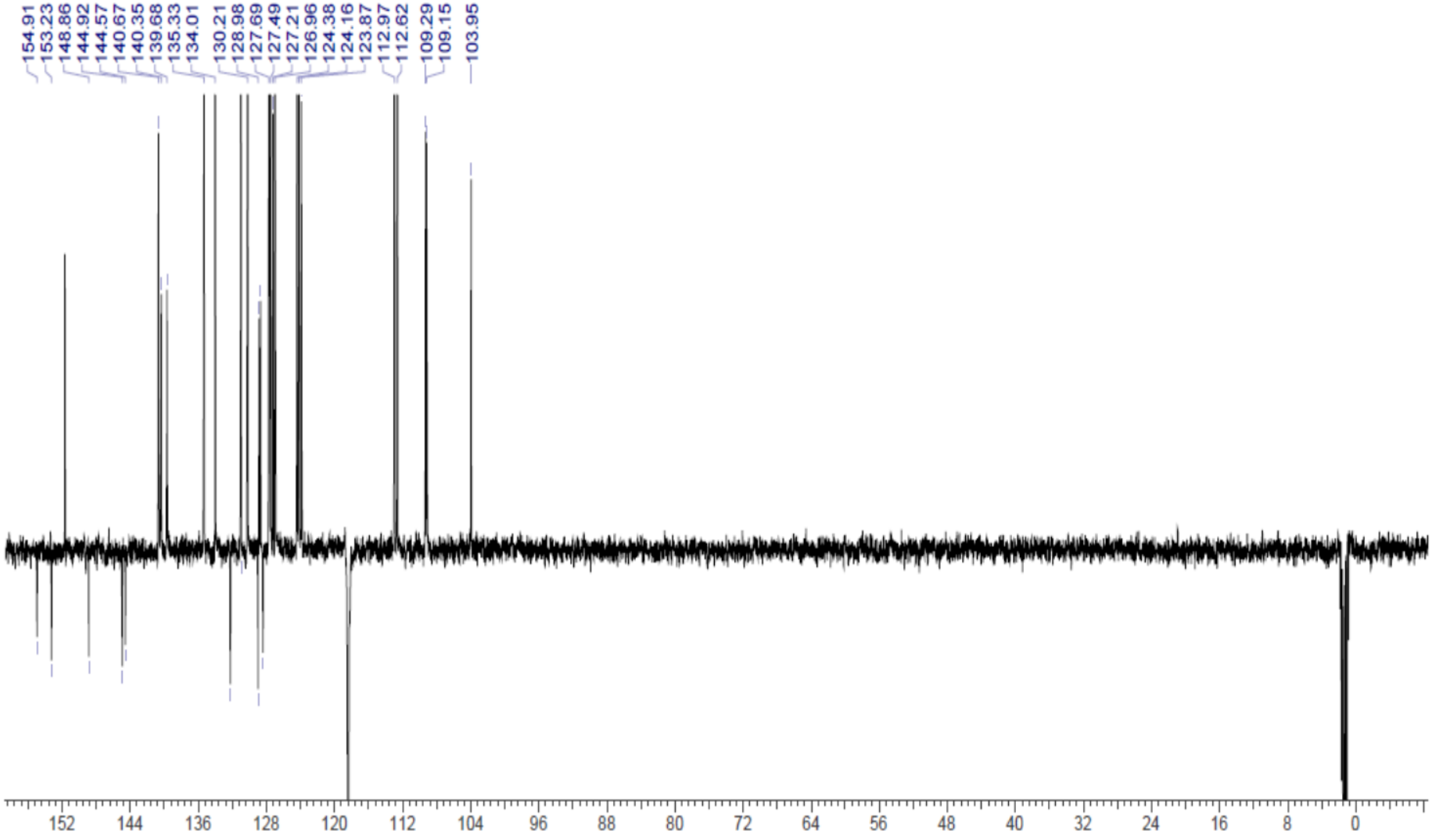
Section of the **NOESY** spectrum of **2aHL$_2$**



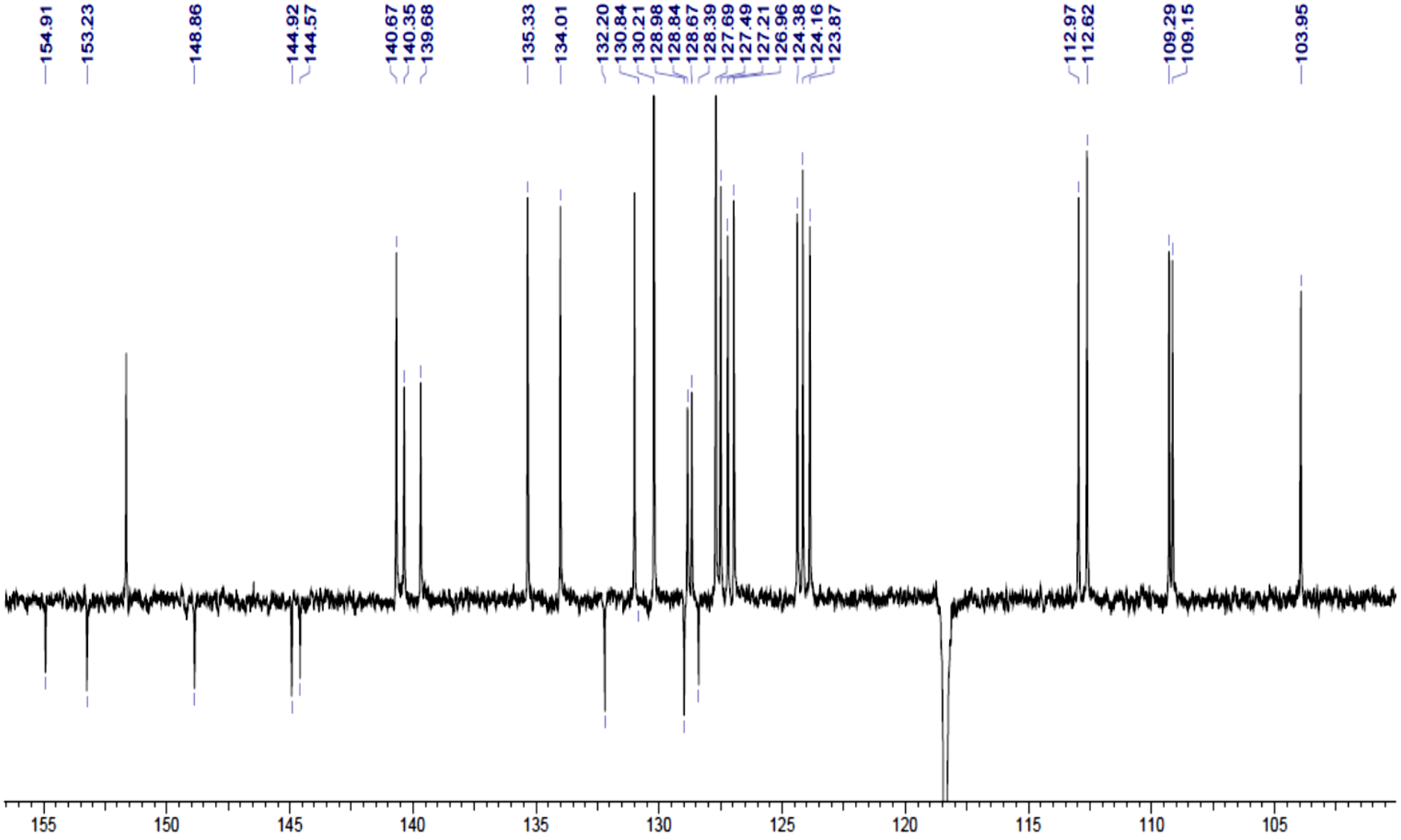
^{1}H NMR spectrum of **2aHL$_3$**



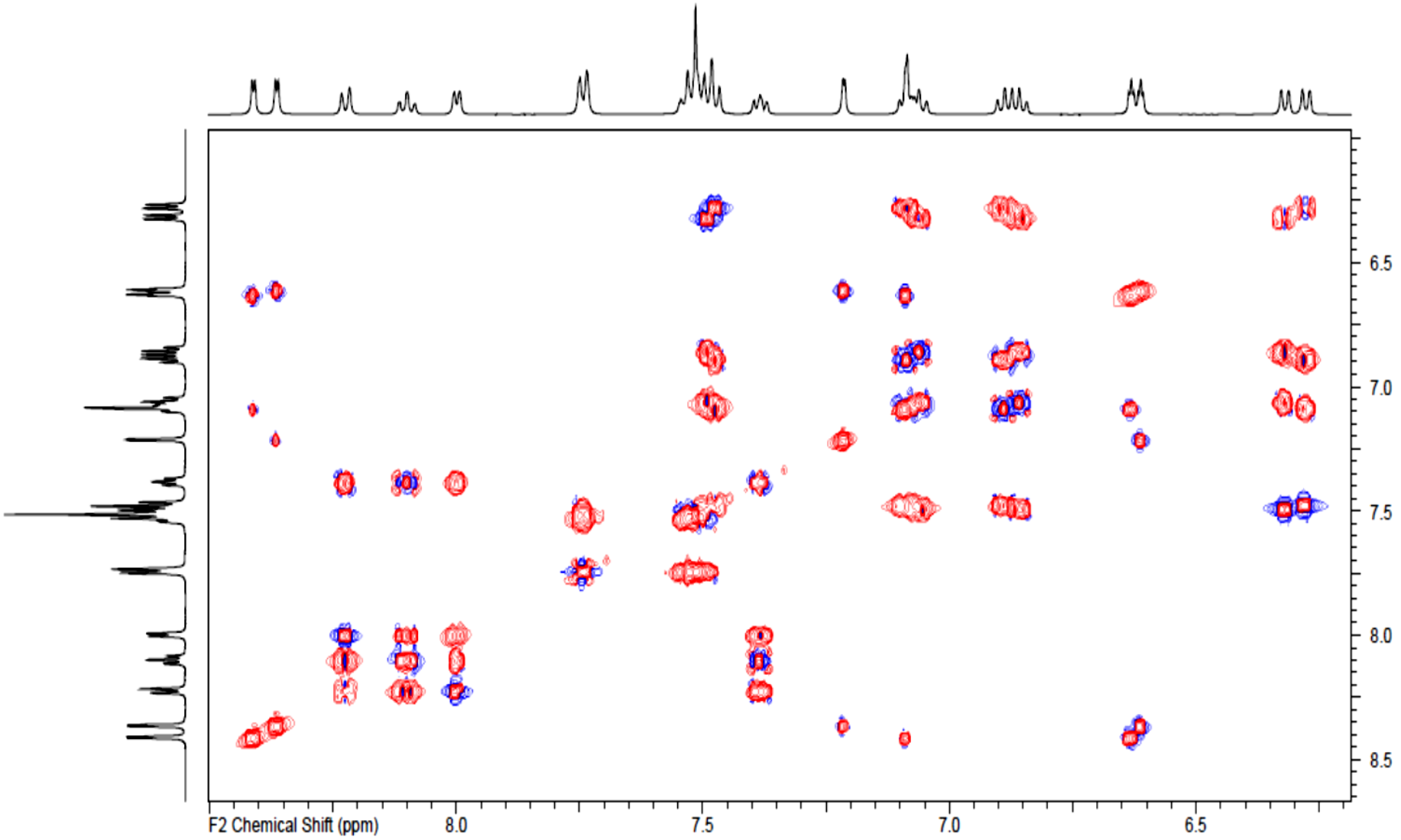
^{13}C NMR APT spectrum of **2aHL$_3$**



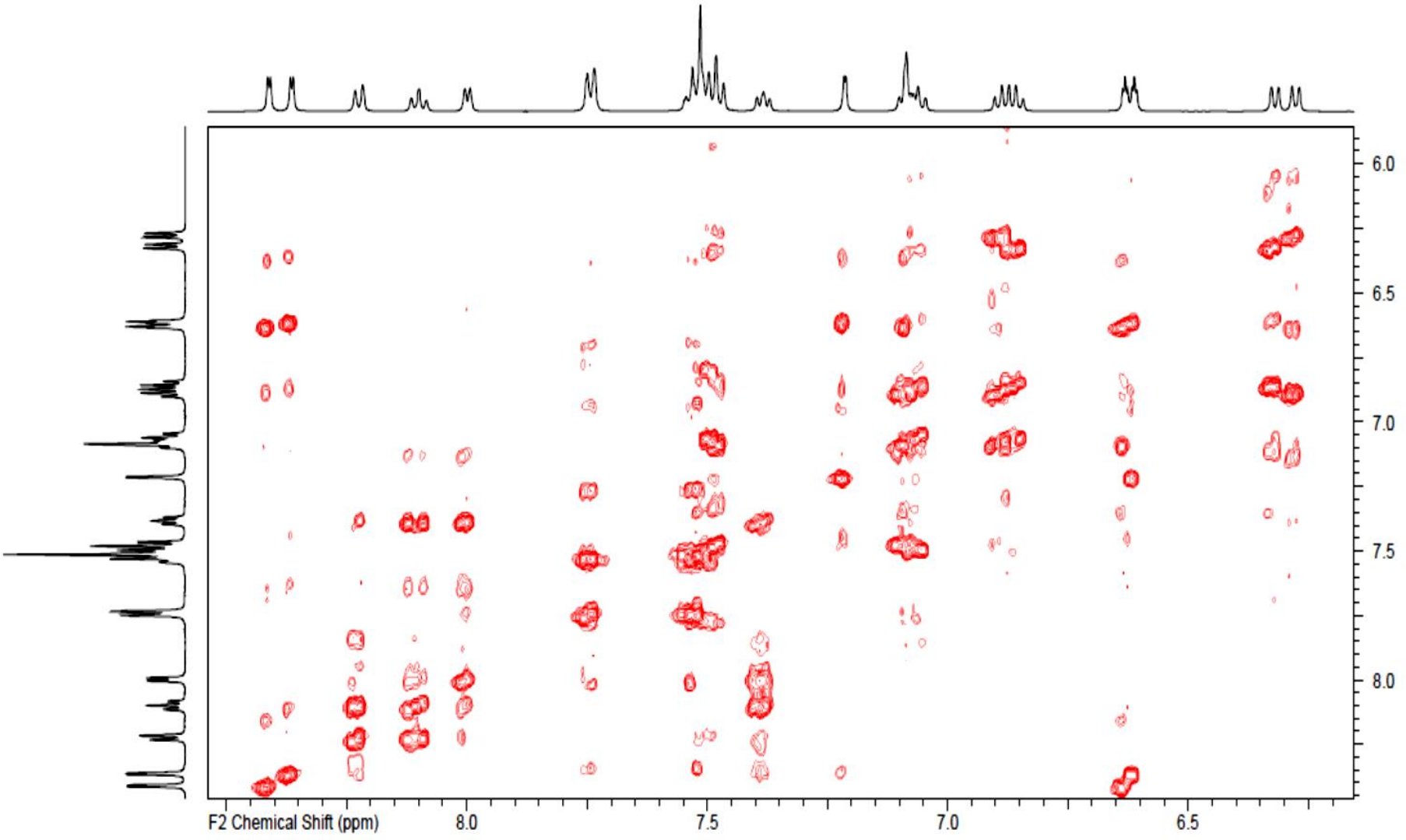
Section of ^{13}**C NMR APT** spectrum of **2aHL**$_3$



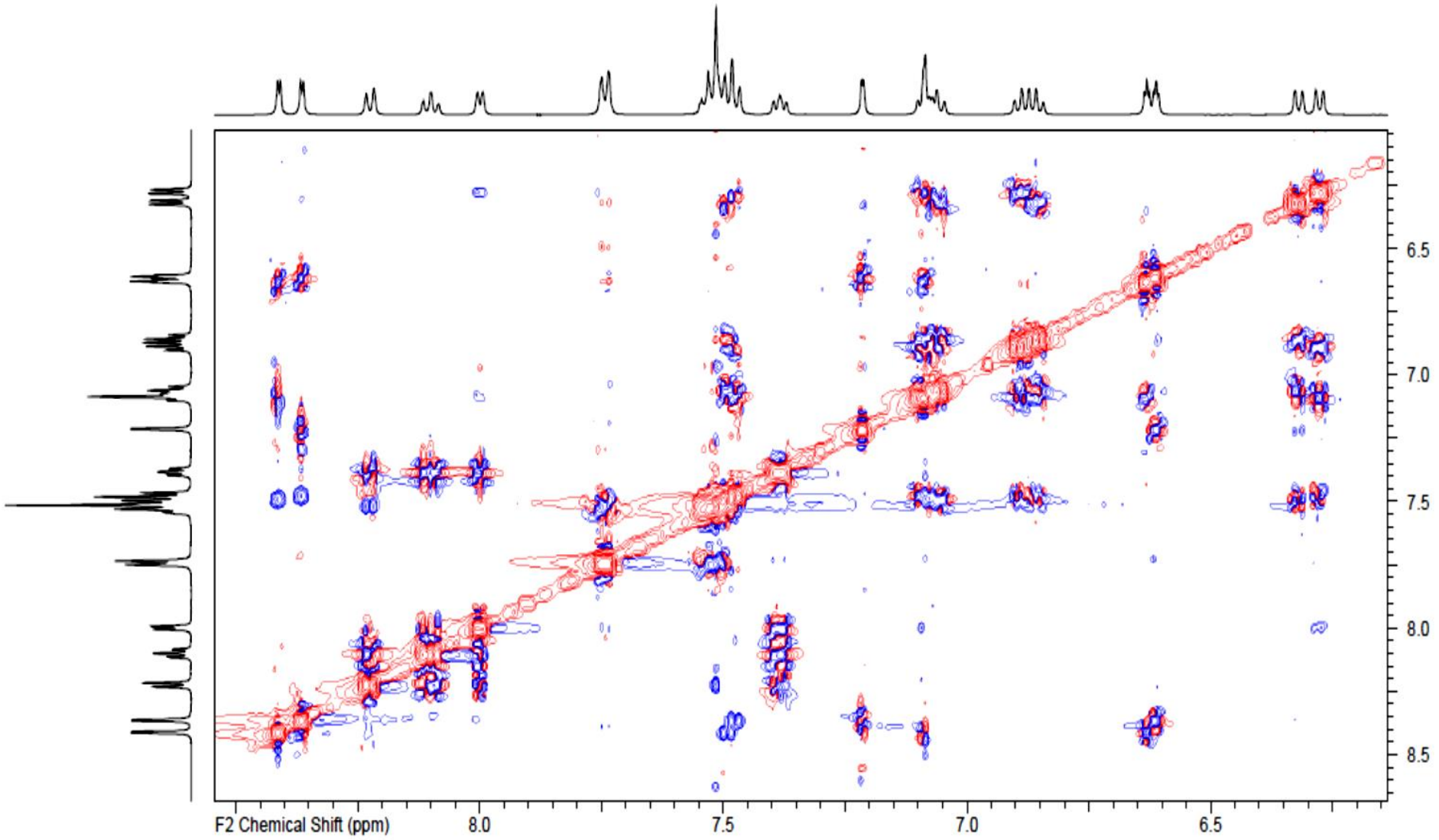
Section of the **TOCSY** spectrum of **2aHL$_3$**



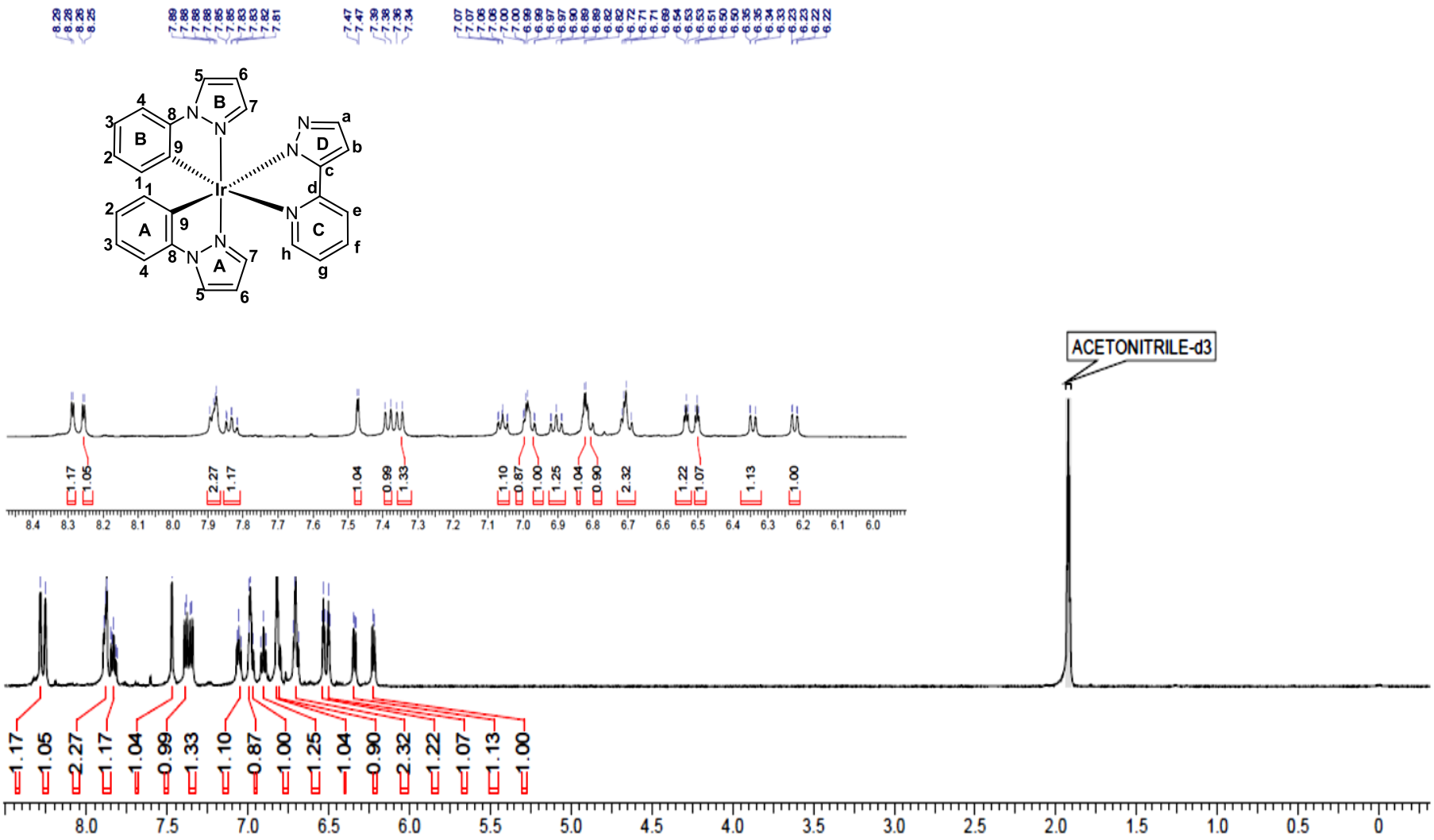
Section of the **COSY** spectrum of **2aHL$_3$**



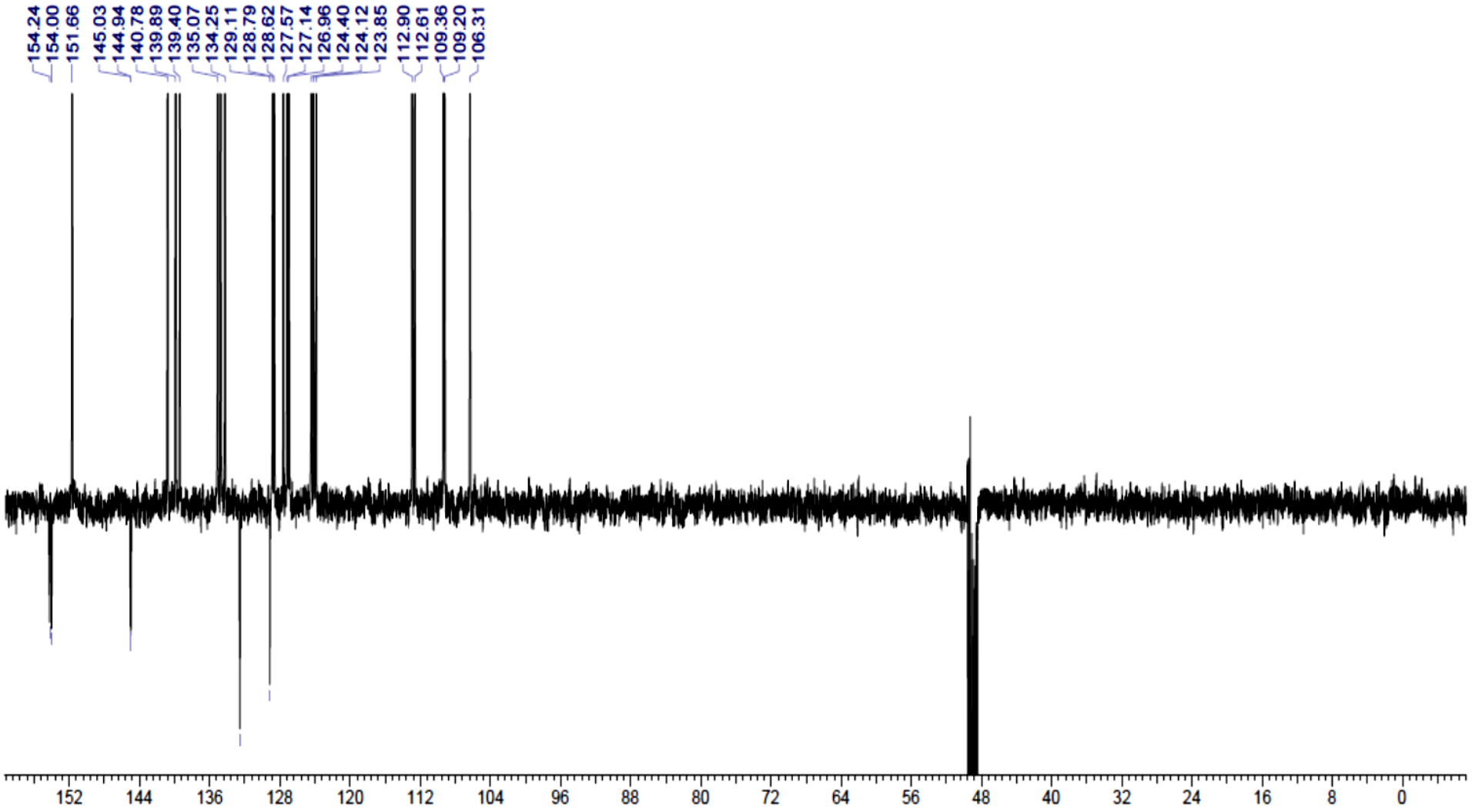
Section of the **NOESY** spectrum of **2aHL$_3$**



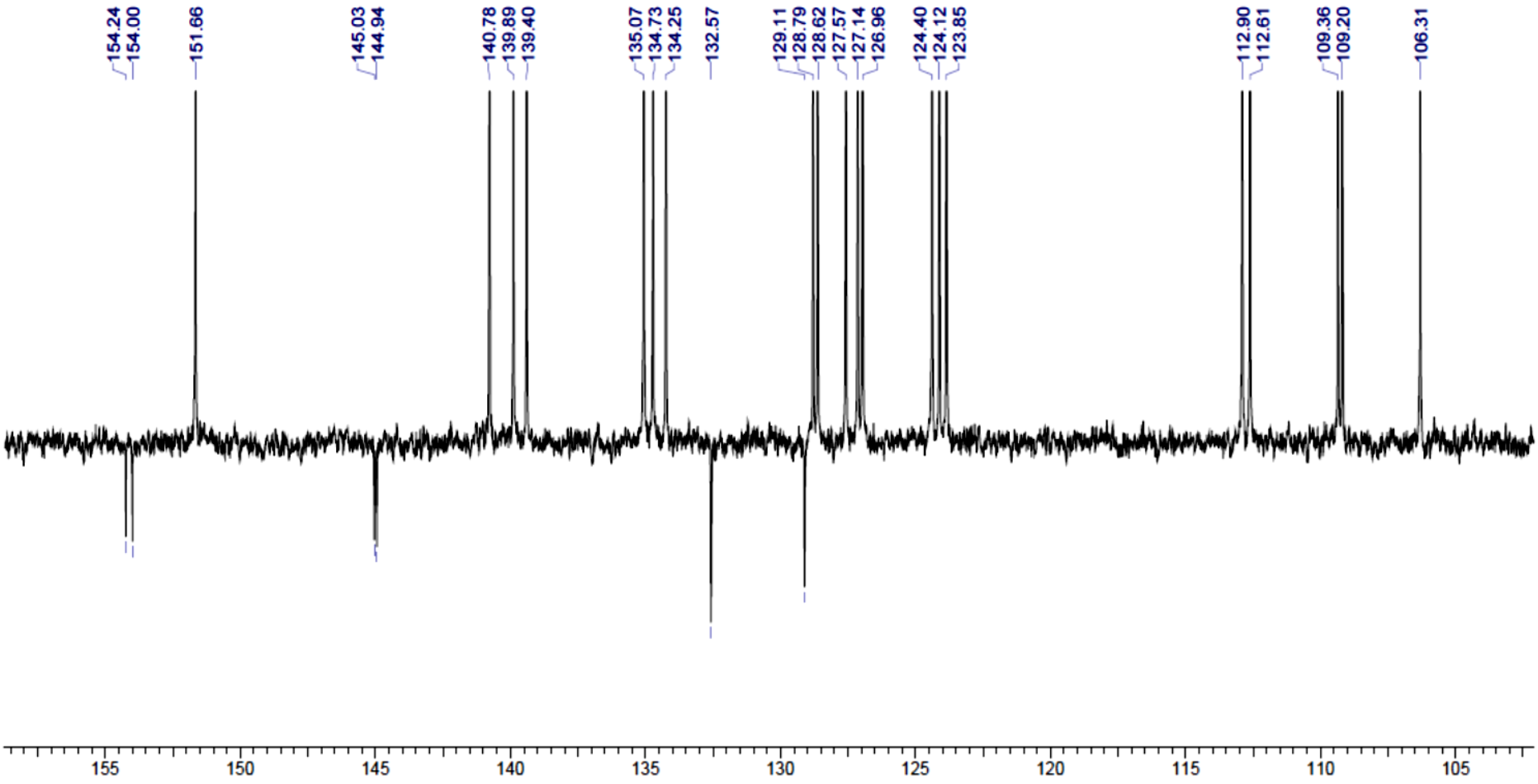
^{1}H NMR spectrum of **3aL$_1$**



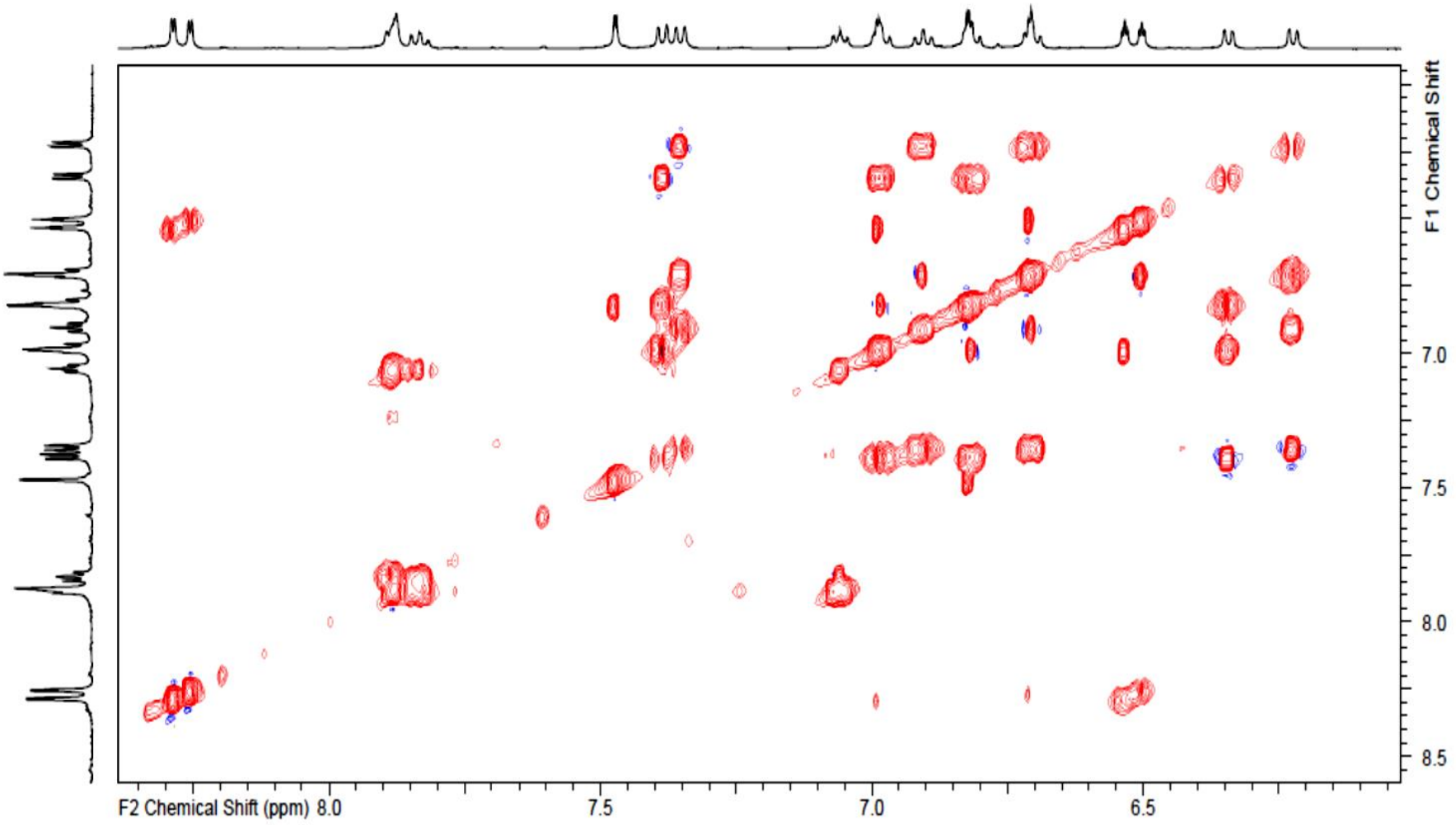
^{13}C DEPT spectrum of **3aL$_1$**



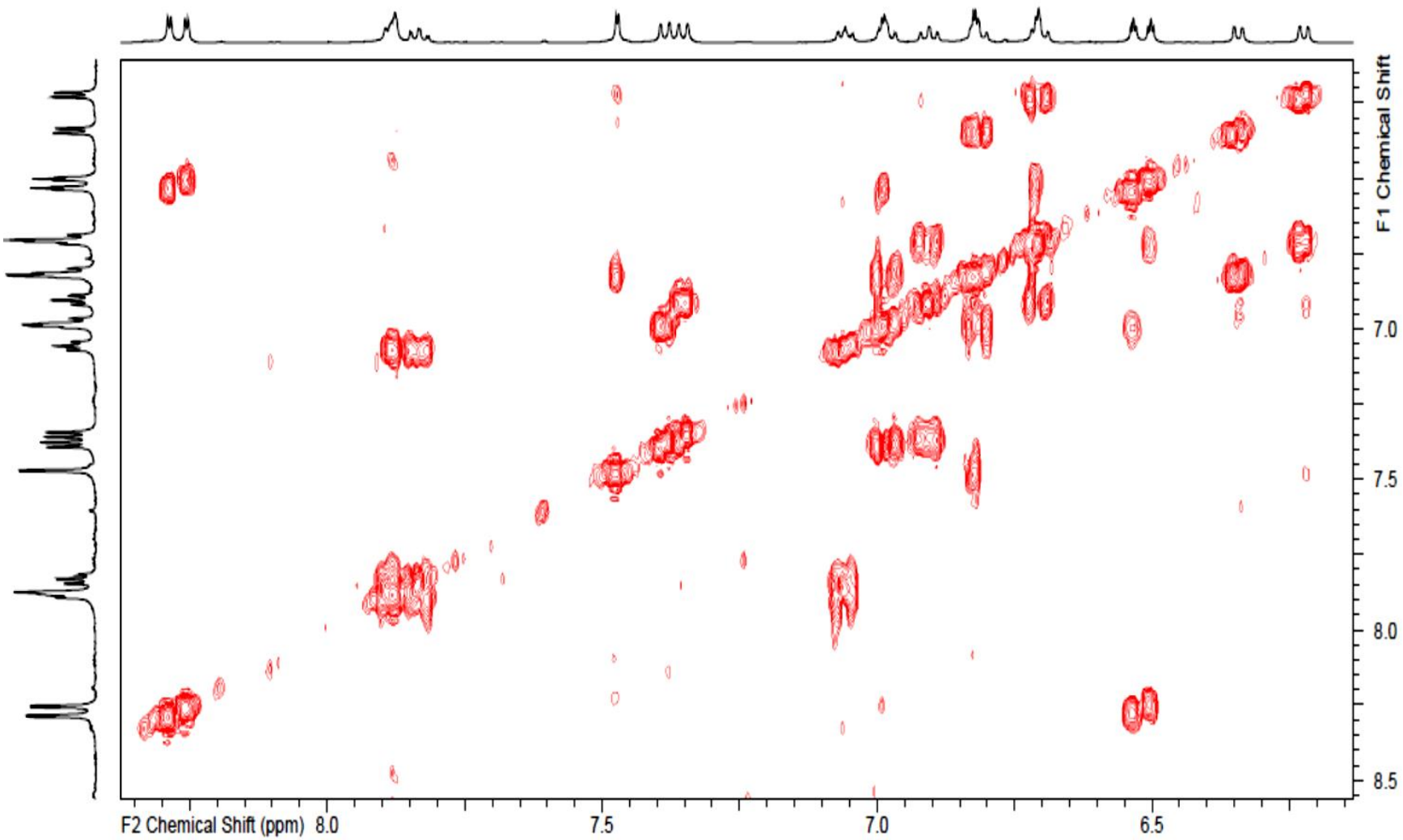
Section of ^{13}**C DEPT** spectrum of **3aL$_1$**



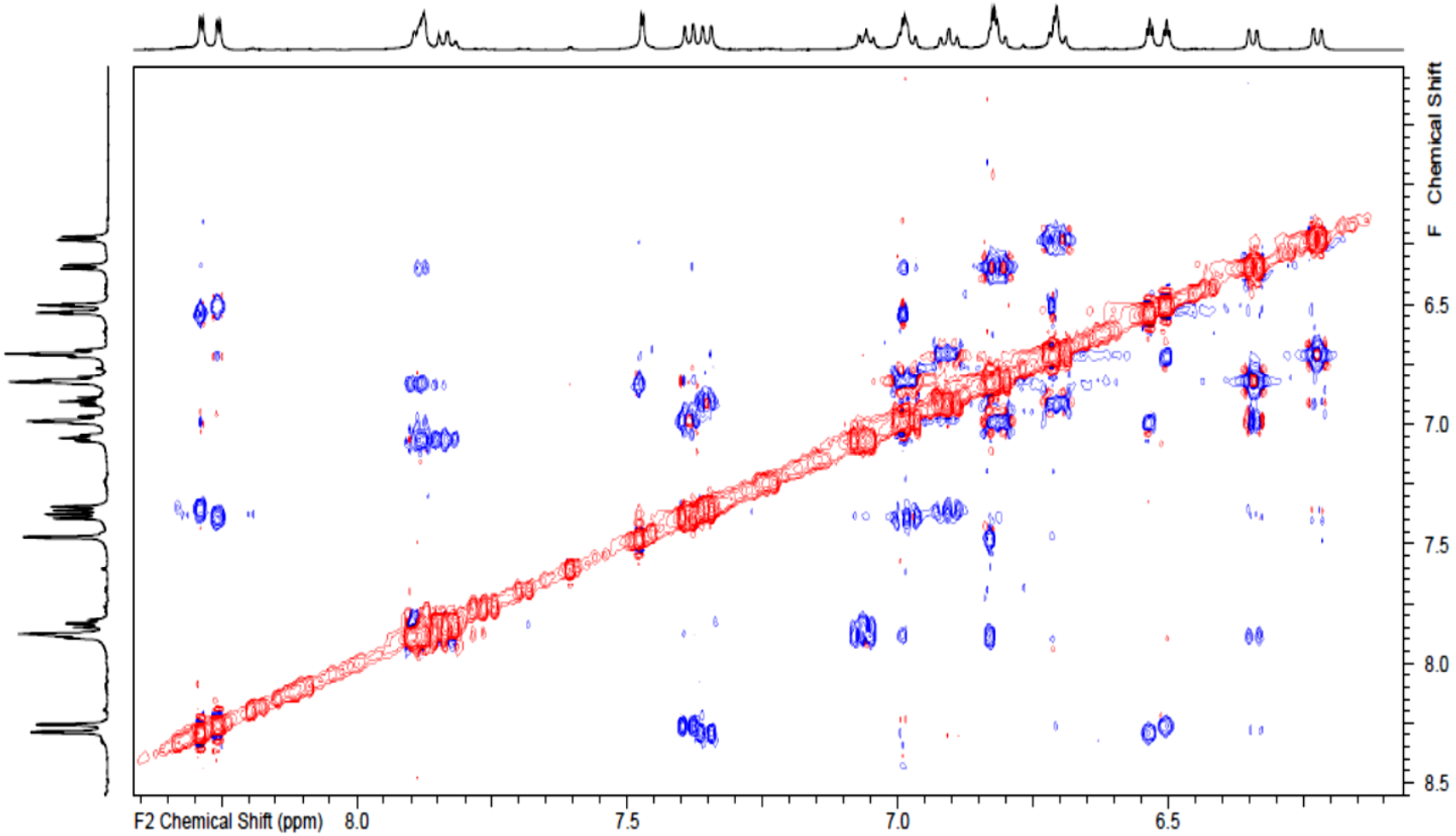
Section of the **TOCSY** spectrum of **3aL$_1$**



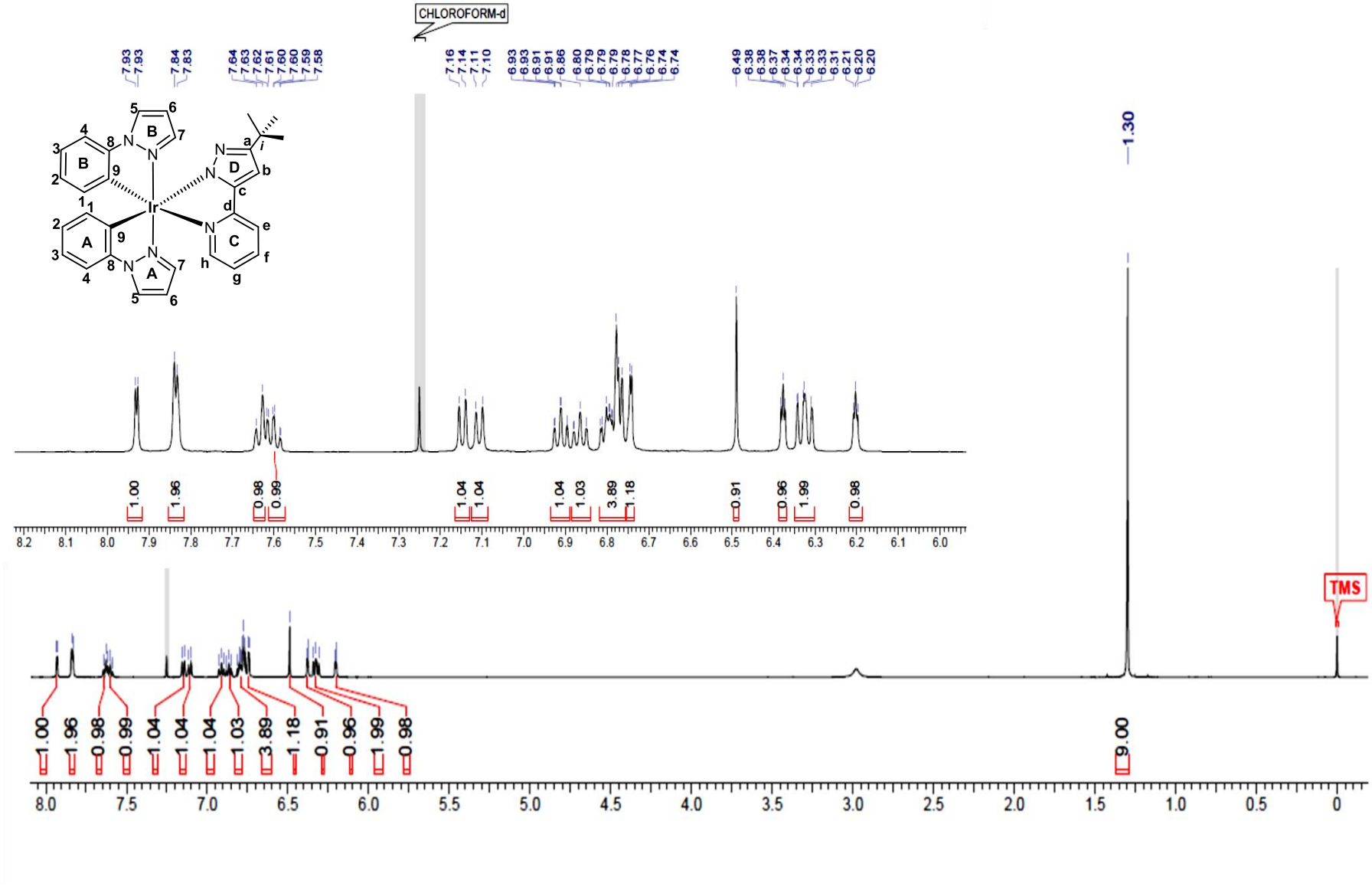
Section of the **COSY** spectrum of **3aL$_1$**



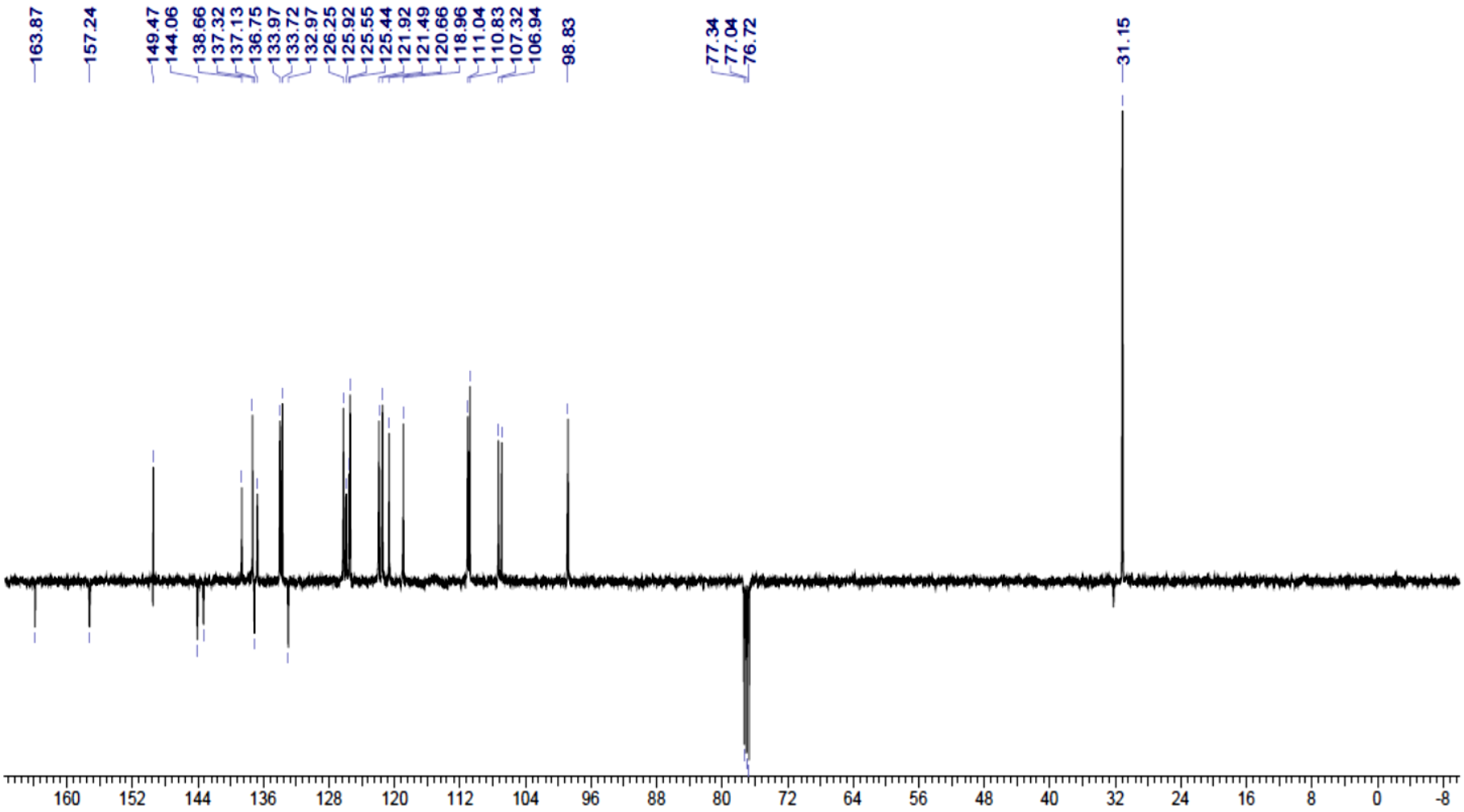
Section of the **NOESY** spectrum of **3aL$_1$**



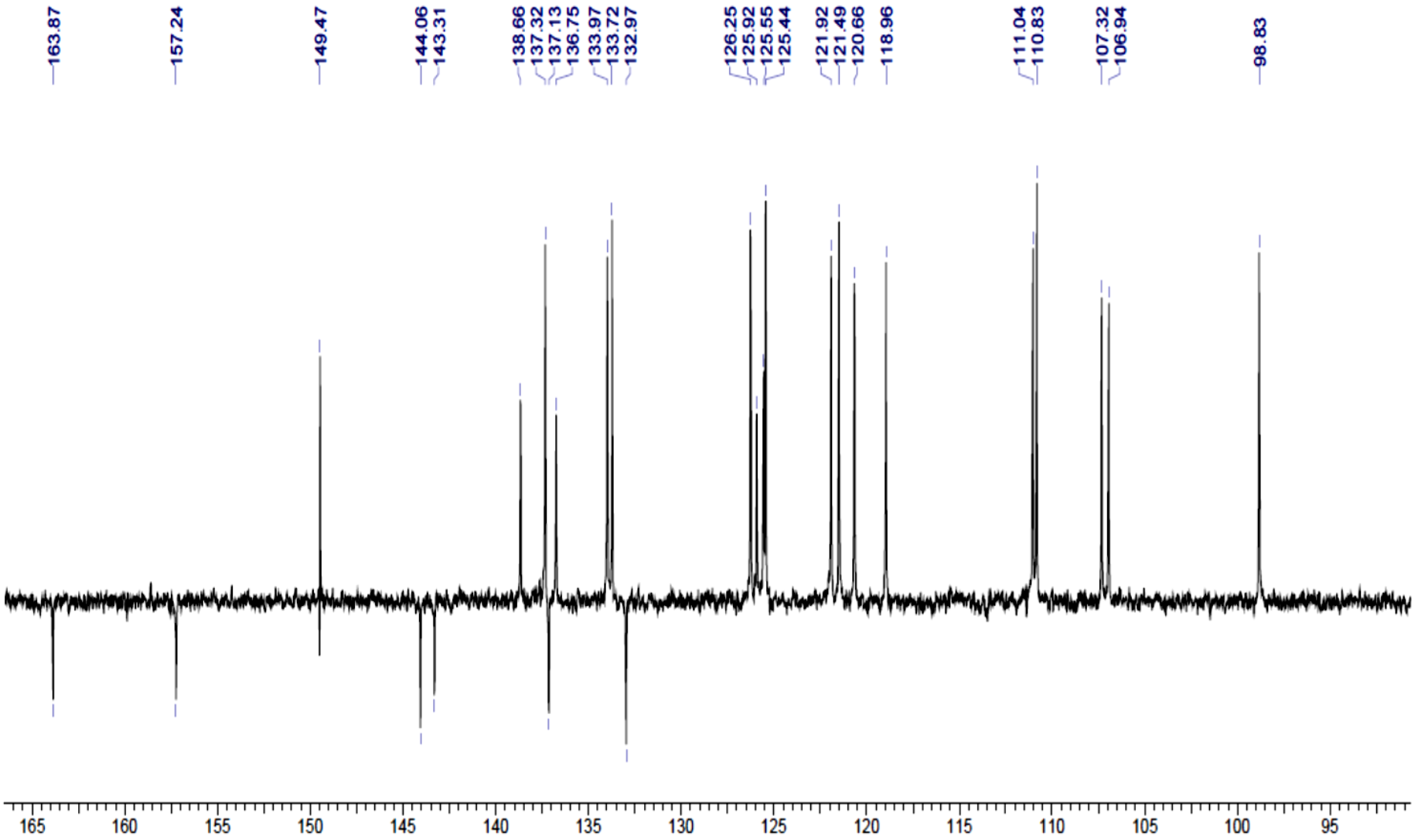
^{1}H NMR spectrum of **3aL$_2$**



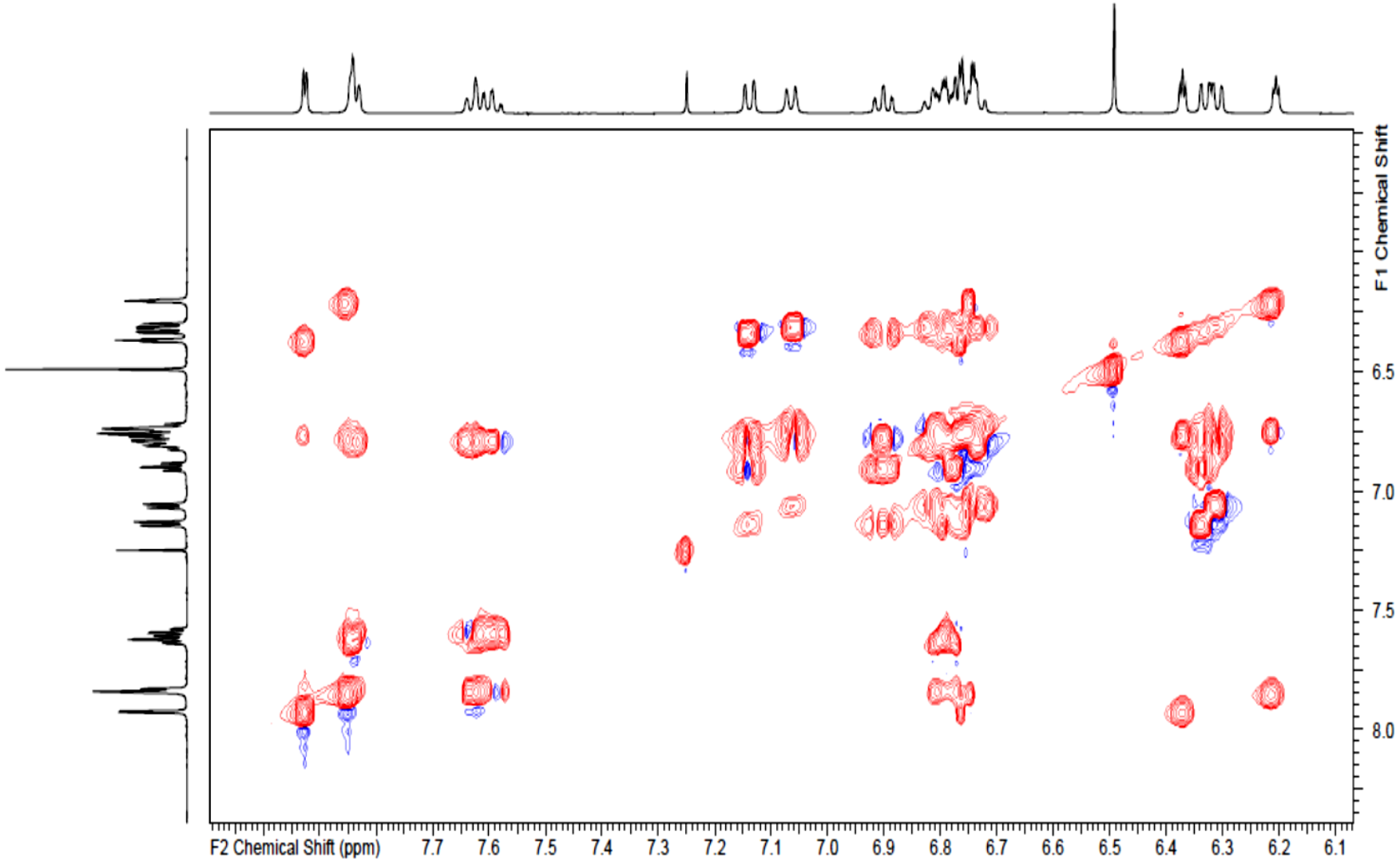
^{13}C APT spectrum of **3aL$_2$**



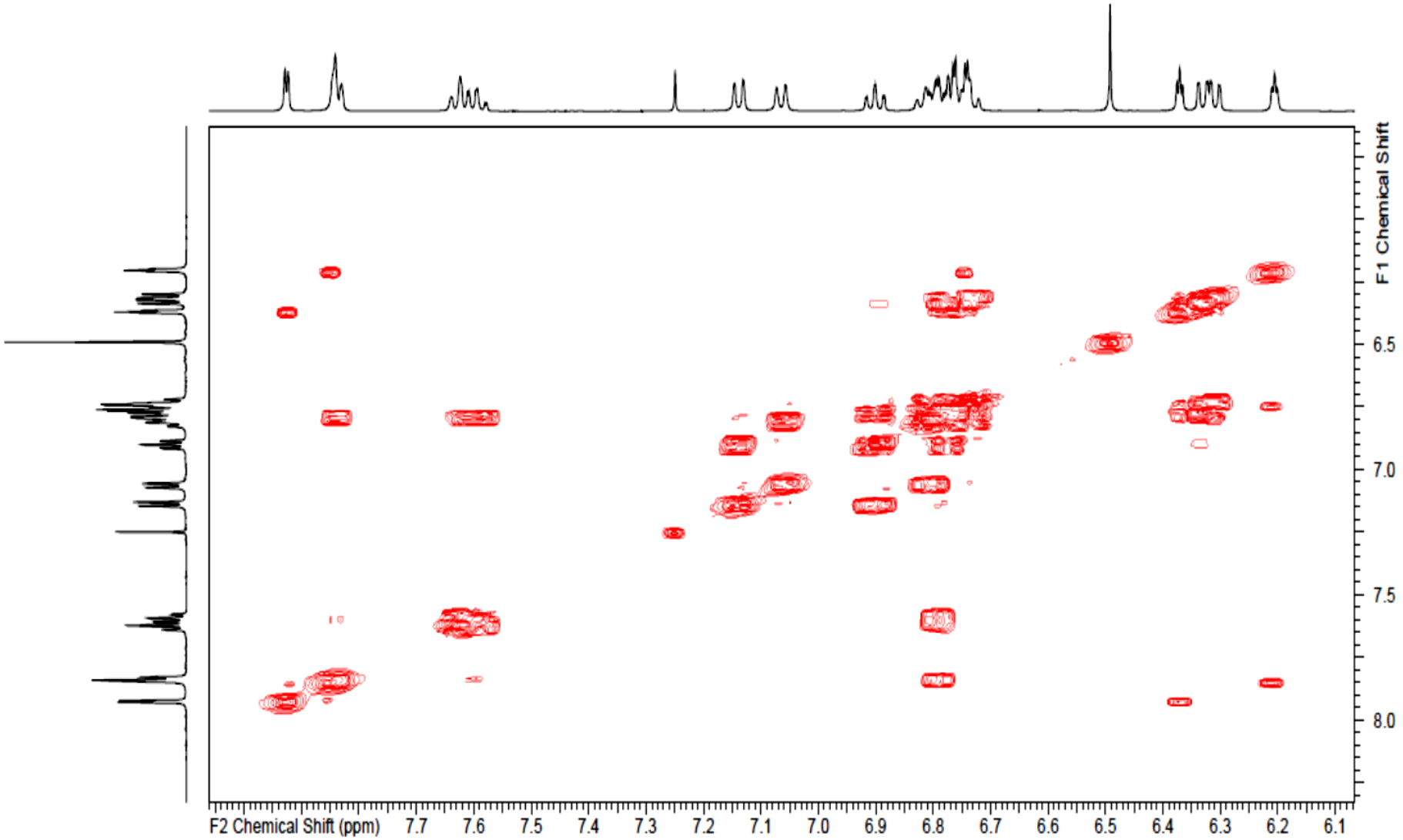
Section of ^{13}**C APT** spectrum of **3aL**$_2$



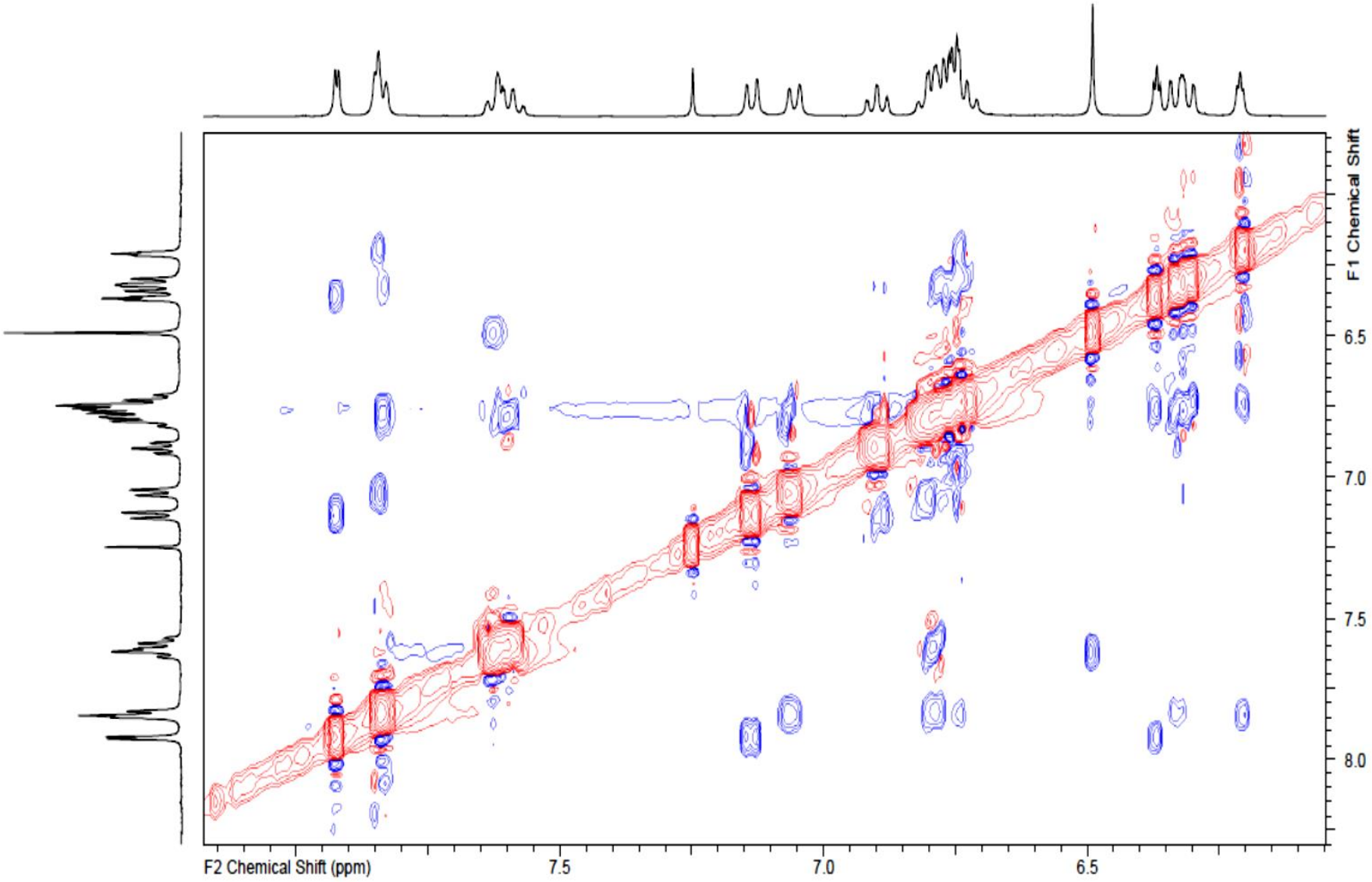
Section of the **TOCSY** spectrum of **3aL$_2$**



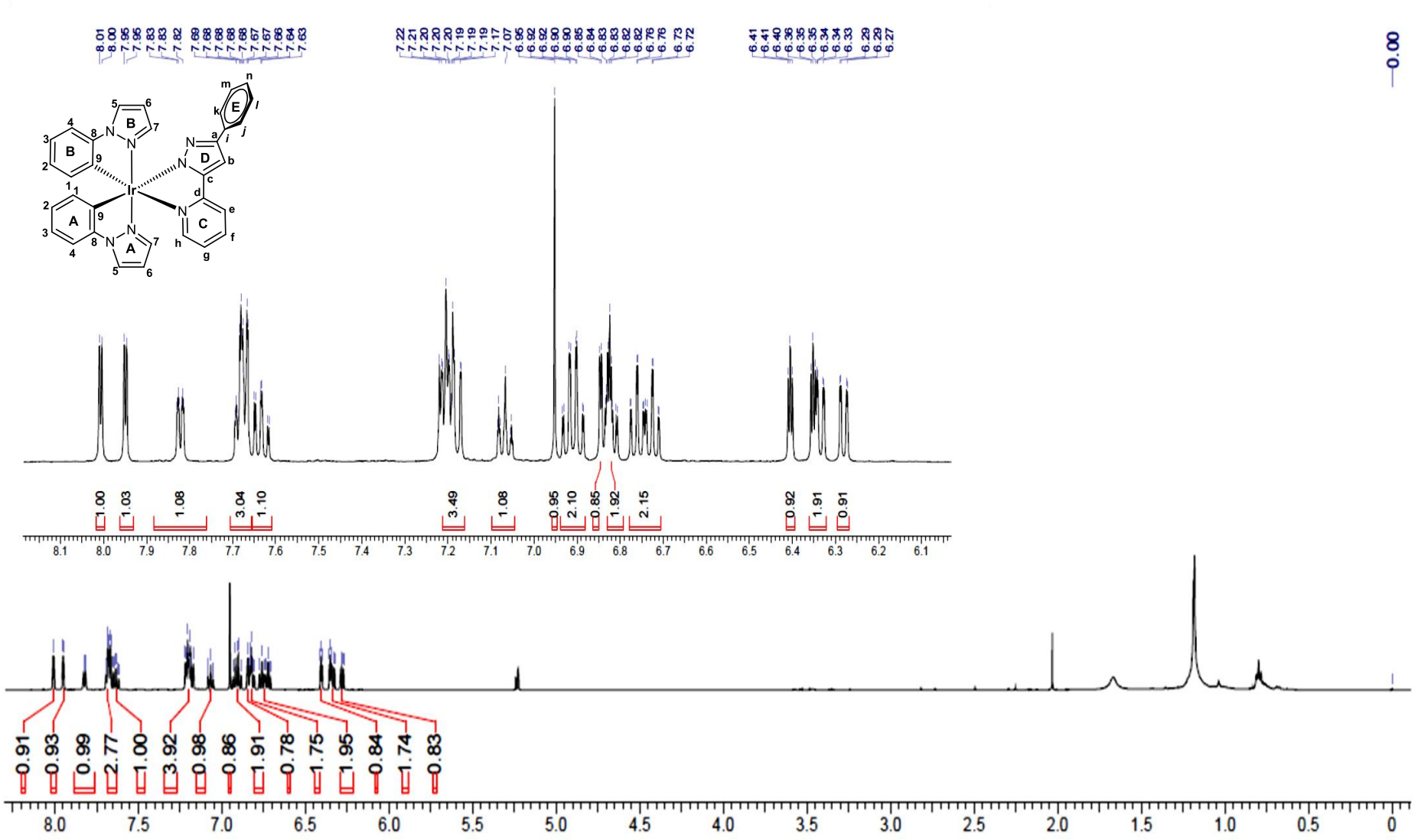
Section of the **COSY** spectrum of **3aL**$_2$



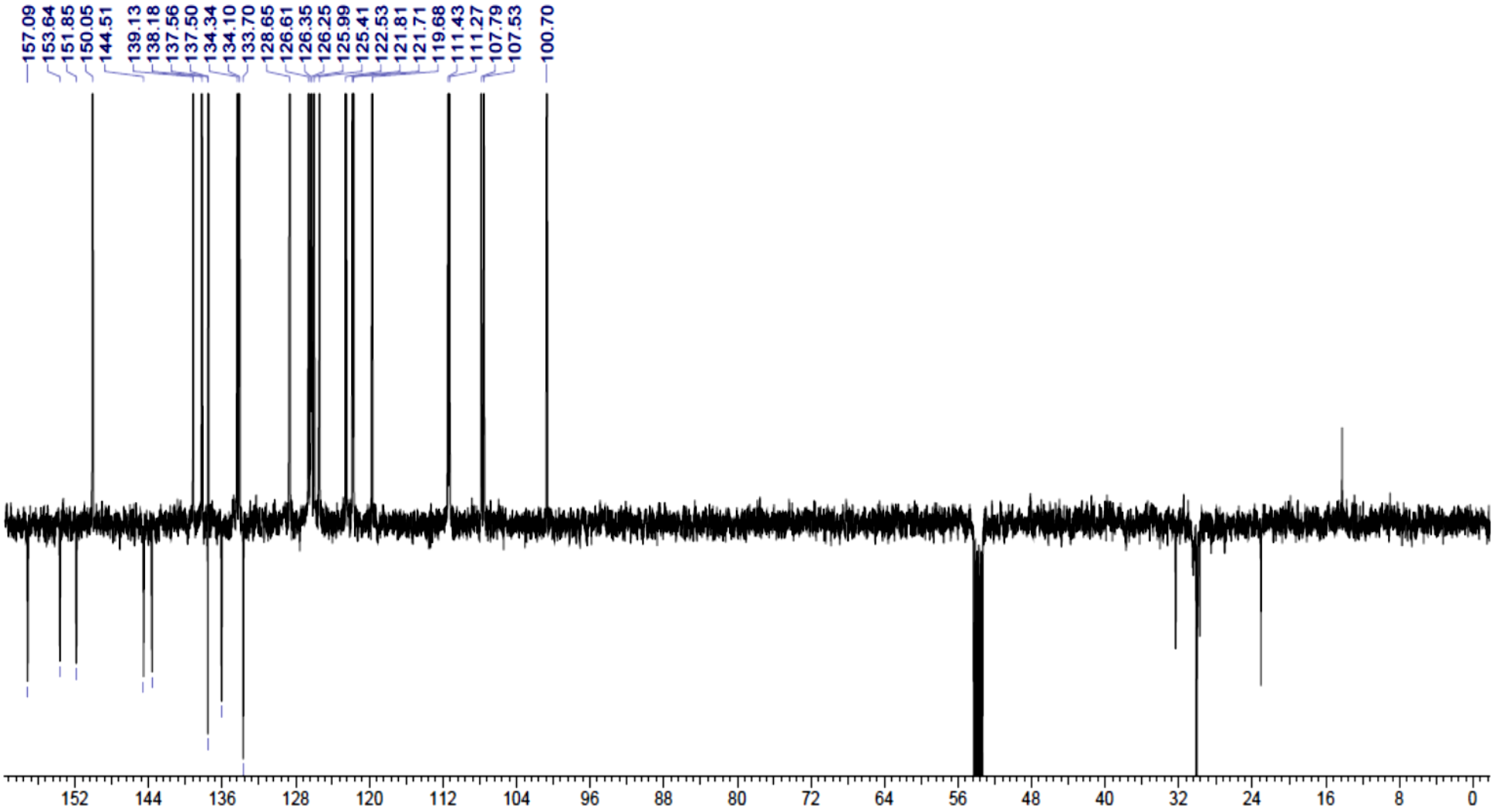
Section of the **NOESY** spectrum of **3aL$_2$**



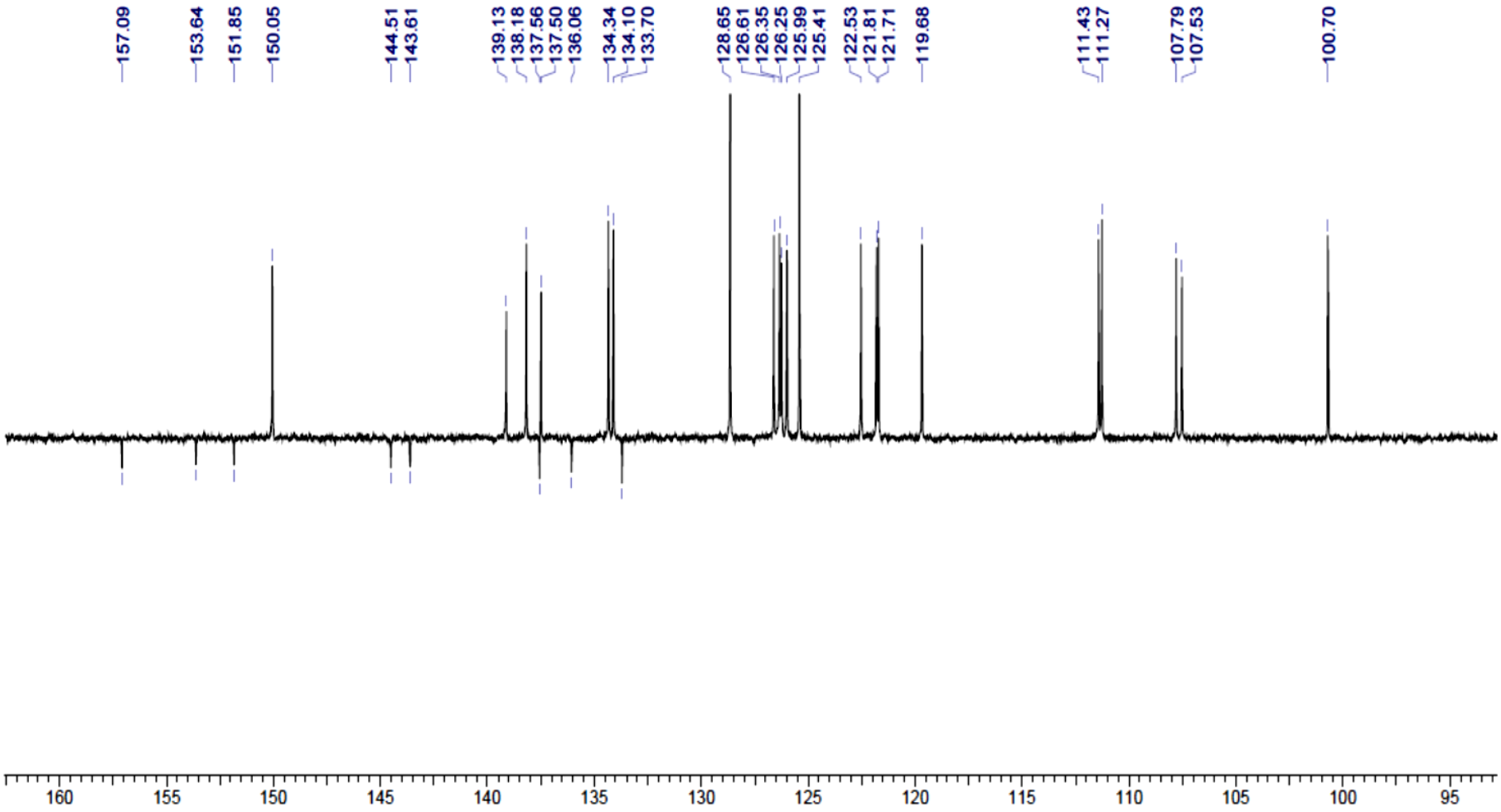
^{1}HNMR spectrum of **3aL$_3$**



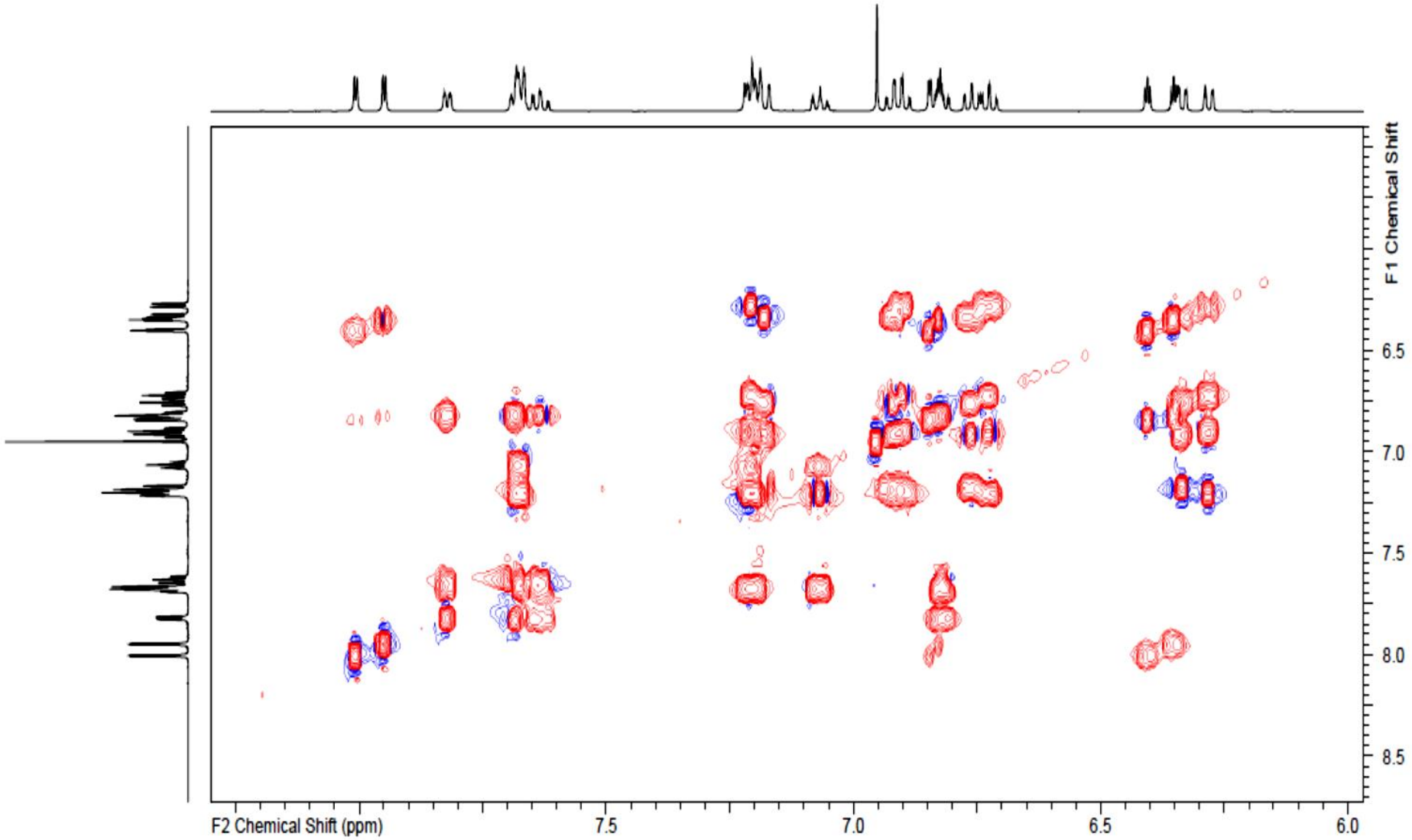
^{13}C NMR DEPT spectrum of **3aL$_3$**



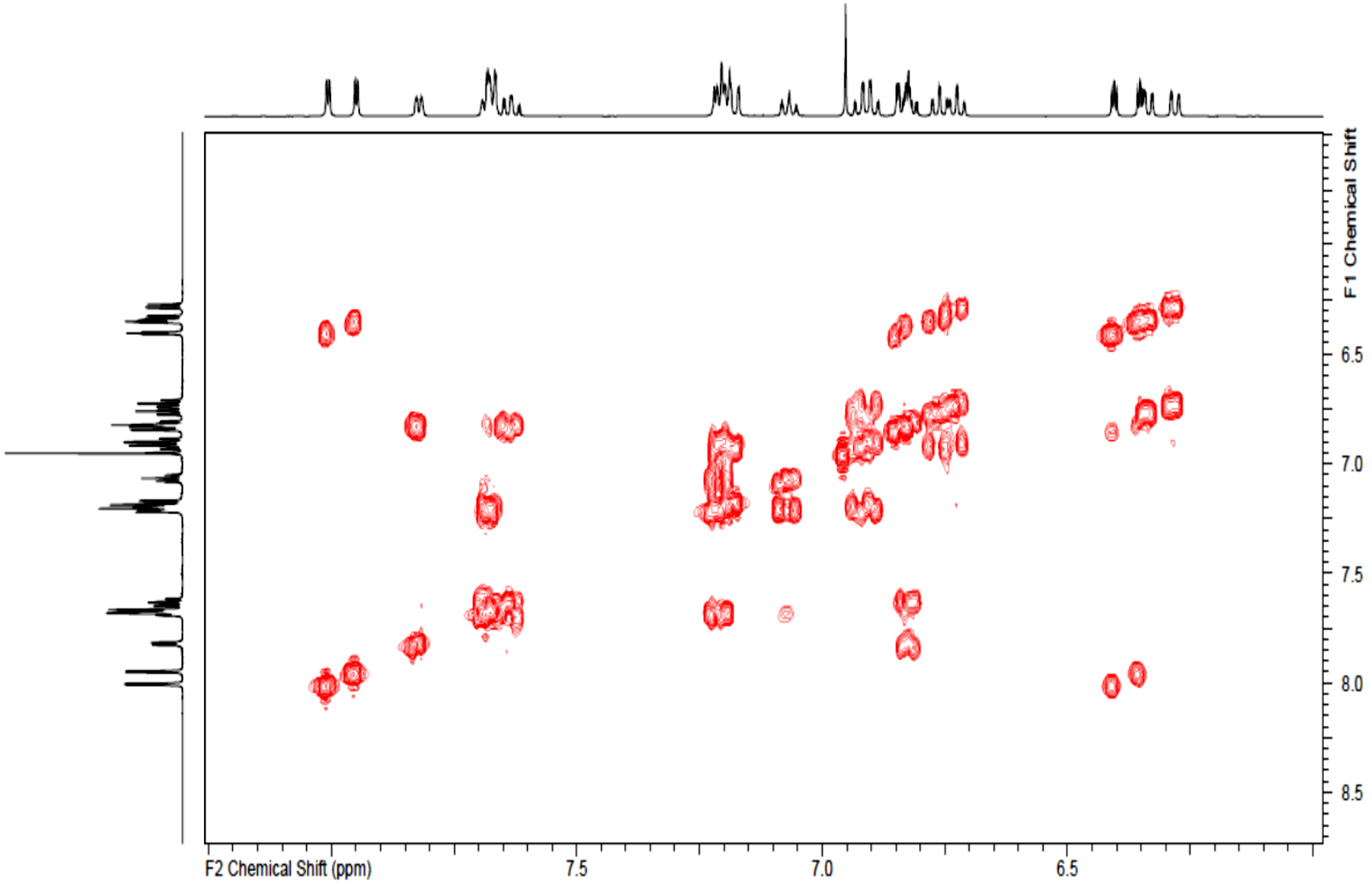
Section of ^{13}**C DEPT** spectrum of **3aL$_3$**



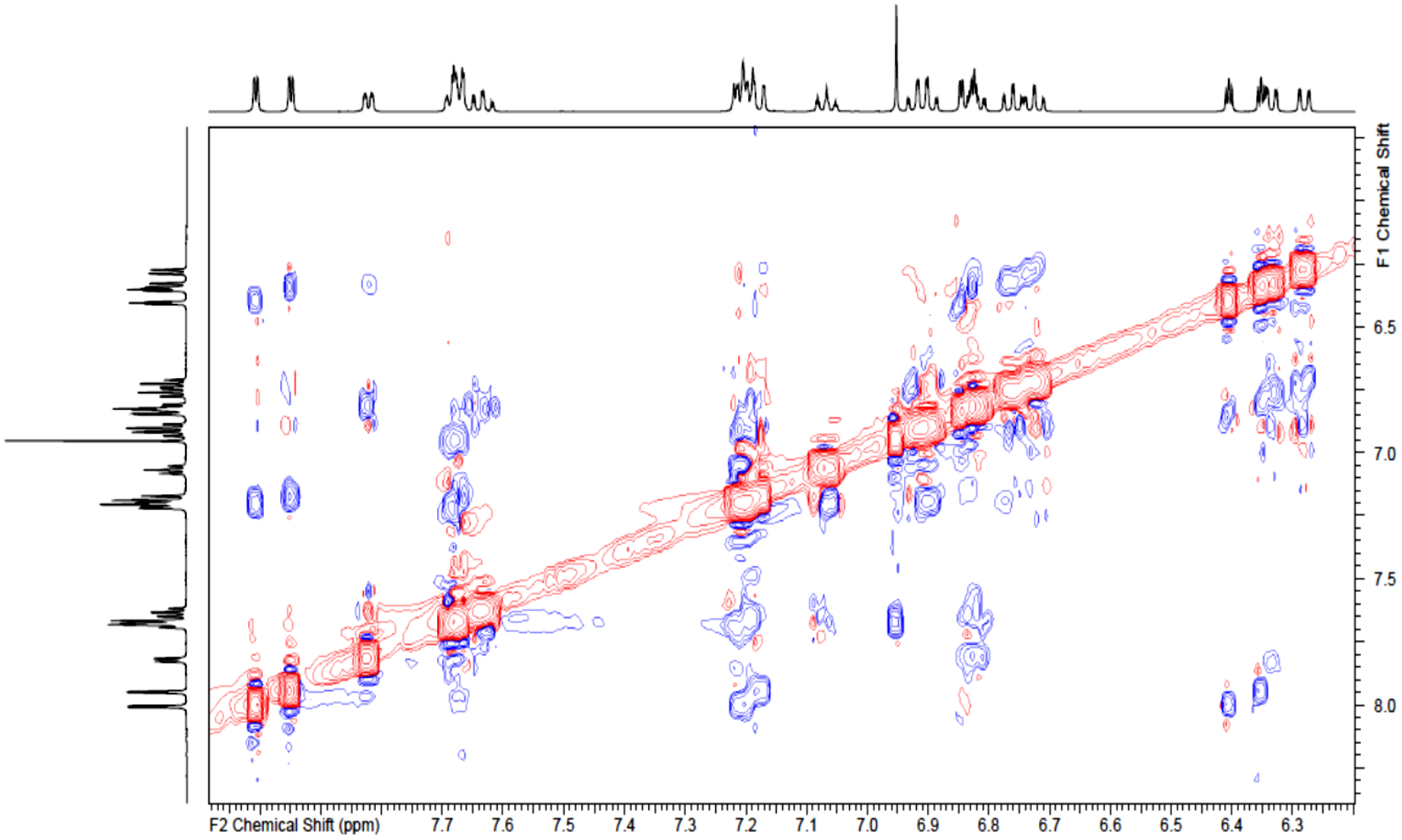
Section of the **TOCSY** spectrum of **3aL$_3$**



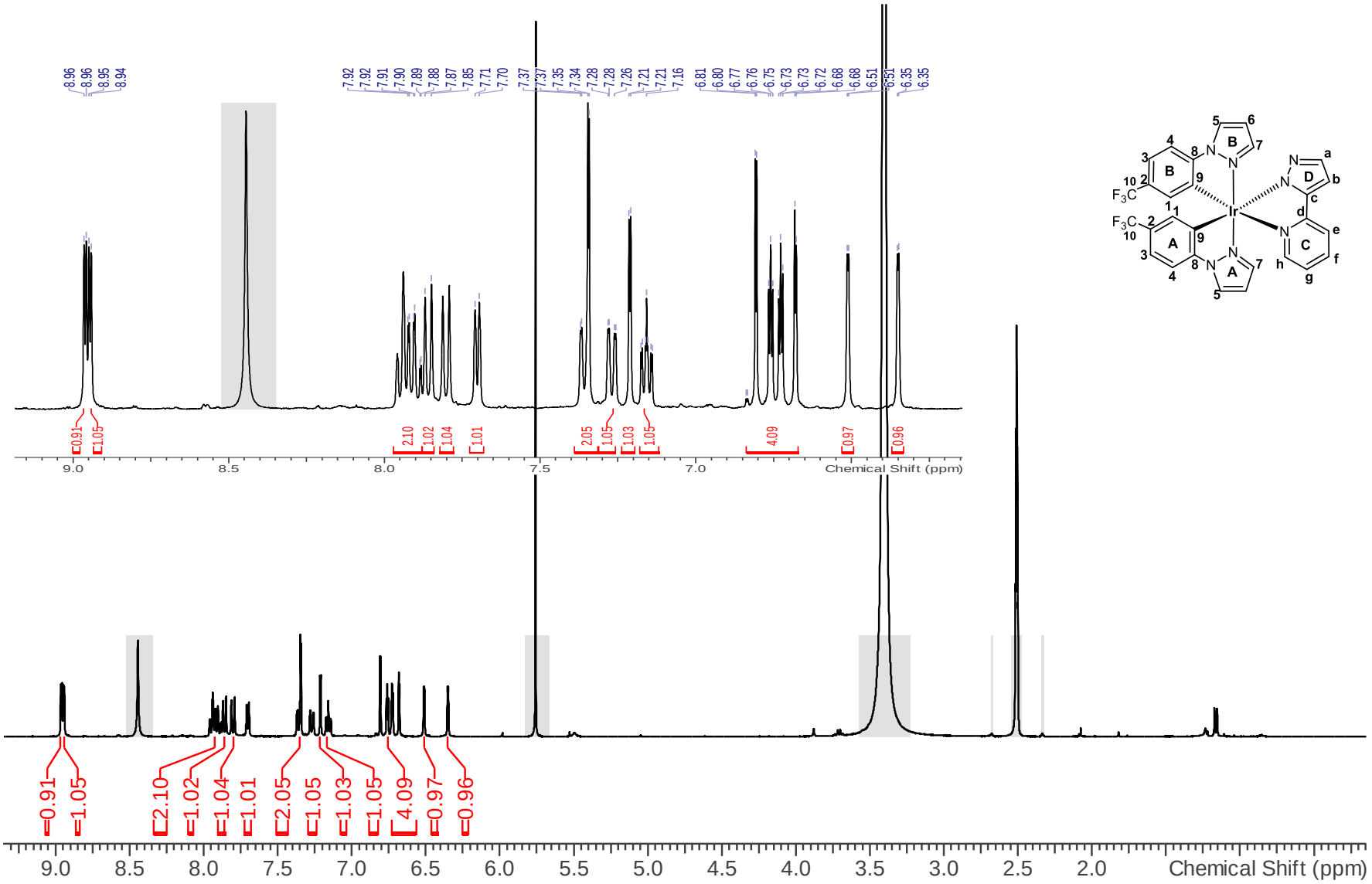
Section of the **COSY** spectrum of **3aL$_3$**



Section of the **NOESY** spectrum of **3aL$_3$**

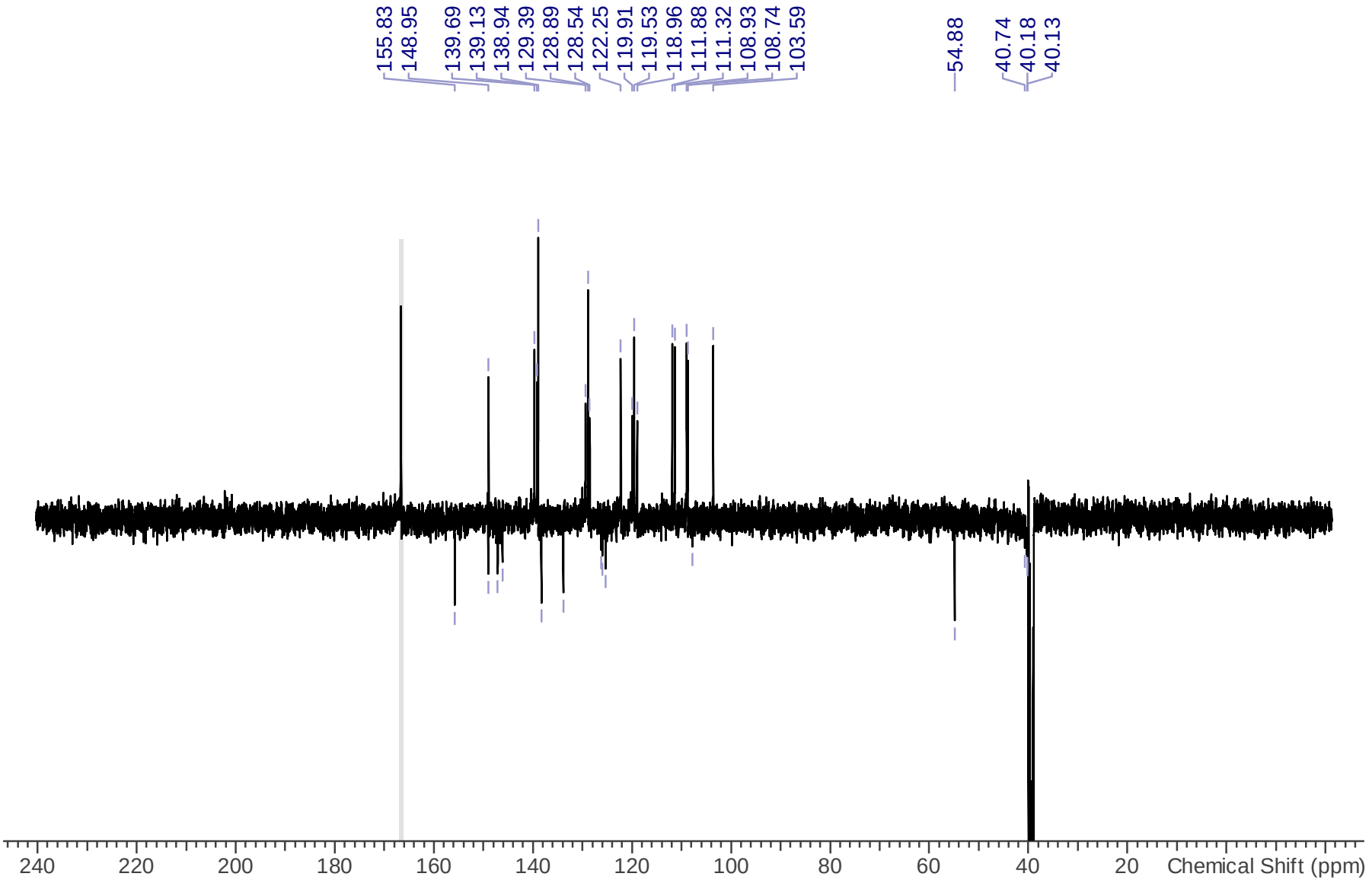


^{1}HNMR spectrum of **3bL$_1$**



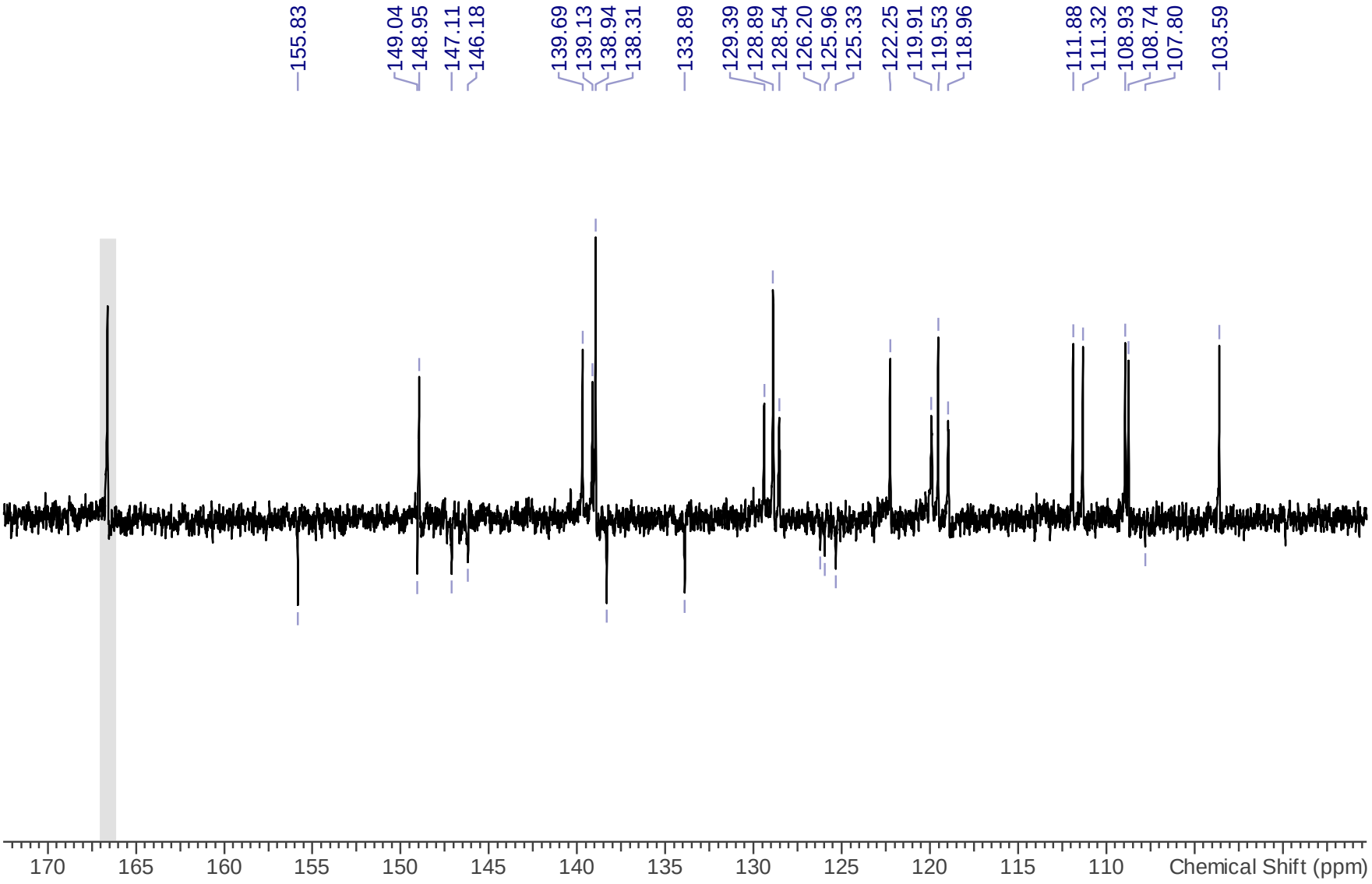
HC(O)NH2 at 8.4 ppm is an impurity

^{13}C DEPT NMR spectrum of **3bL$_1$**

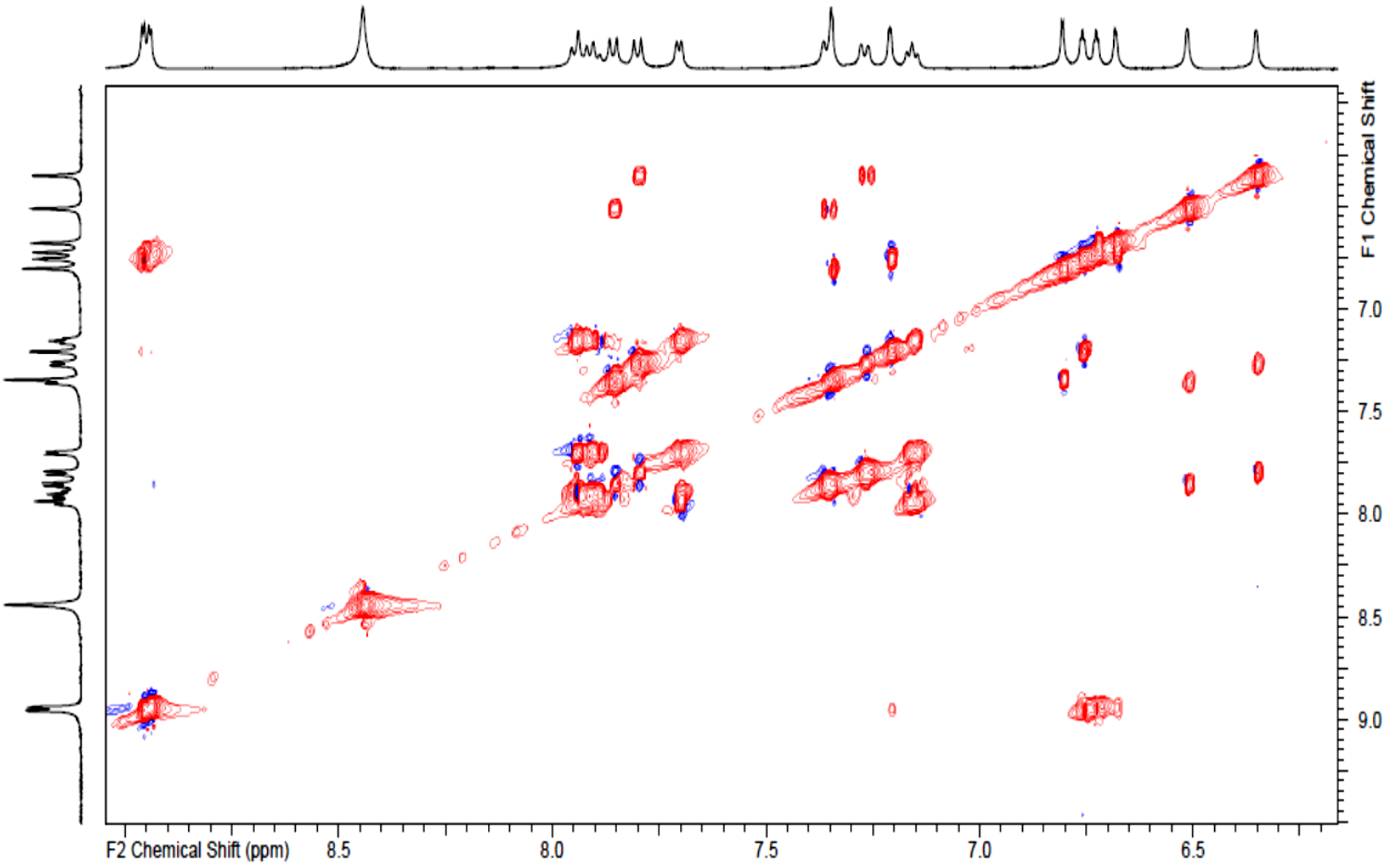


HC(O)NH2 at 166 ppm is an impurity

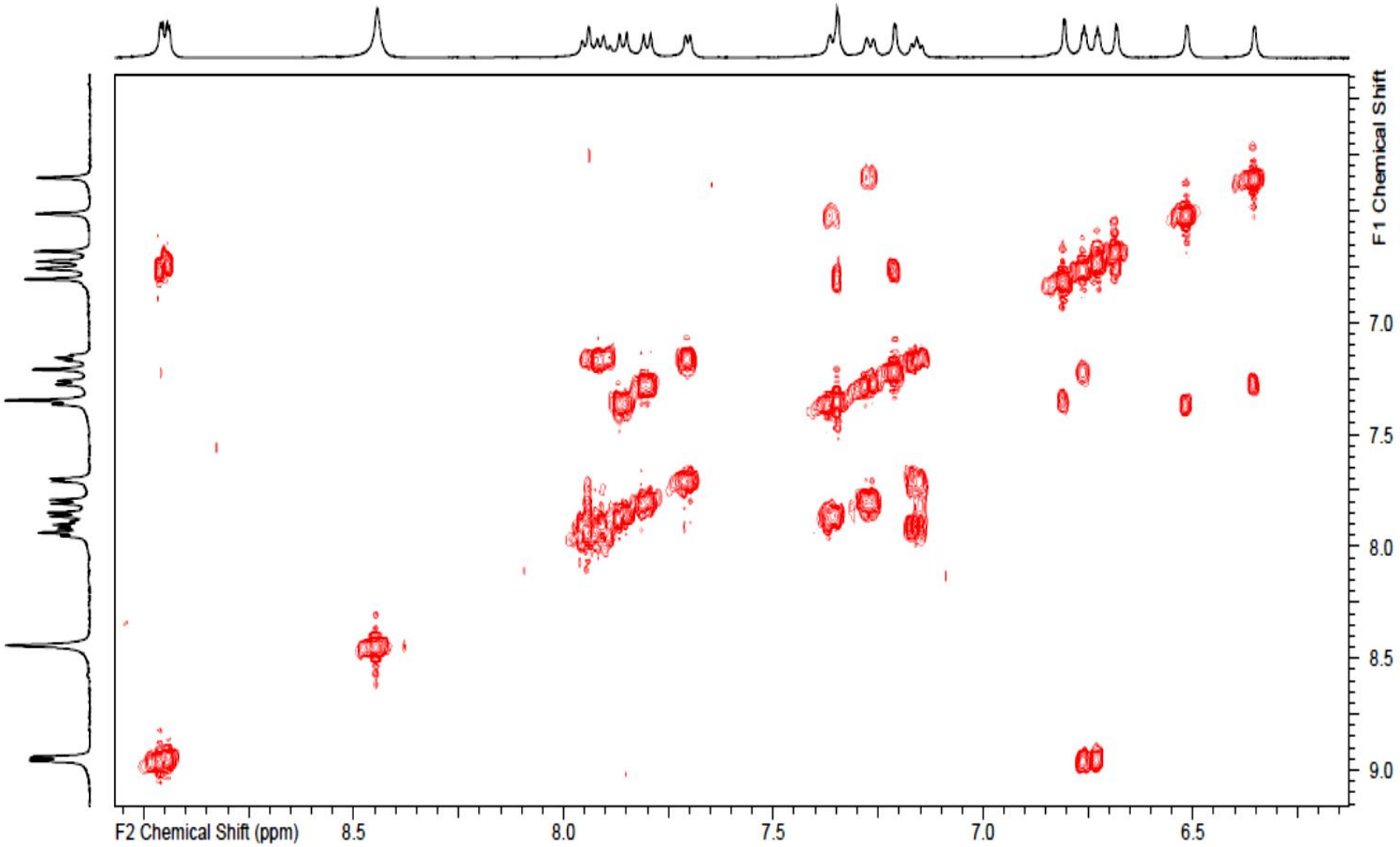
Section of ^{13}**C DEPT** NMR spectrum of **3bL$_1$**



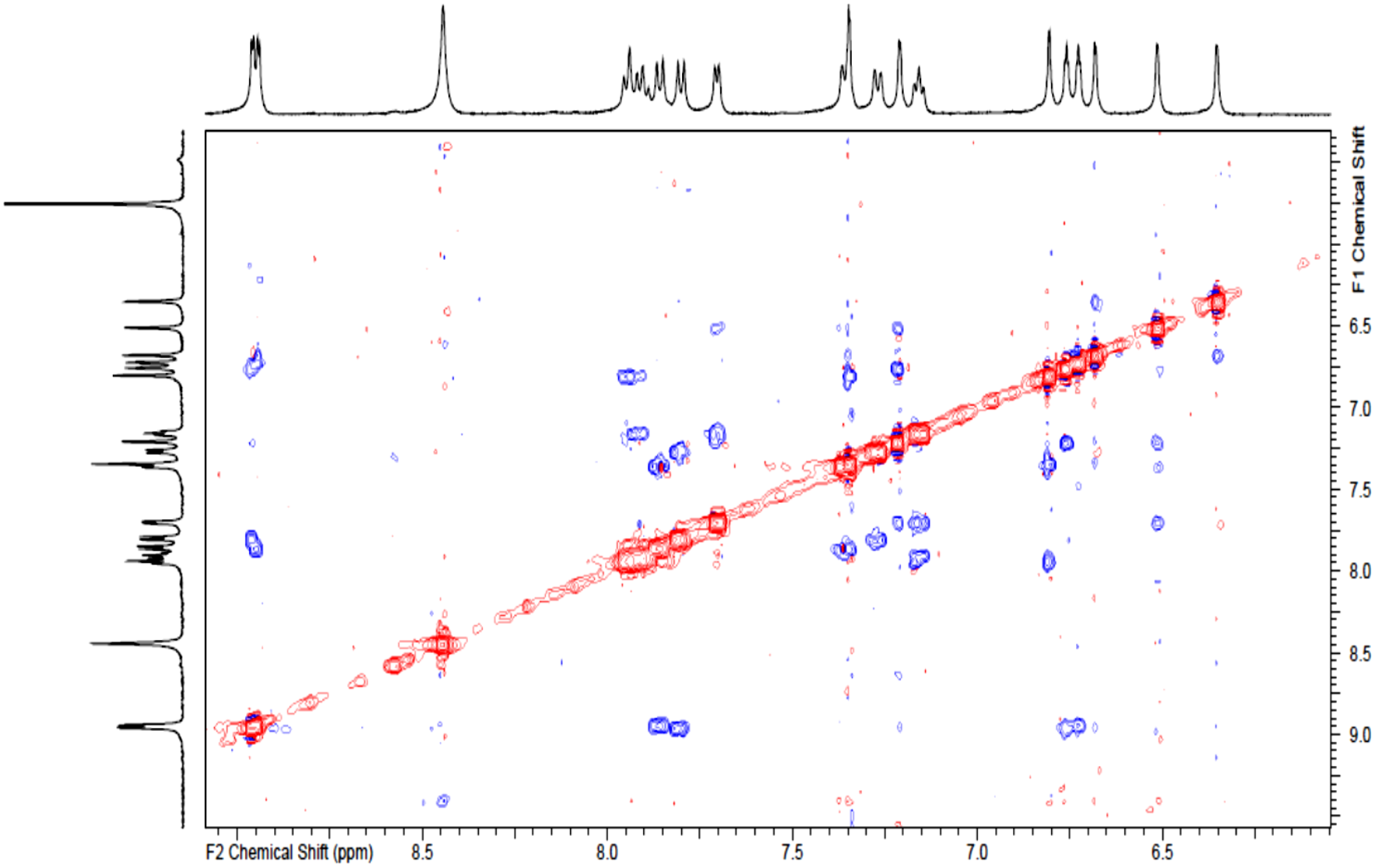
Section of the **TOCSY** spectrum of **3bL$_1$**



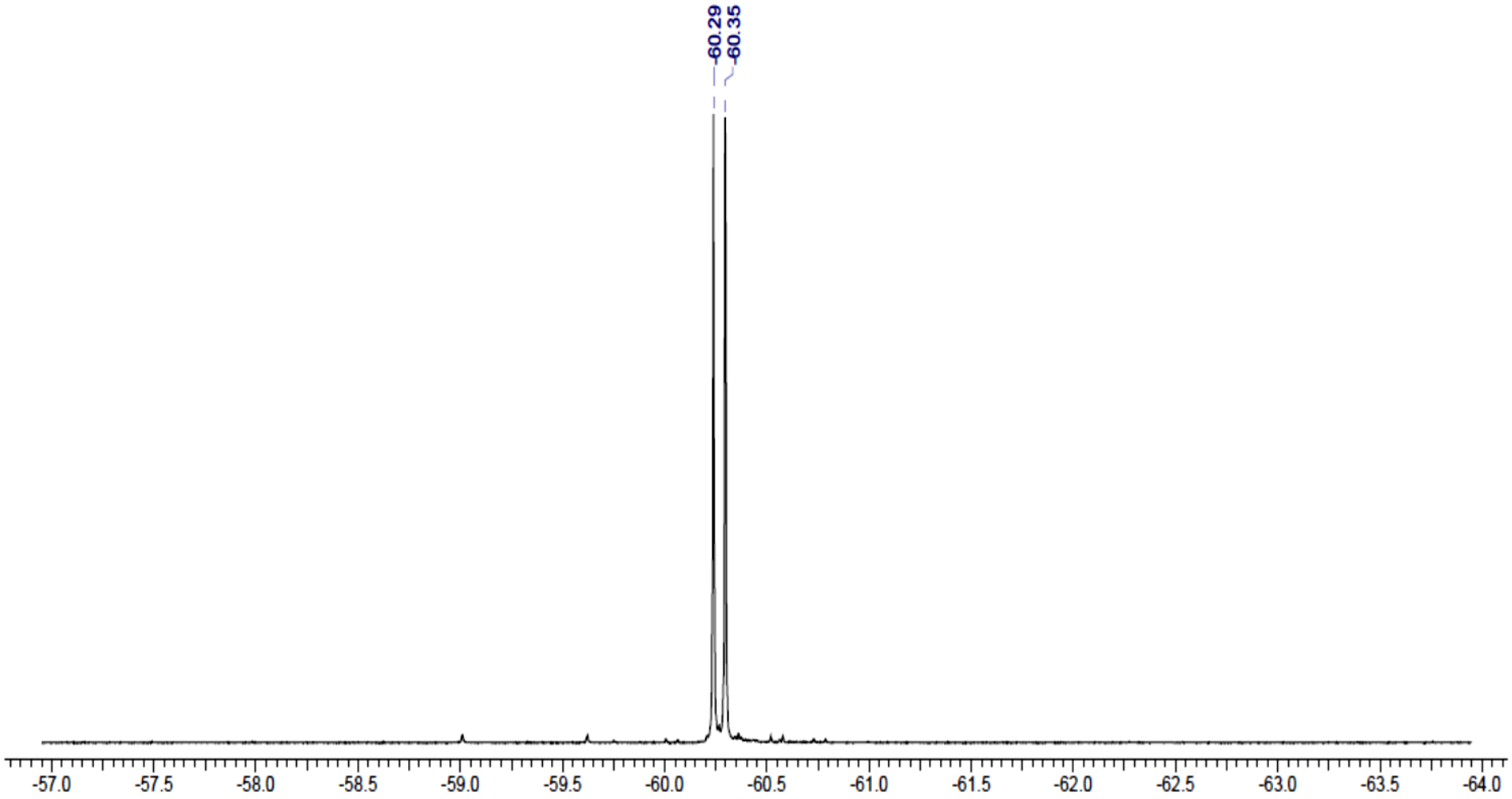
Section of the **COSY** spectrum of **3bL$_1$**



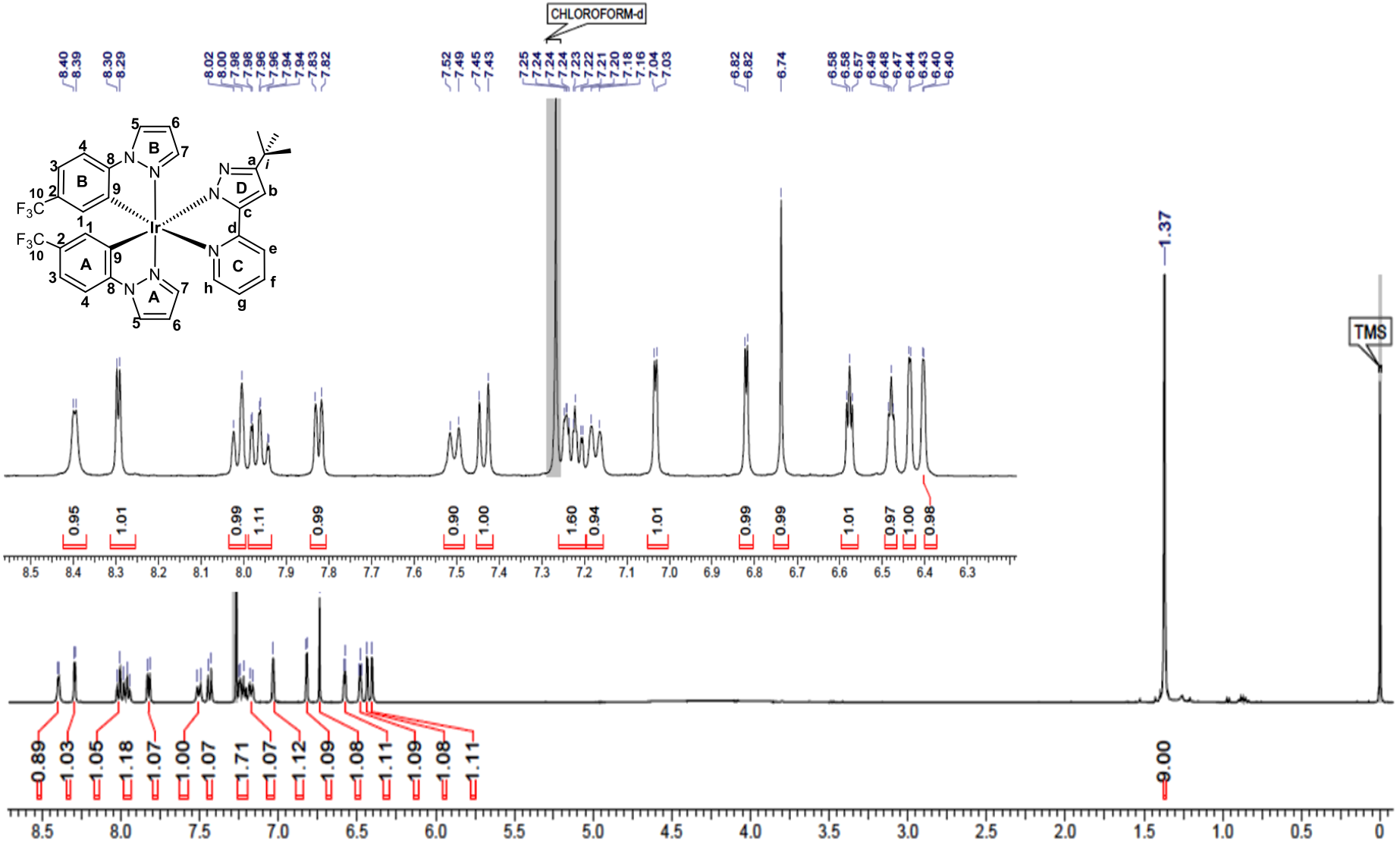
Section of the **NOESY** spectrum of **3bL$_1$**



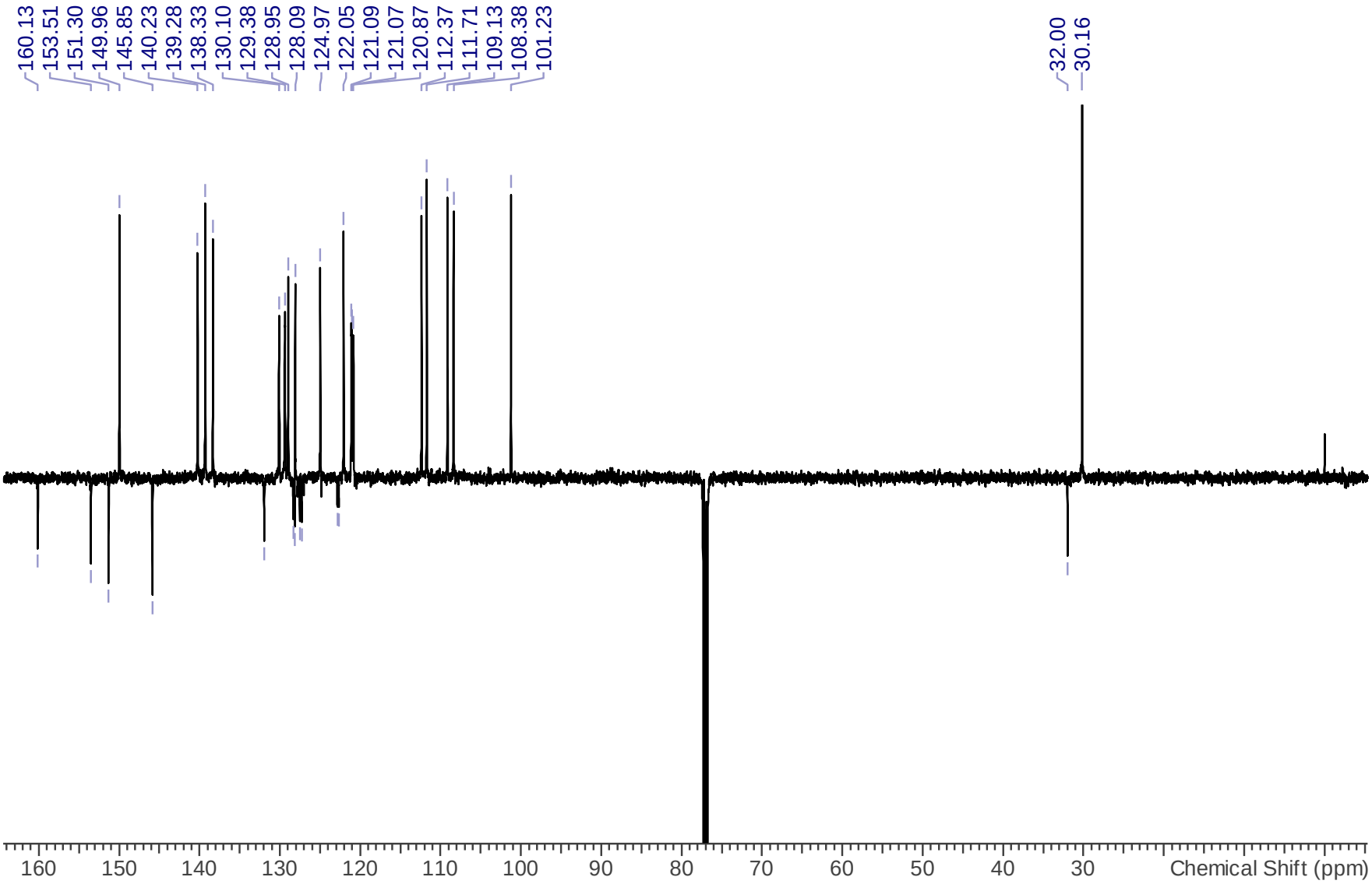
^{19}F NMR spectrum of **3bL$_1$**



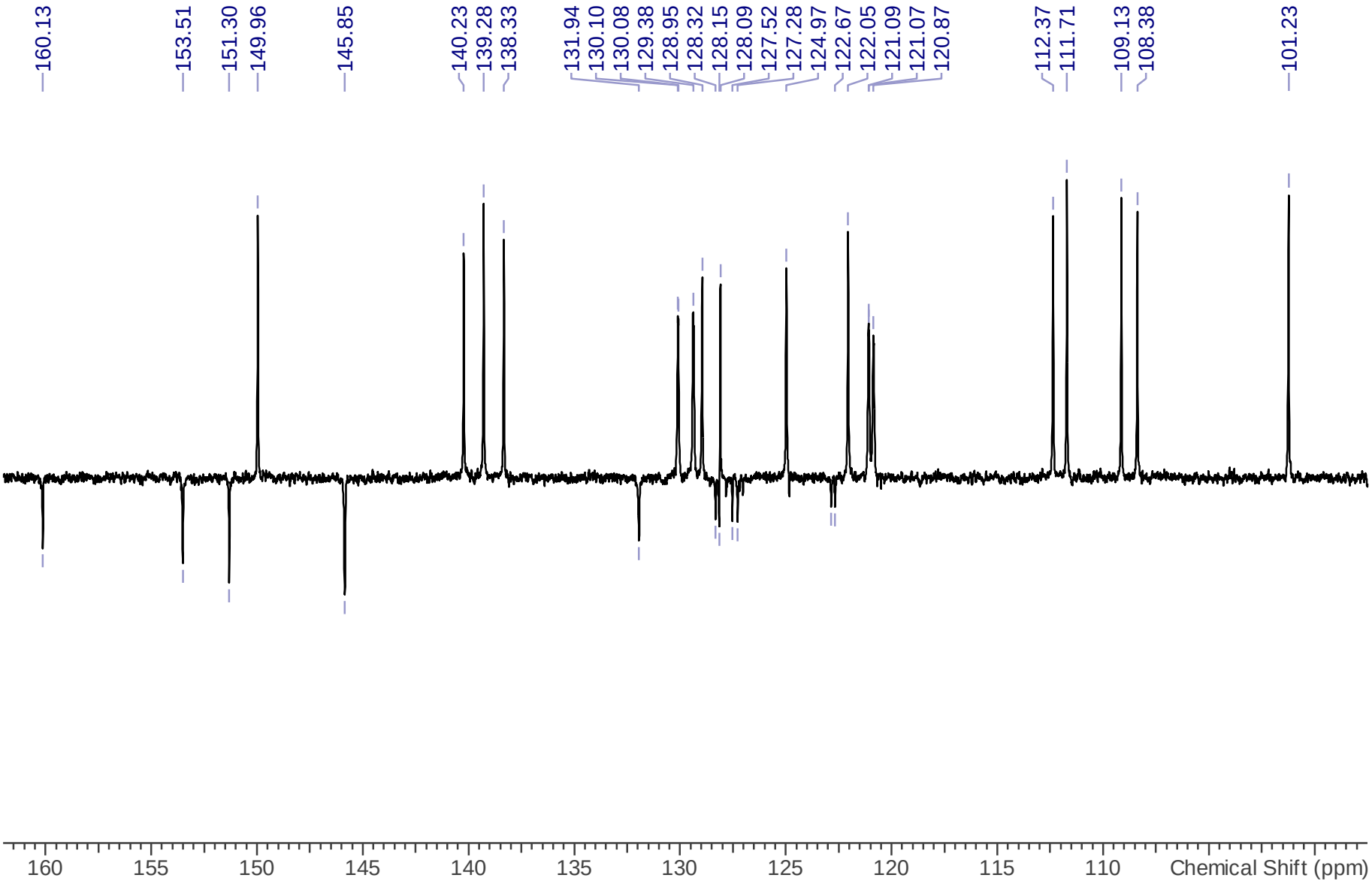
^{1}H NMR spectrum of **3bL$_2$**



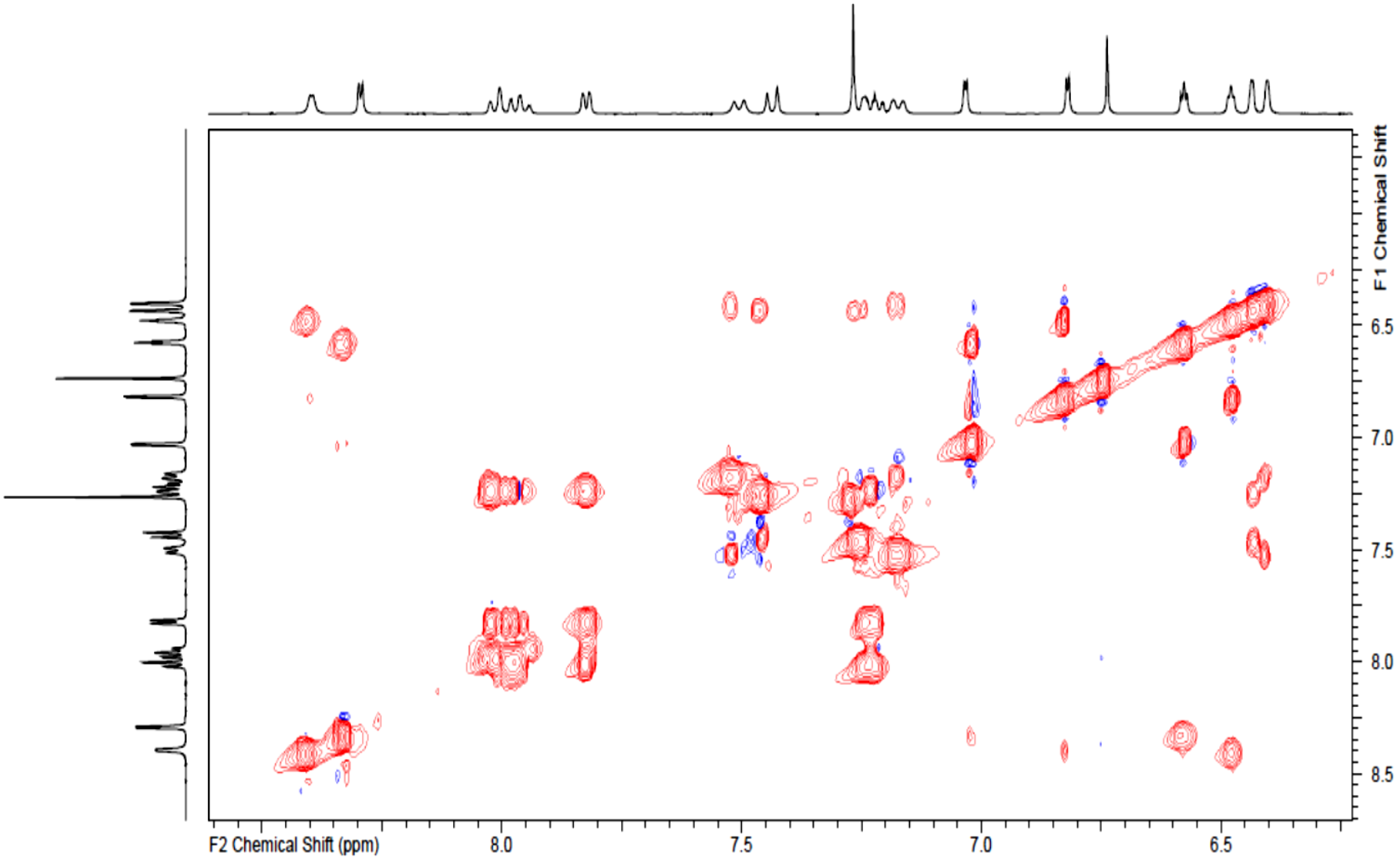
^{13}C DEPT NMR spectrum of **3bL$_2$**



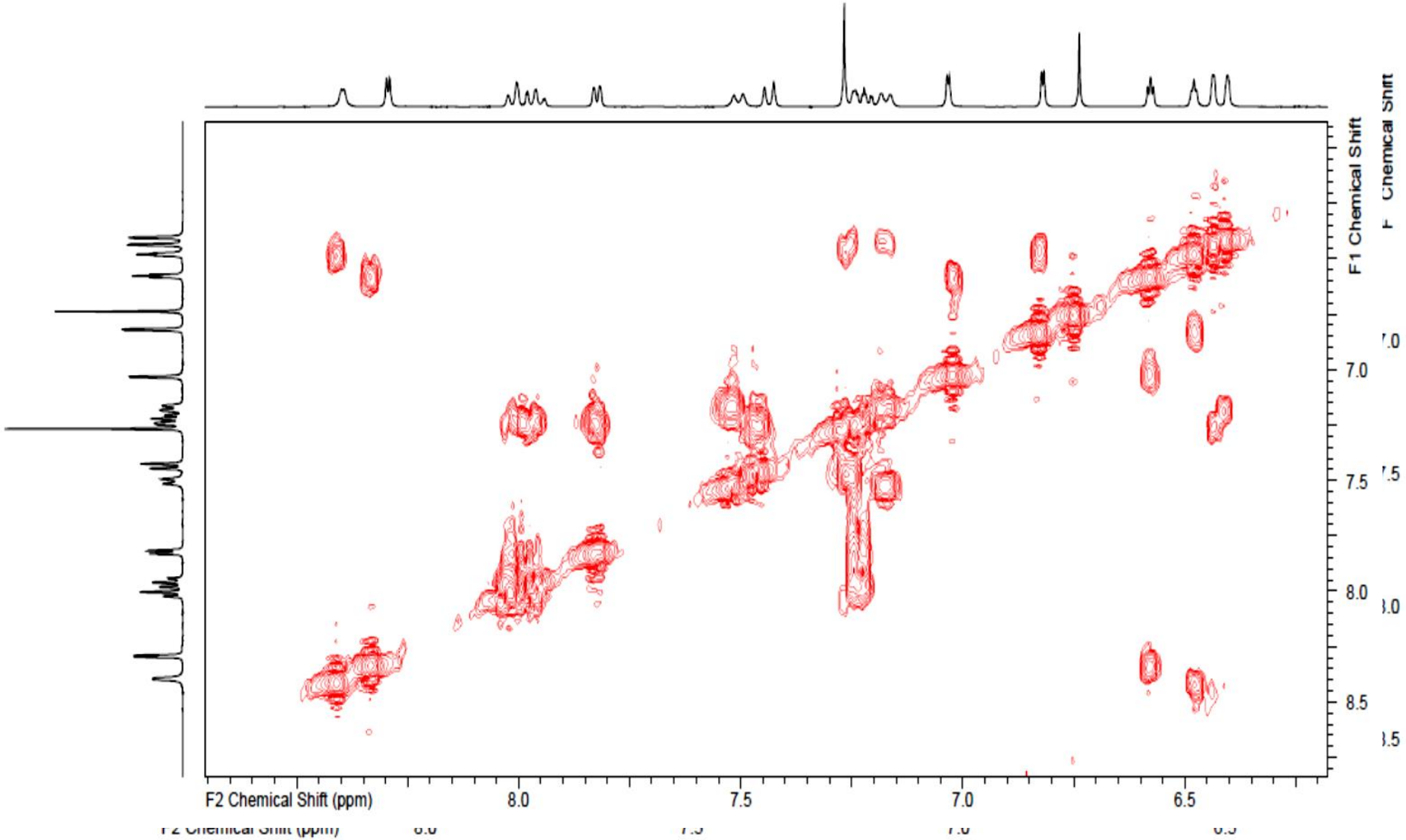
Section of ^{13}**C DEPT** spectrum of **3bL**$_2$



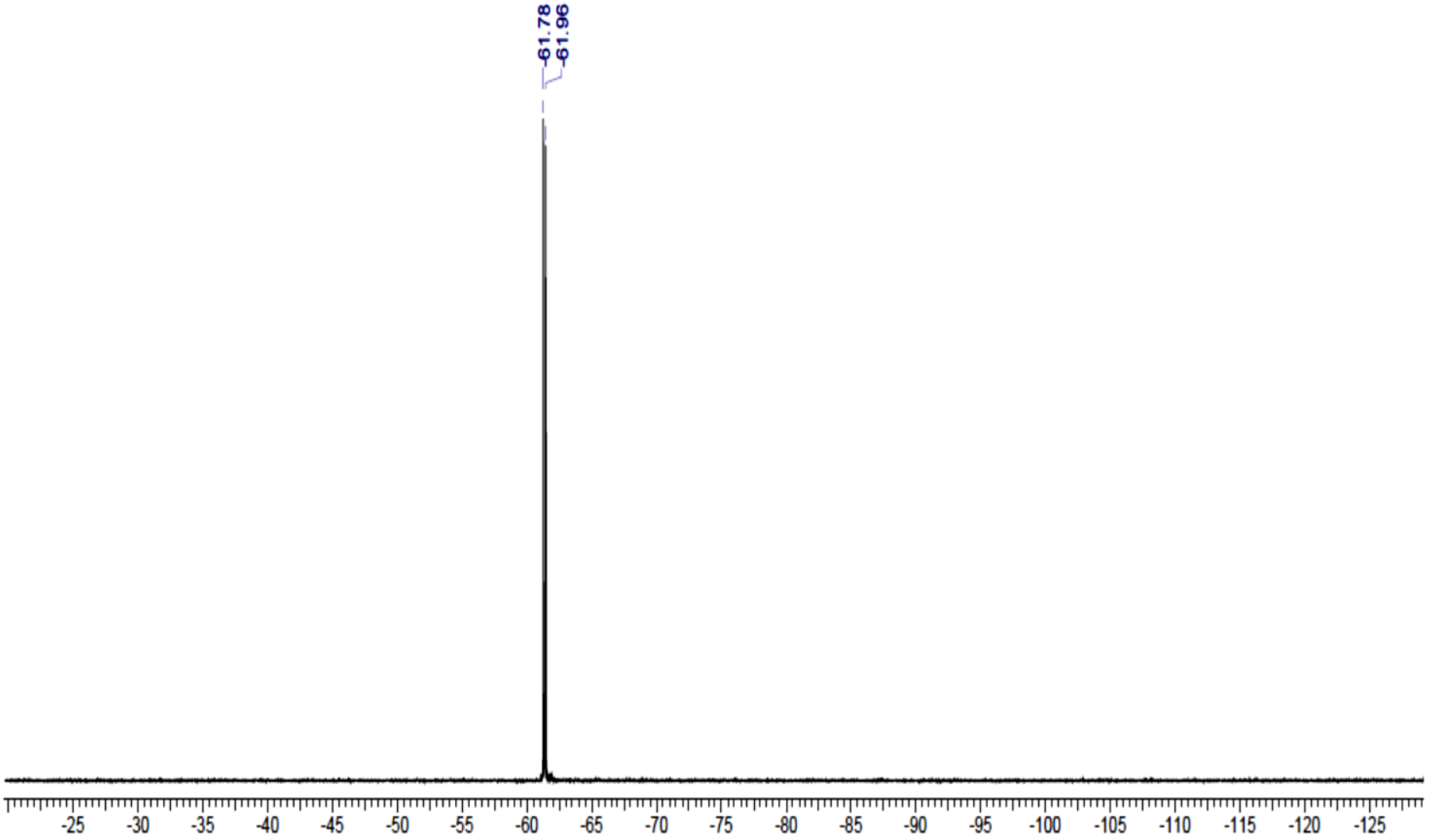
Section of the **TOCSY** spectrum of **3bL$_2$**



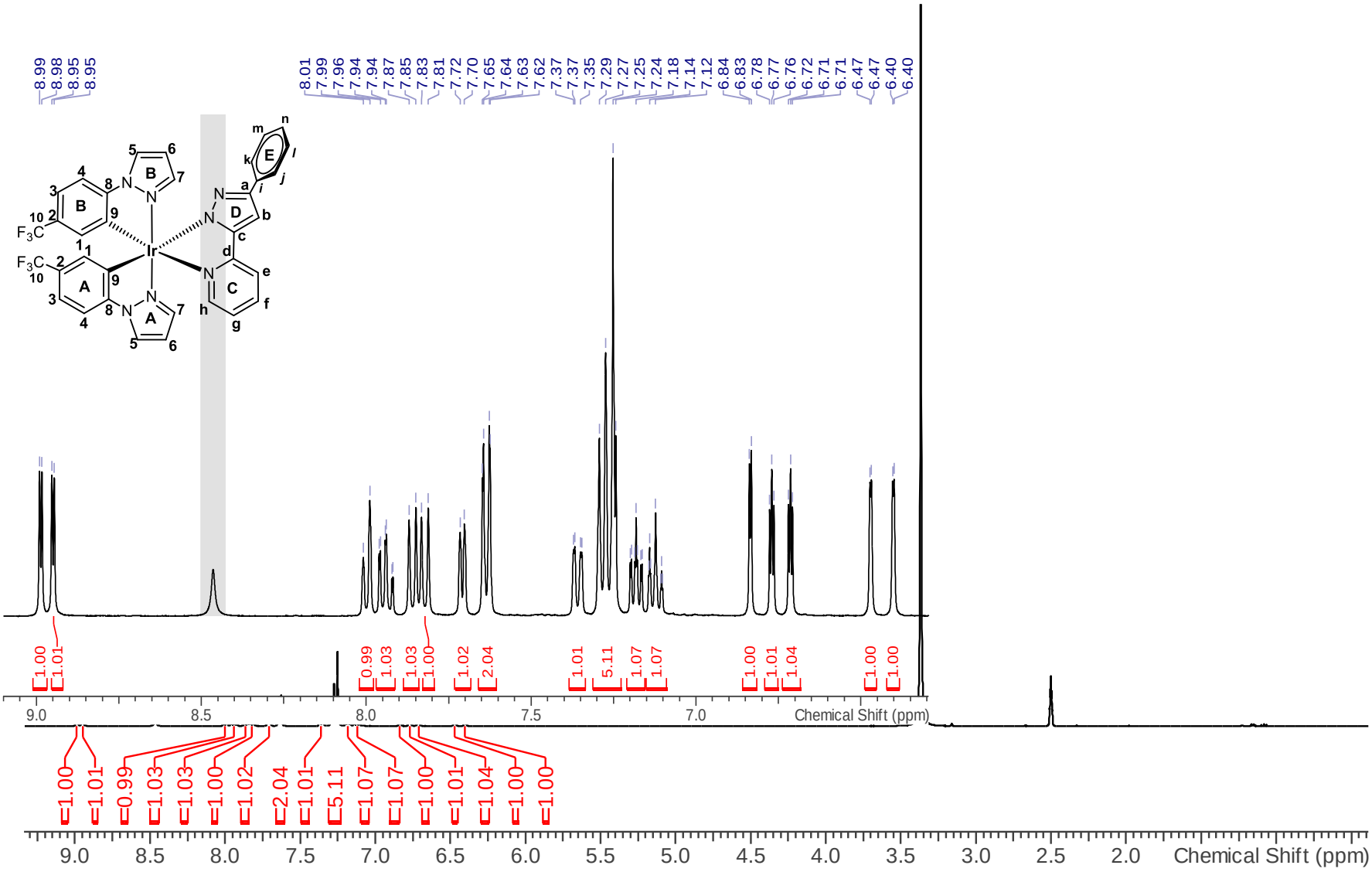
Section of the **COSY** spectrum of **3bL**$_2$



^{19}F NMR spectrum of **3bL$_2$**

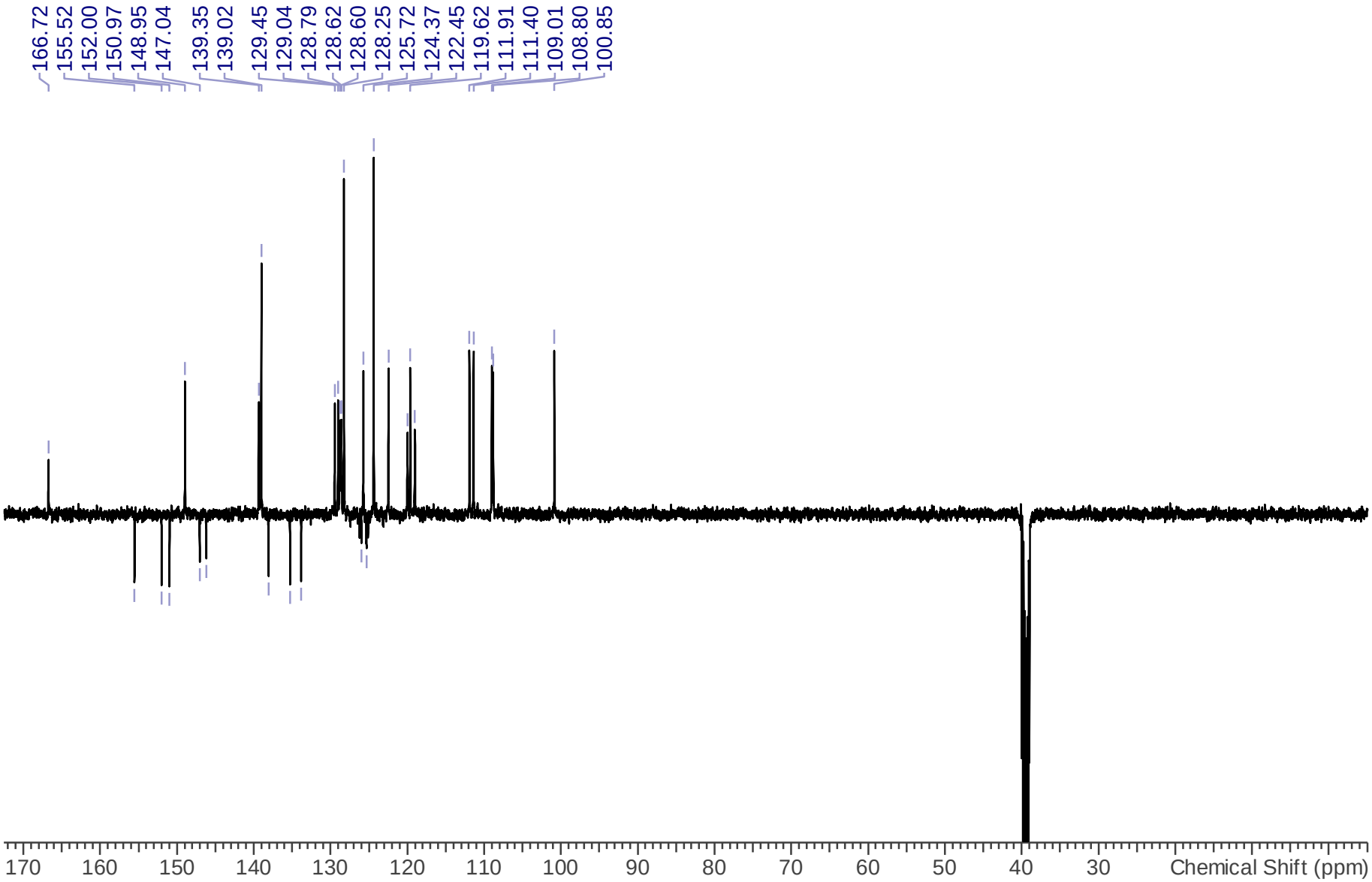


^{1}H NMR spectrum of **3bL$_3$**

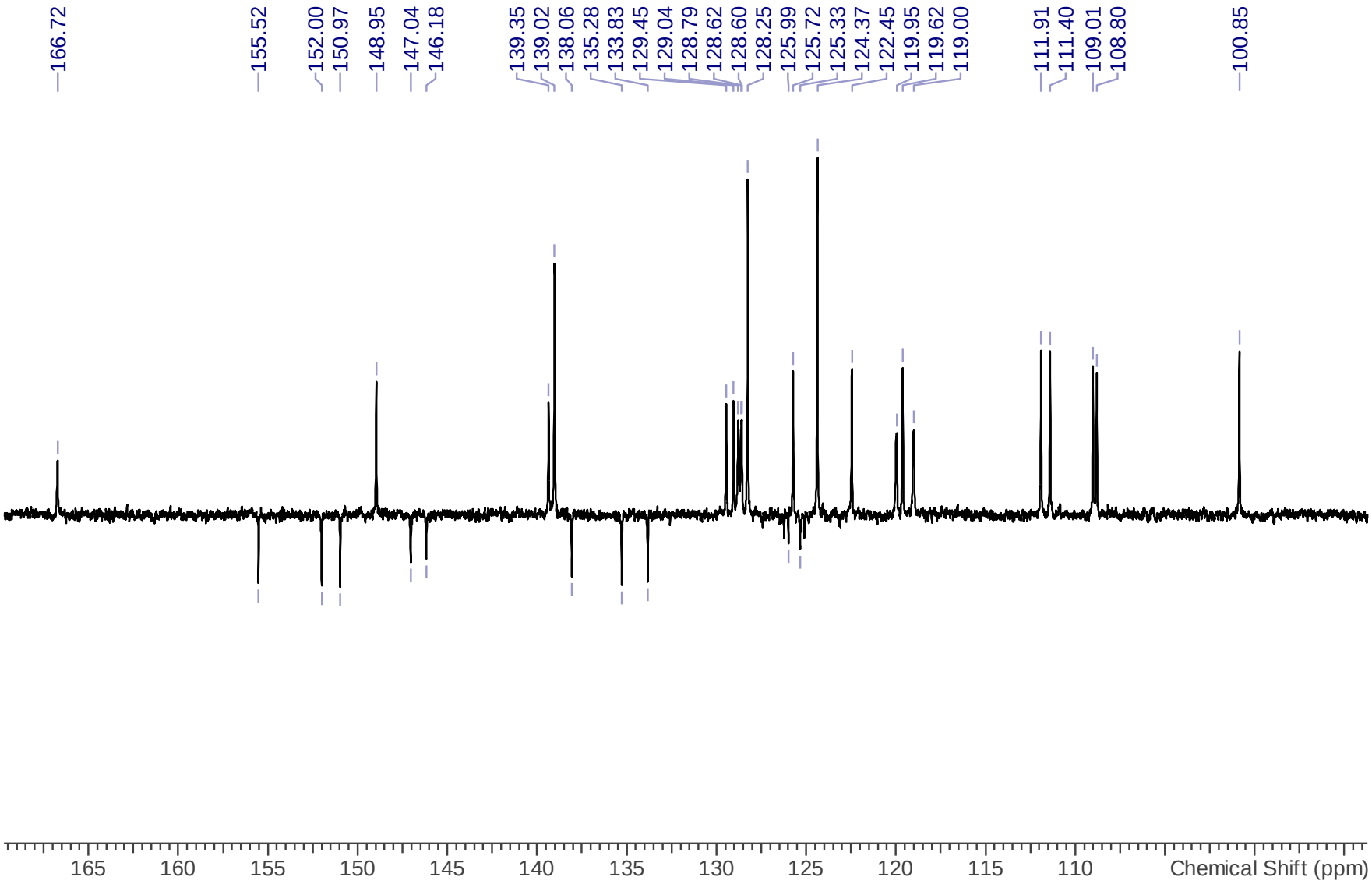


Peak at 8.45 is HC(O)NH2 impurity in the solvent

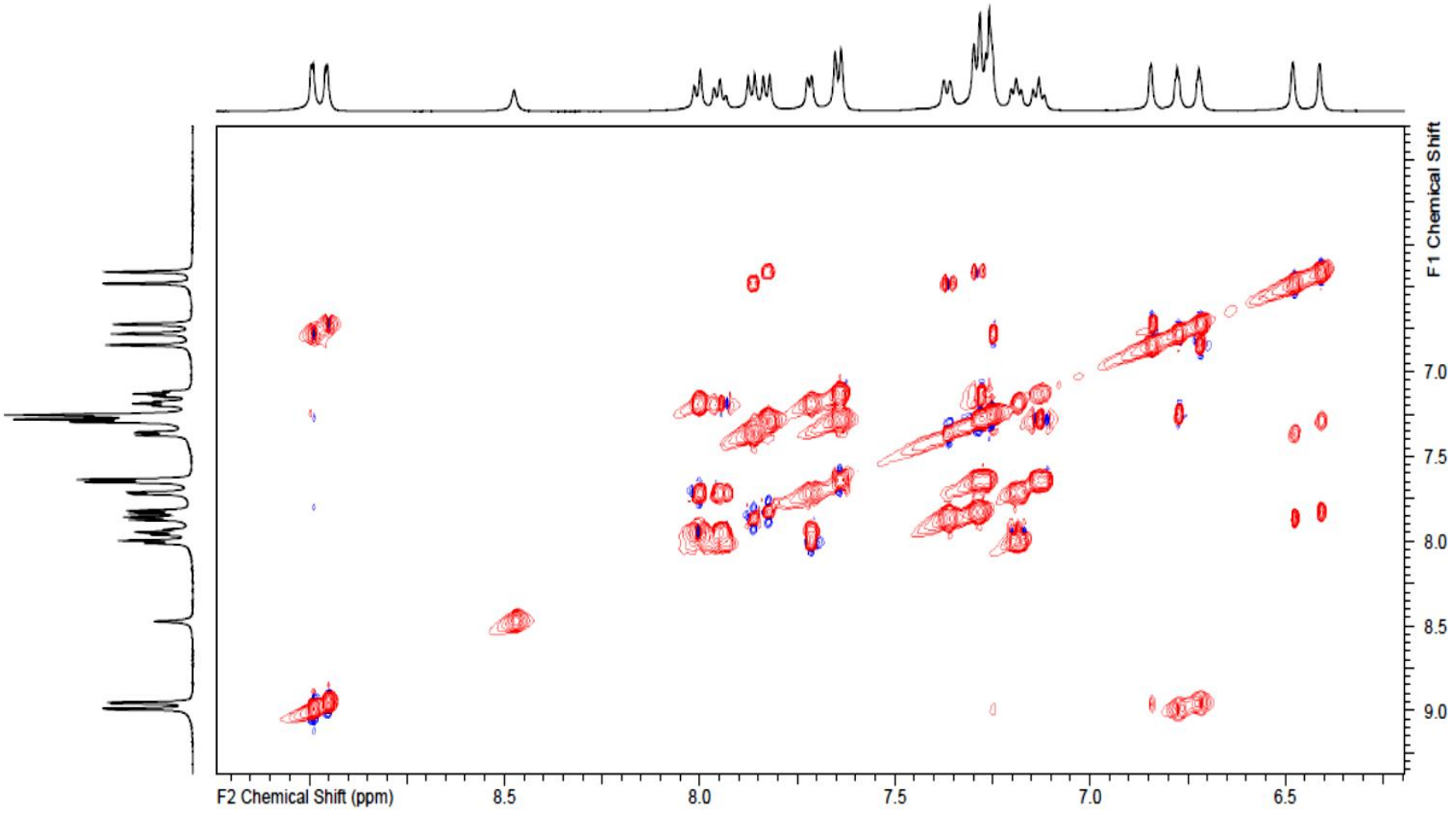
^{13}C DEPT spectrum of **3bL$_3$**



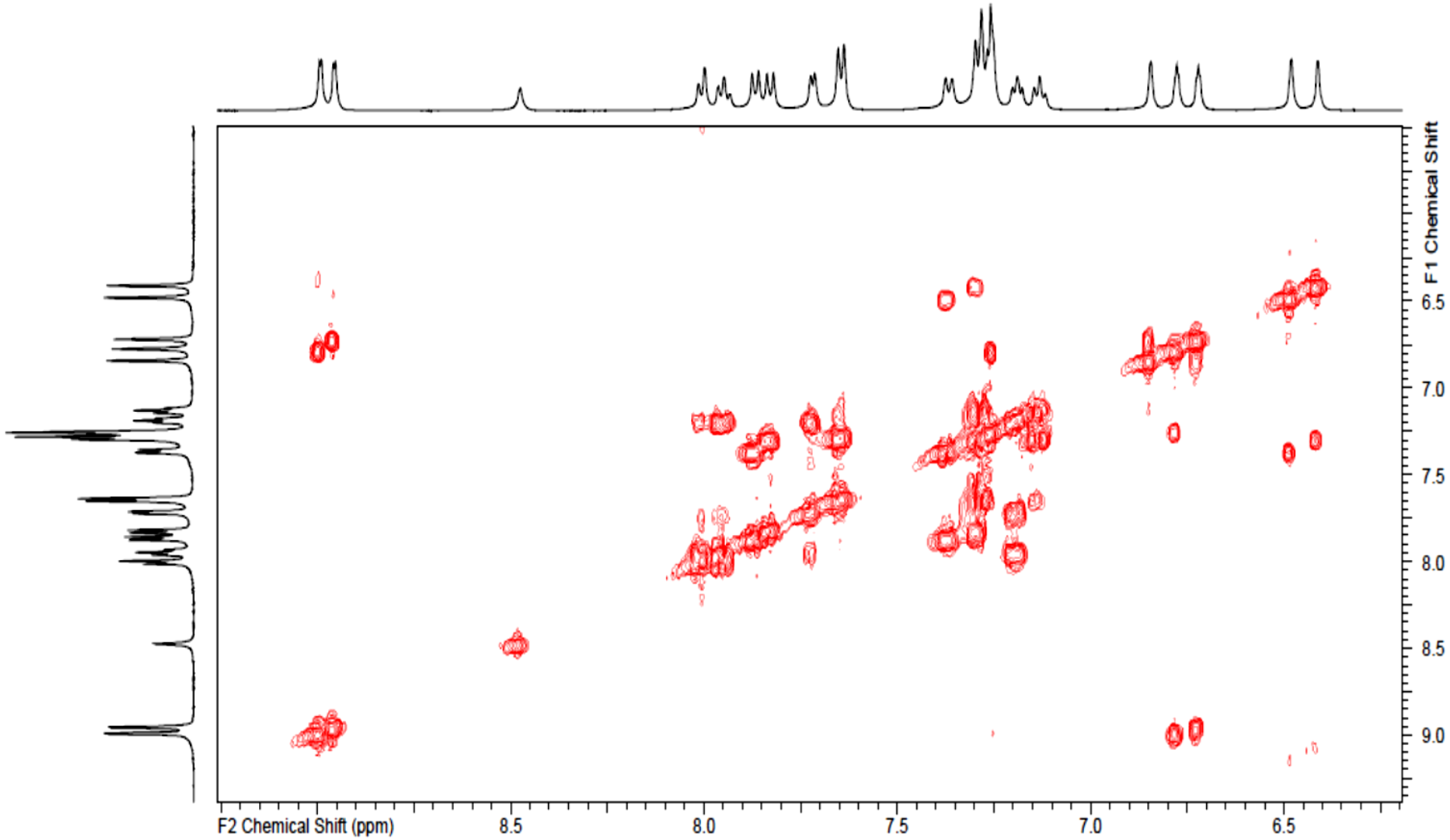
Section of ^{13}**C DEPT** spectrum of **3bL$_3$**



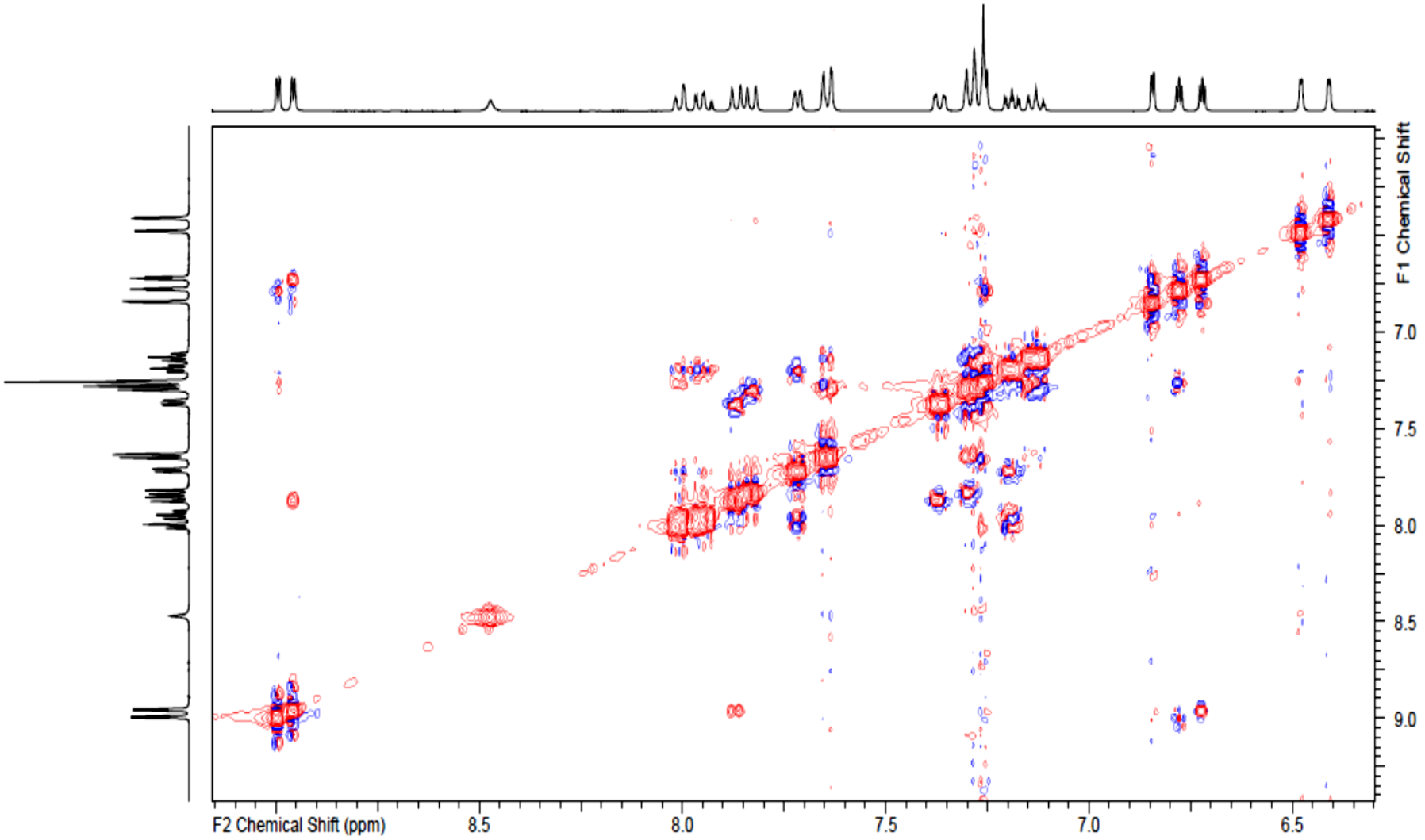
Section of the **TOCSY** spectrum of **3bL$_3$**



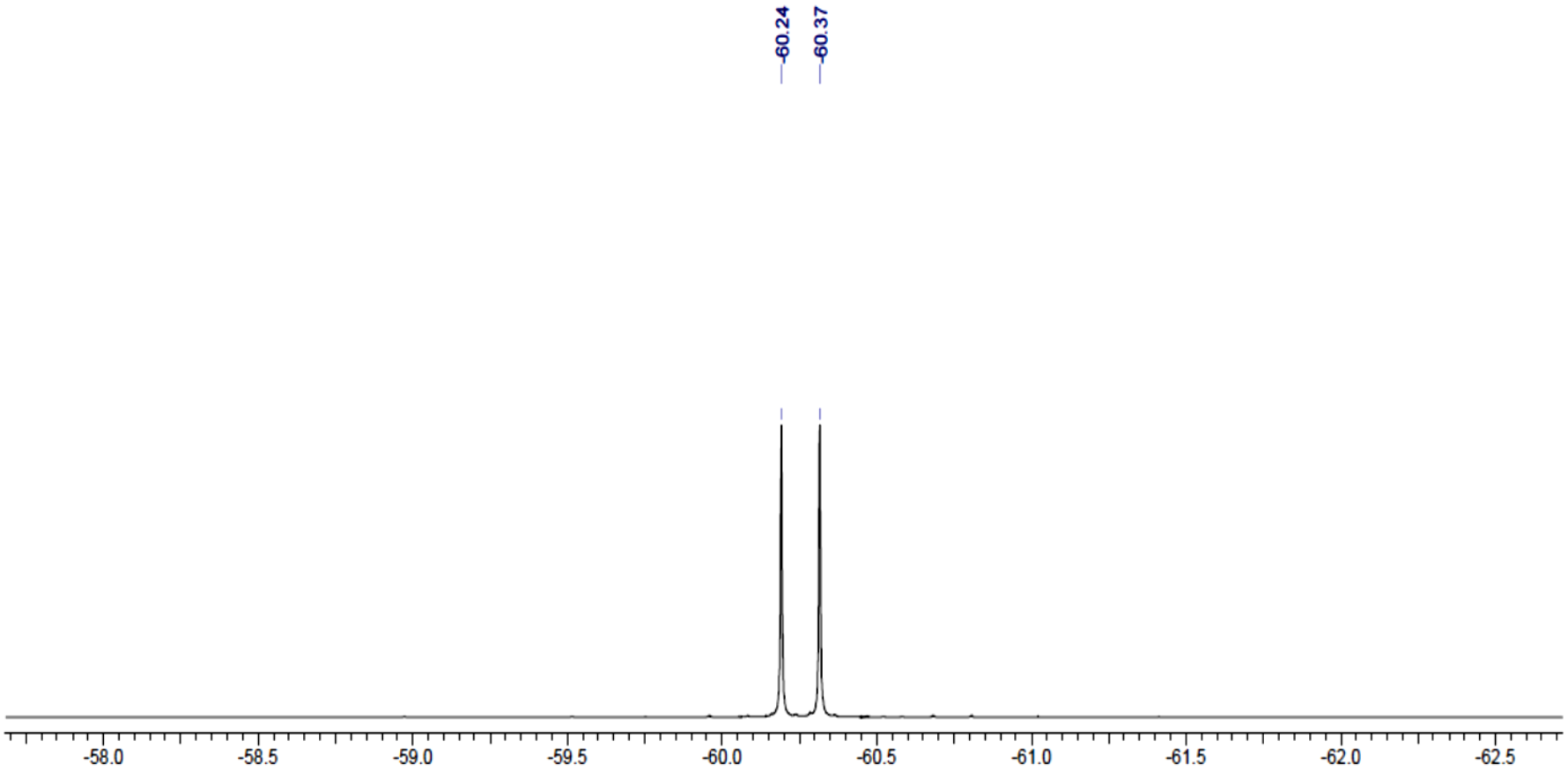
Section of the **COSY** spectrum of **3bL$_3$**



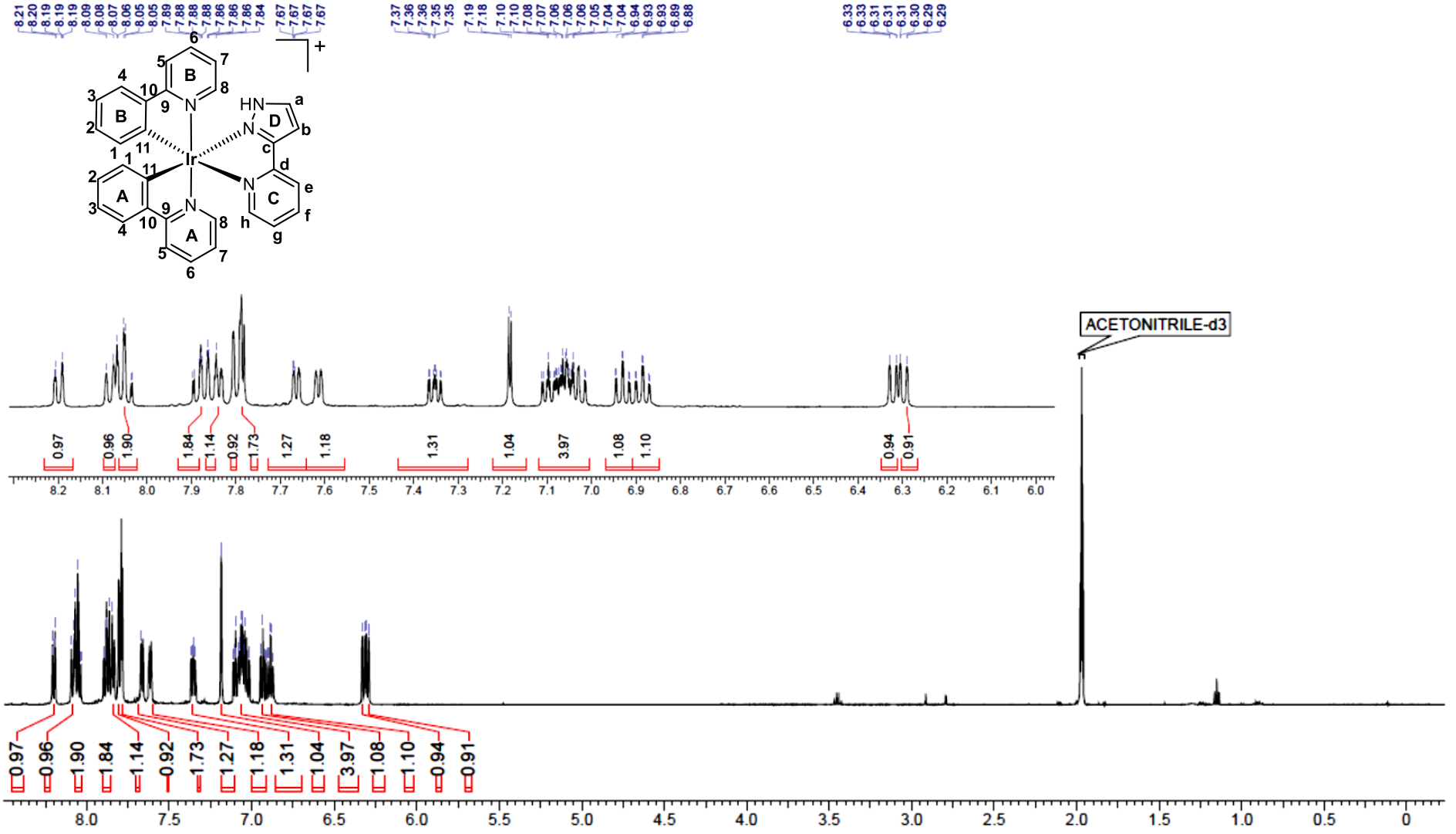
Section of the **NOESY** spectrum of **3bL$_3$**



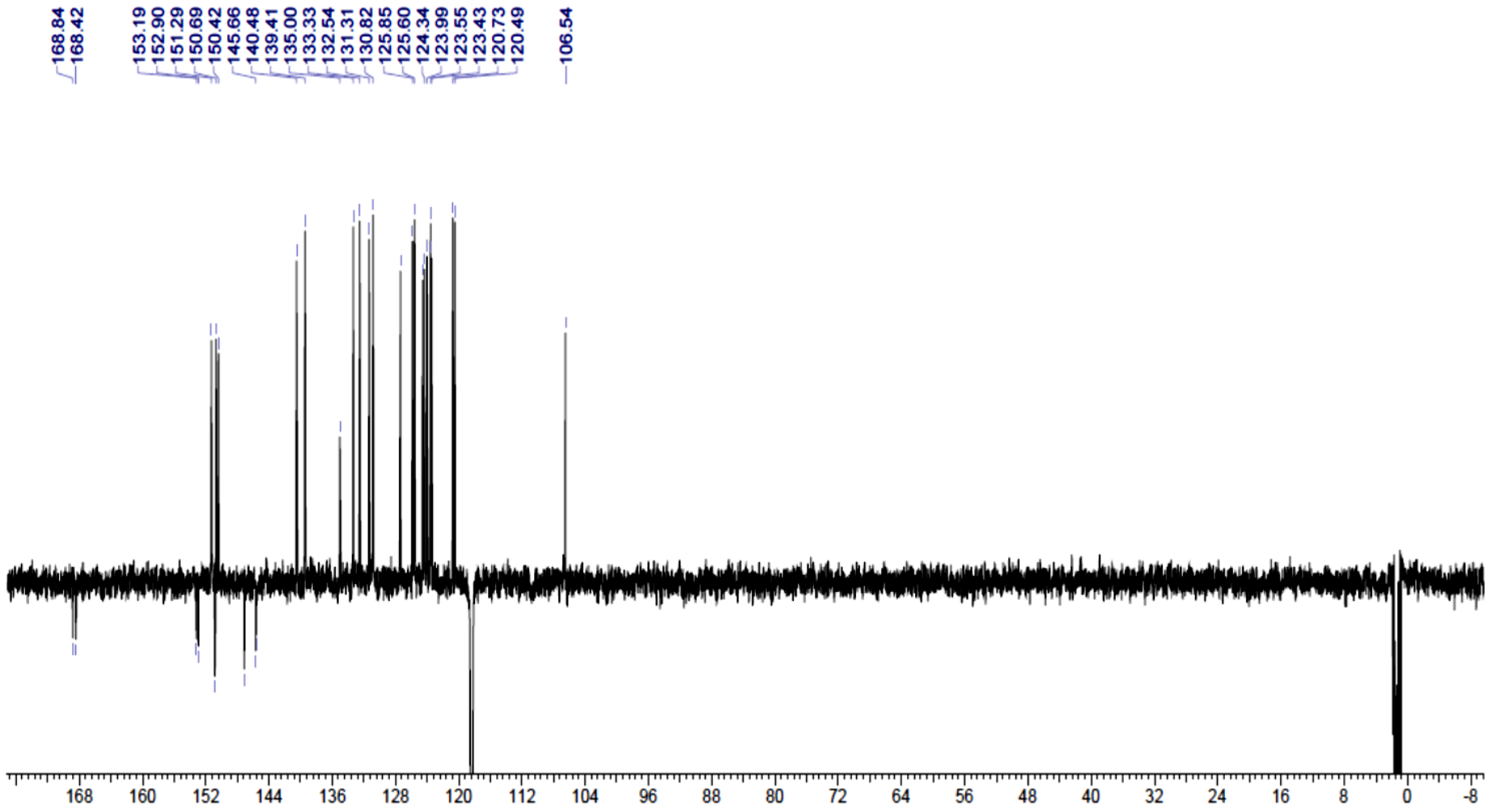
^{19}F NMR spectrum of **3bL$_3$**



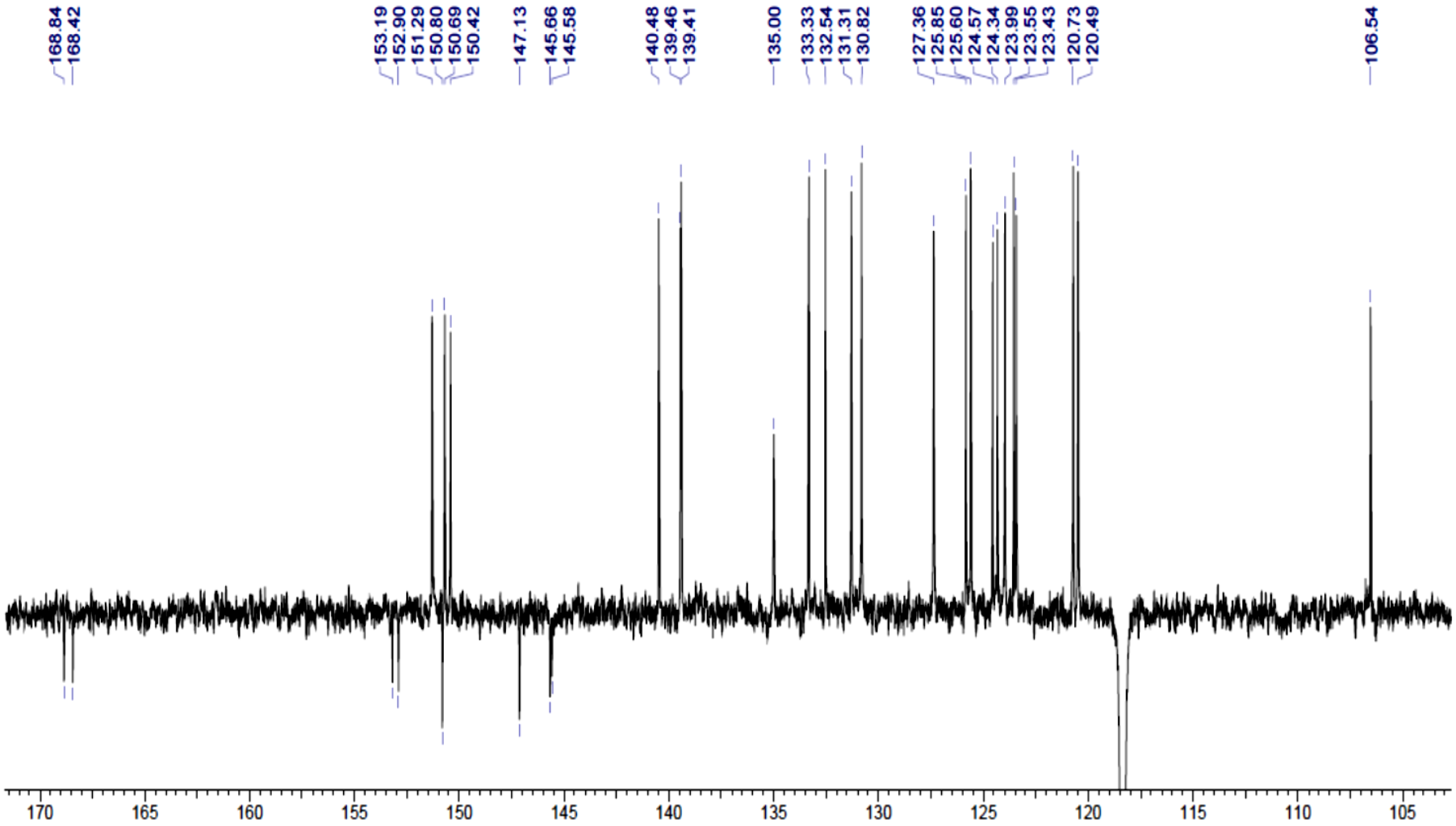
^{1}H NMR spectrum of **2cHL$_1$**



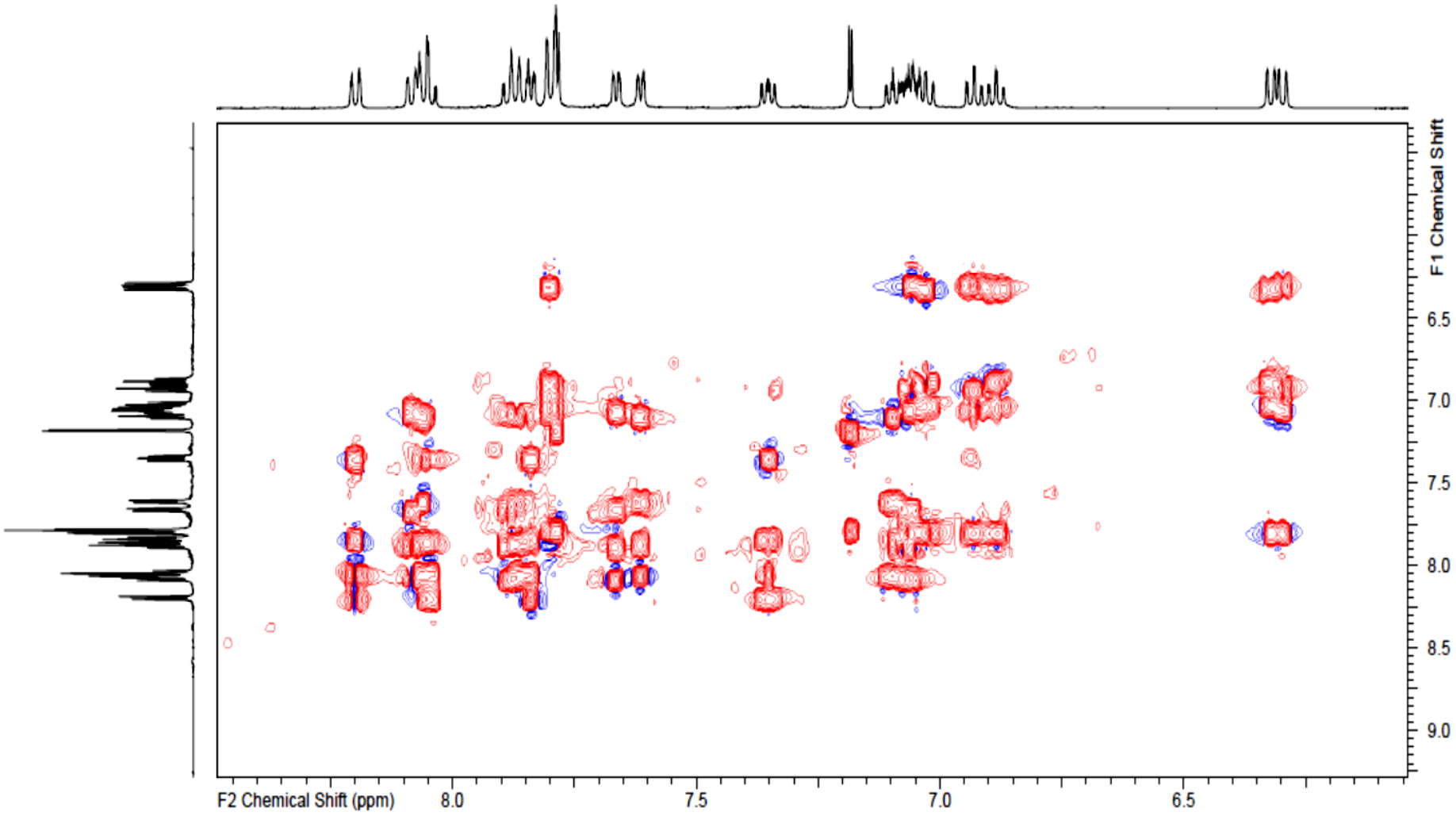
^{13}C NMR APT spectrum of **2cHL$_1$**



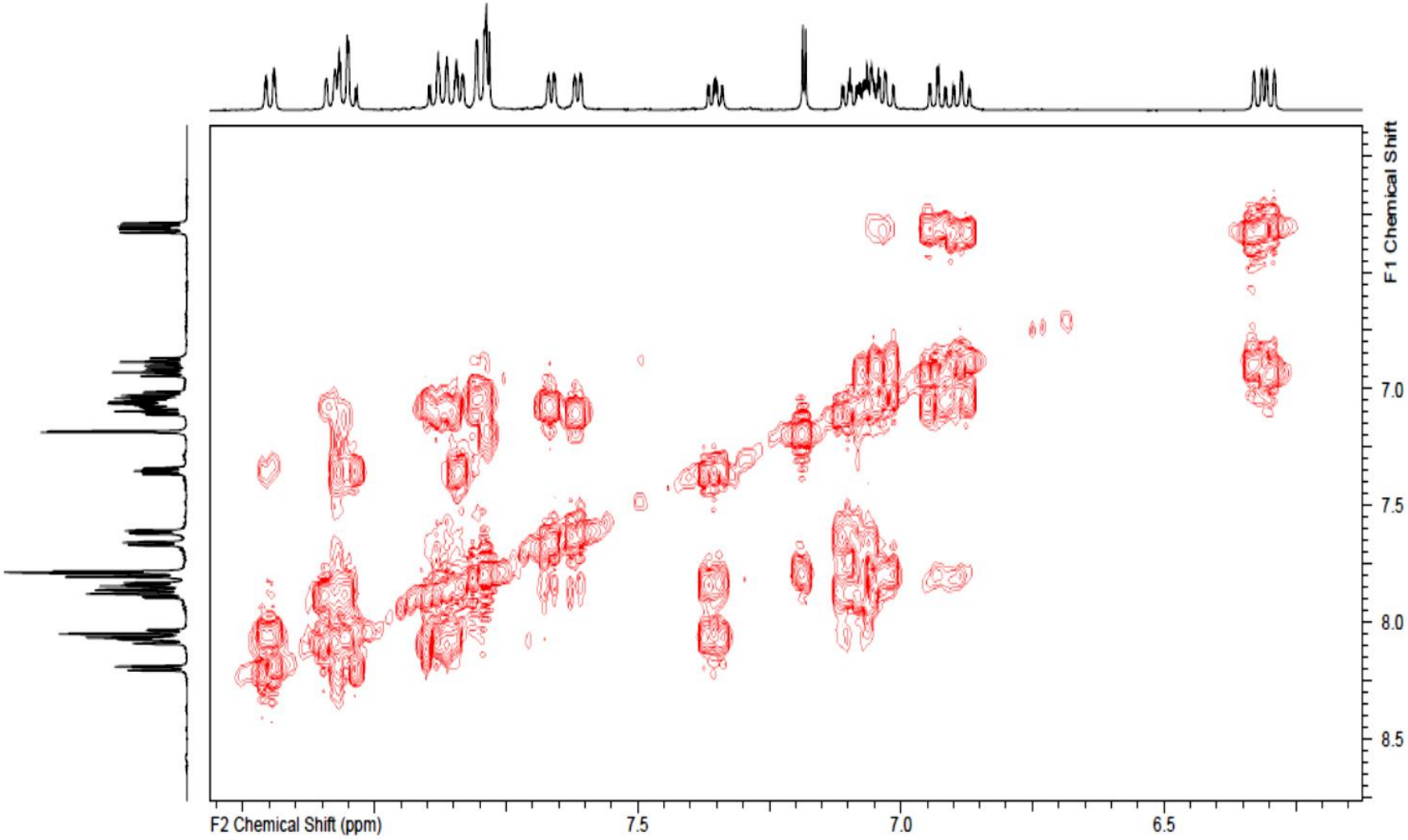
Section of ^{13}**C NMR APT** spectrum of **2cHL$_1$**



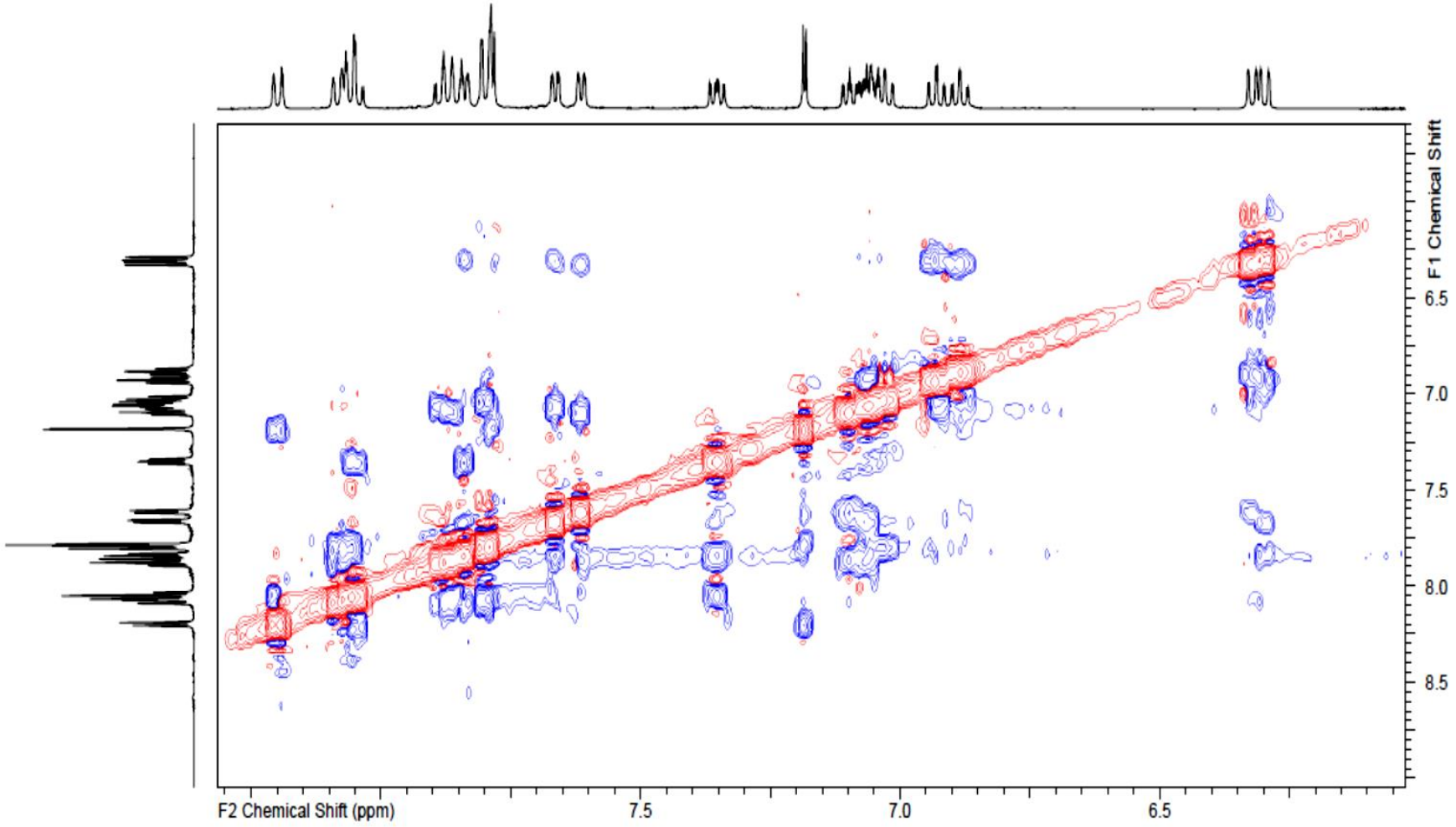
Section of the **TOCSY** spectrum of **2cHL$_1$**



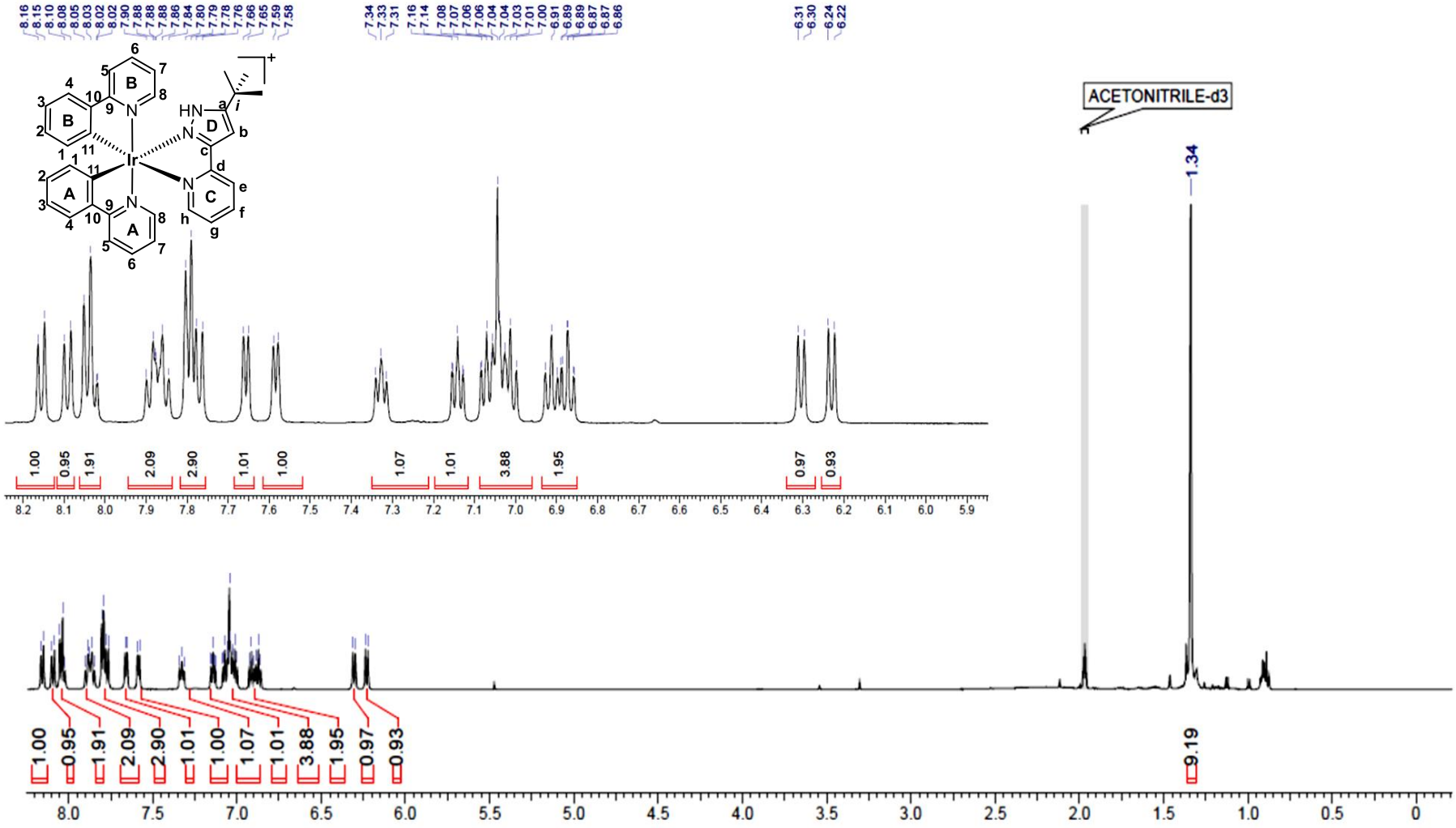
Section of the **COSY** spectrum of **2cHL$_1$**



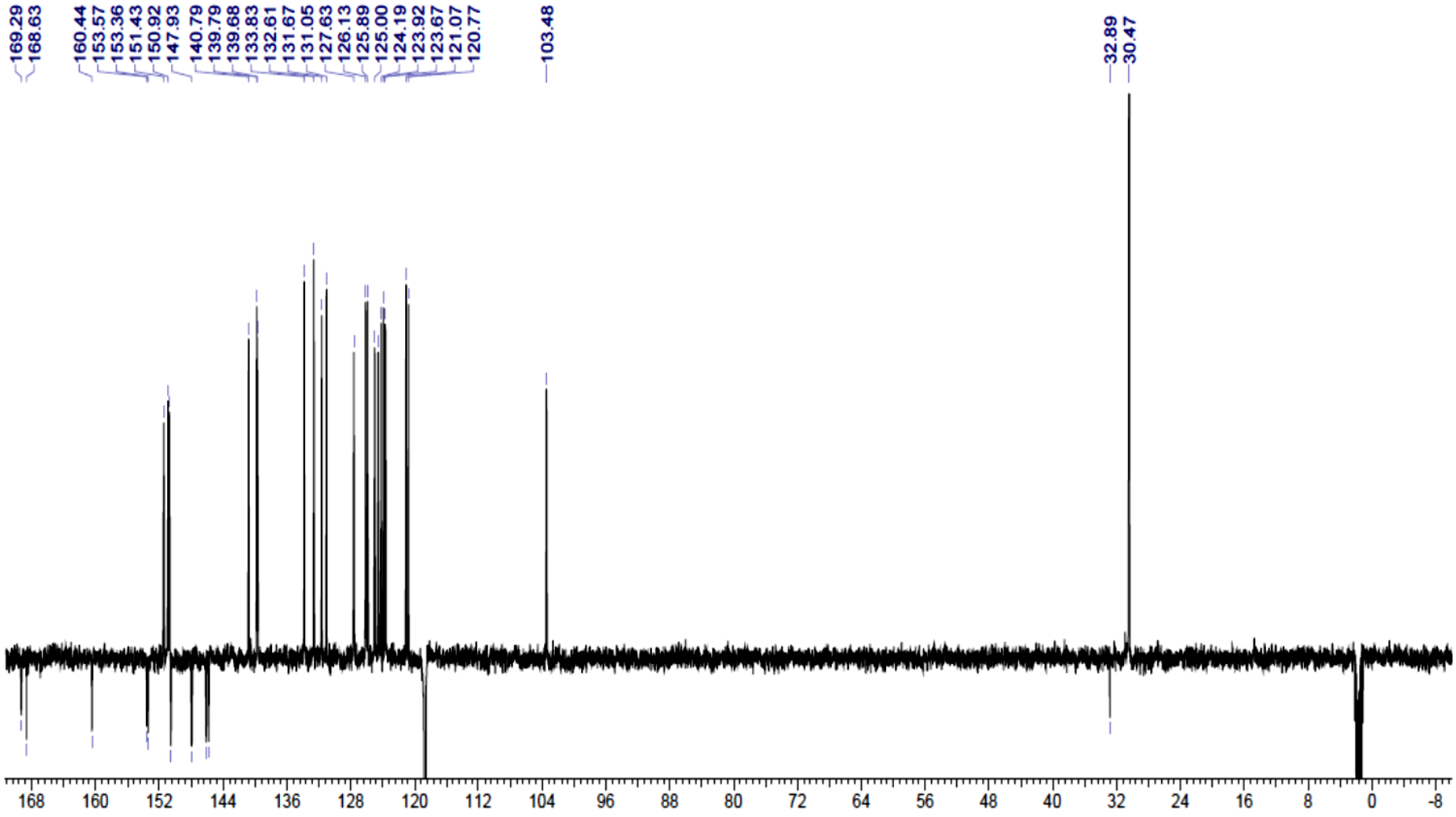
Section of the **NOESY** spectrum of **2cHL$_1$**



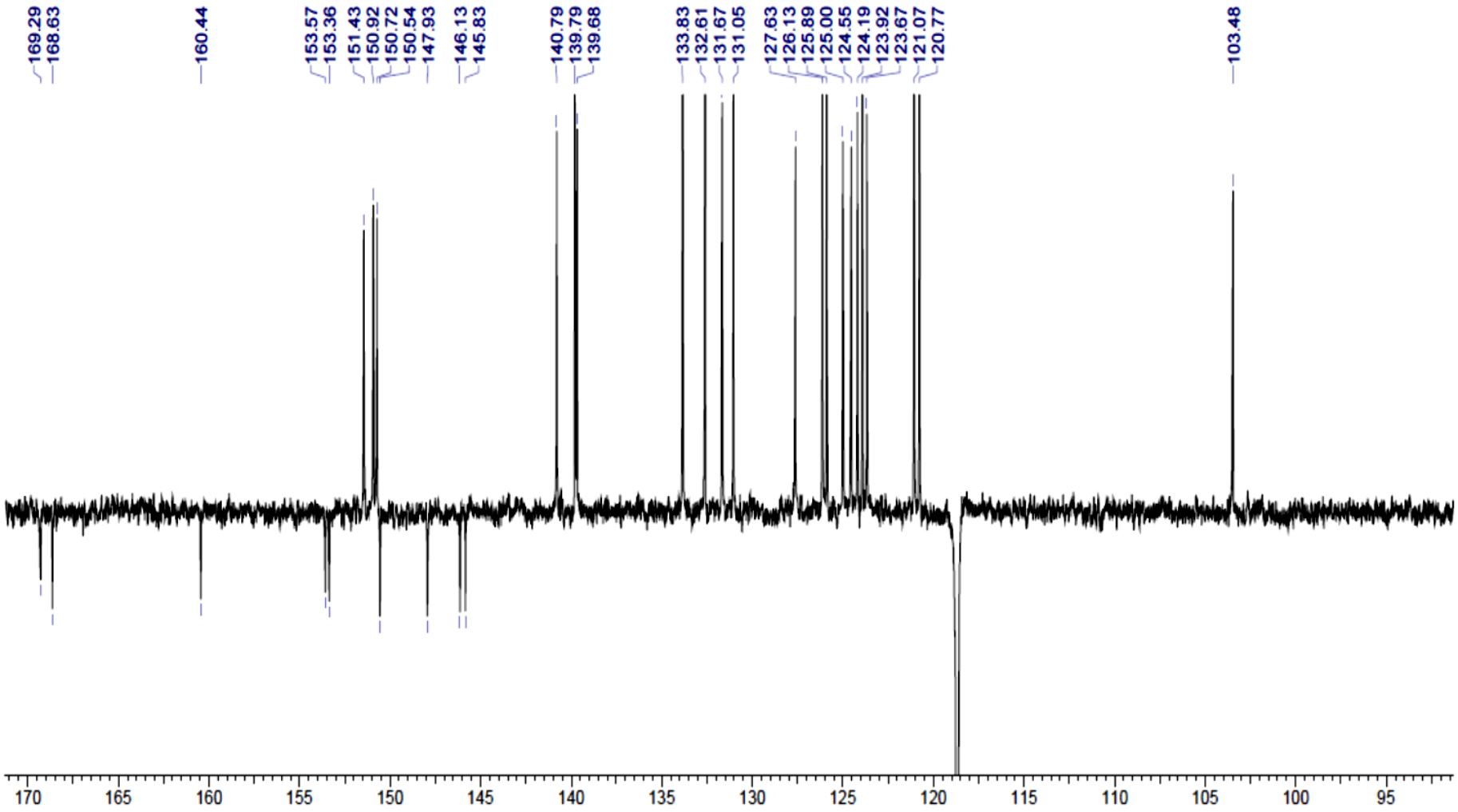
^{1}H NMR spectrun of **2cHL$_2$**



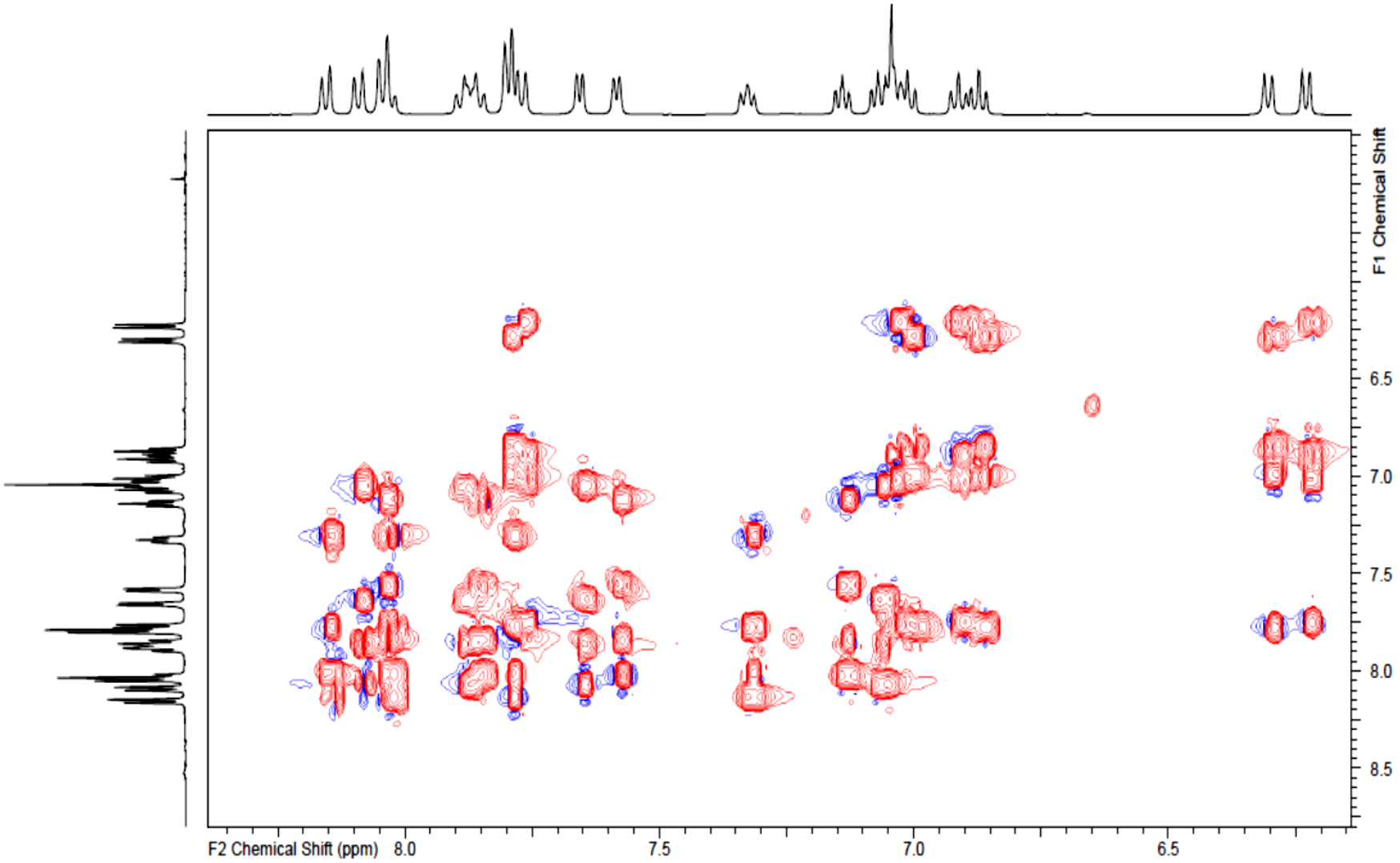
^{13}C NMR APT spectrum of **2cHL$_2$**



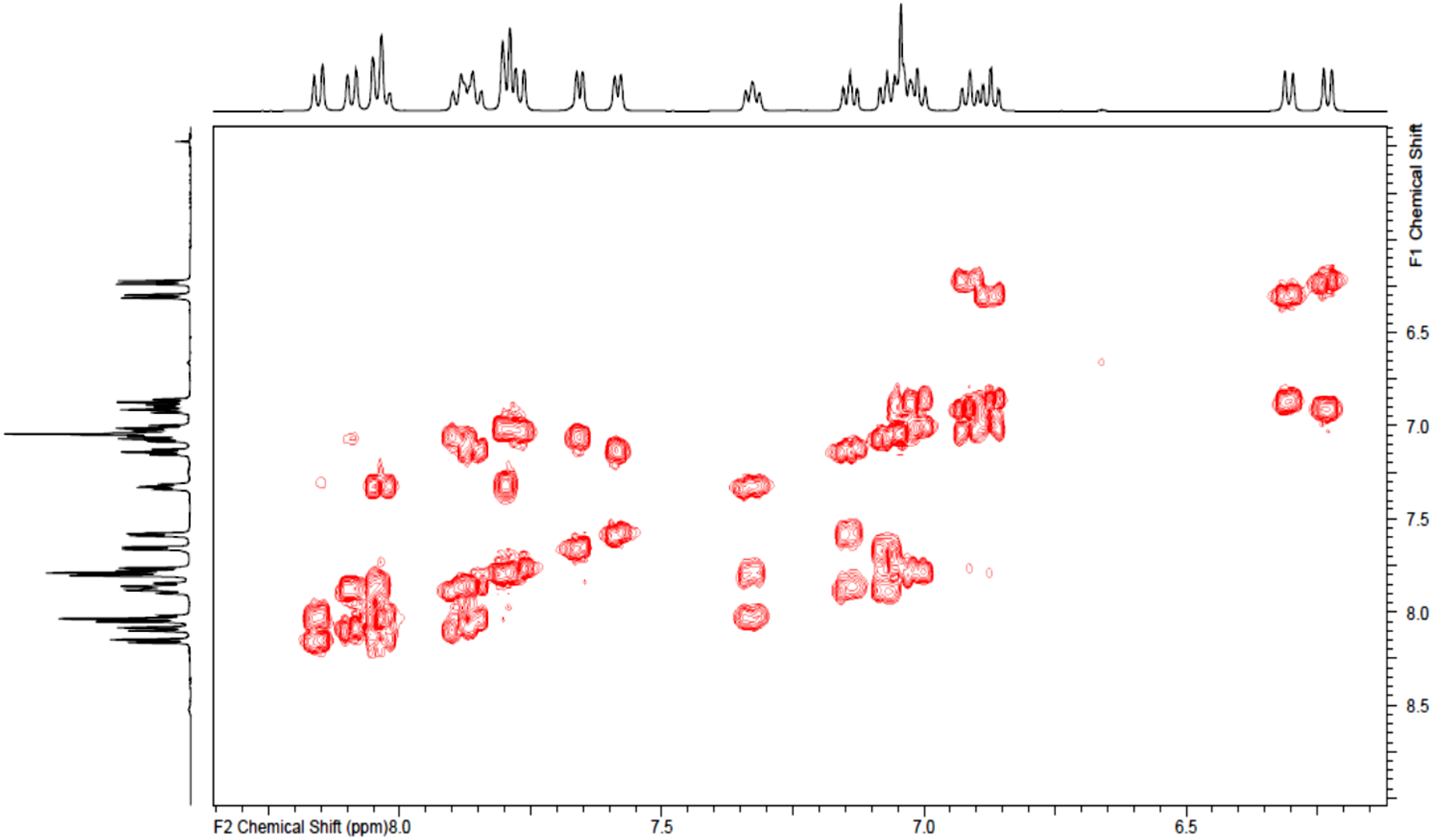
Section of ^{13}**C NMR APT spectrum** of **2cHL$_2$**



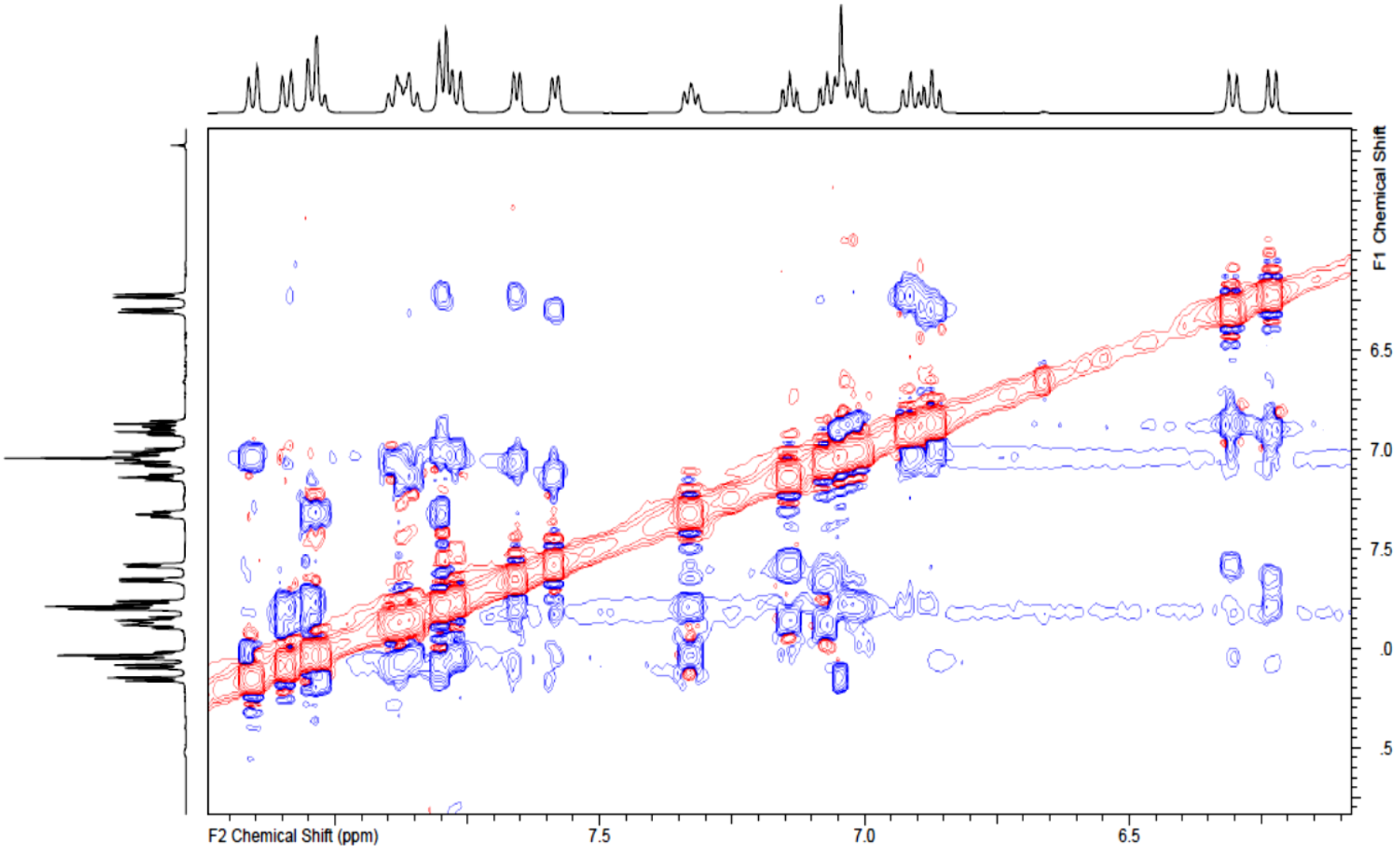
Section of the **TOCSY** spectrum of **2cHL$_2$**



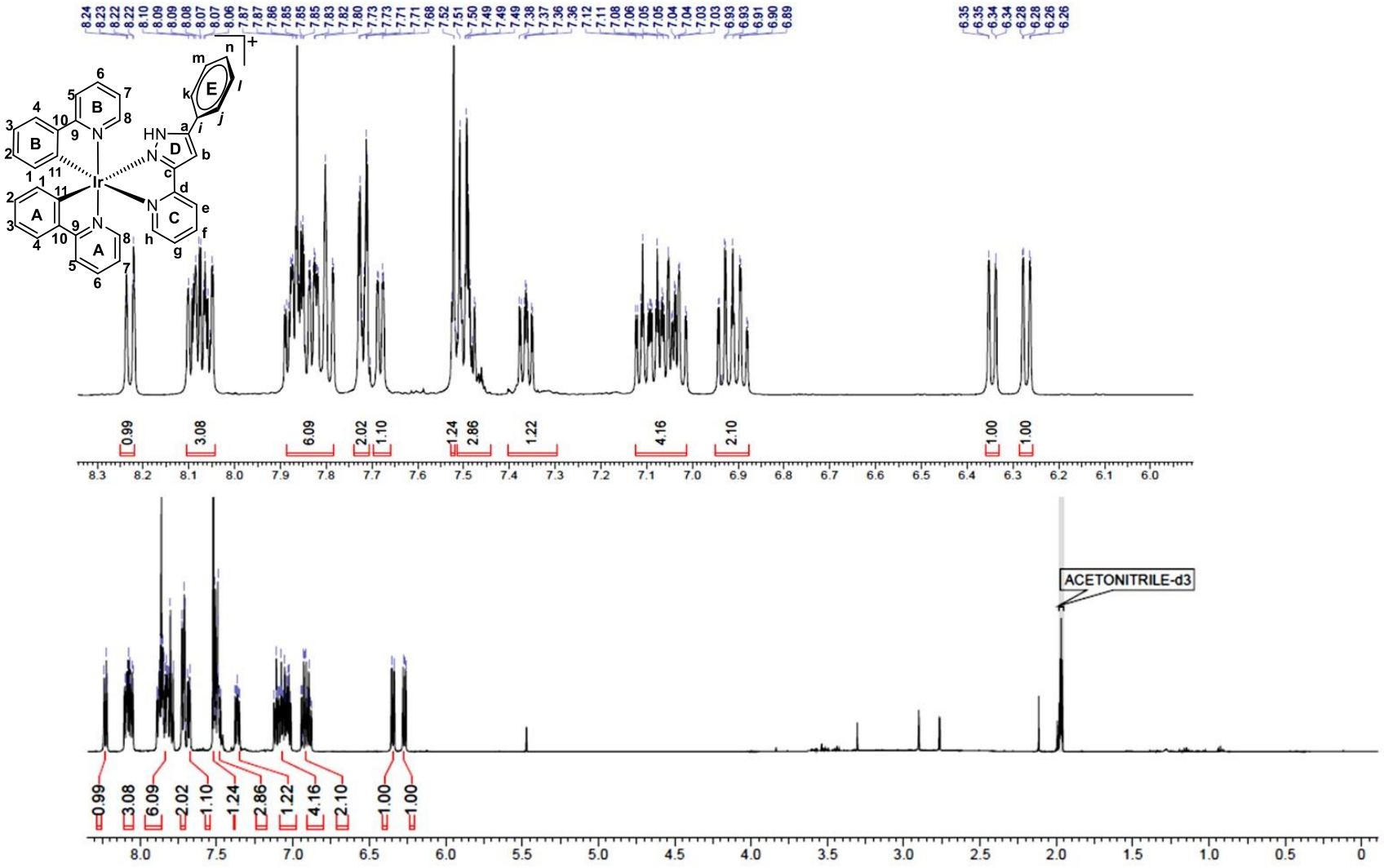
Section of the **COSY** spectrum of **2cHL$_2$**



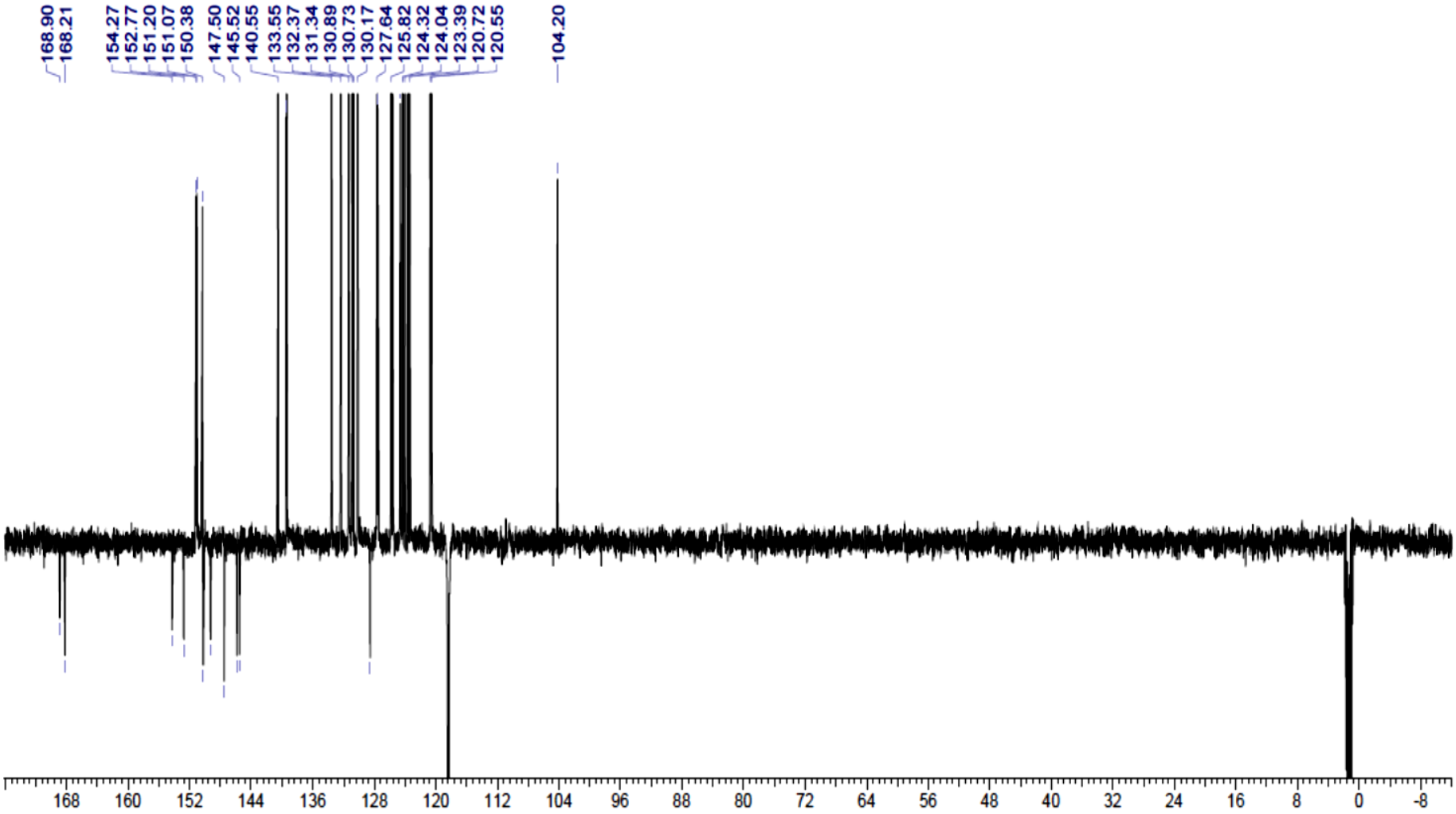
Section of the **NOESY** spectrum of **2cHL$_2$**



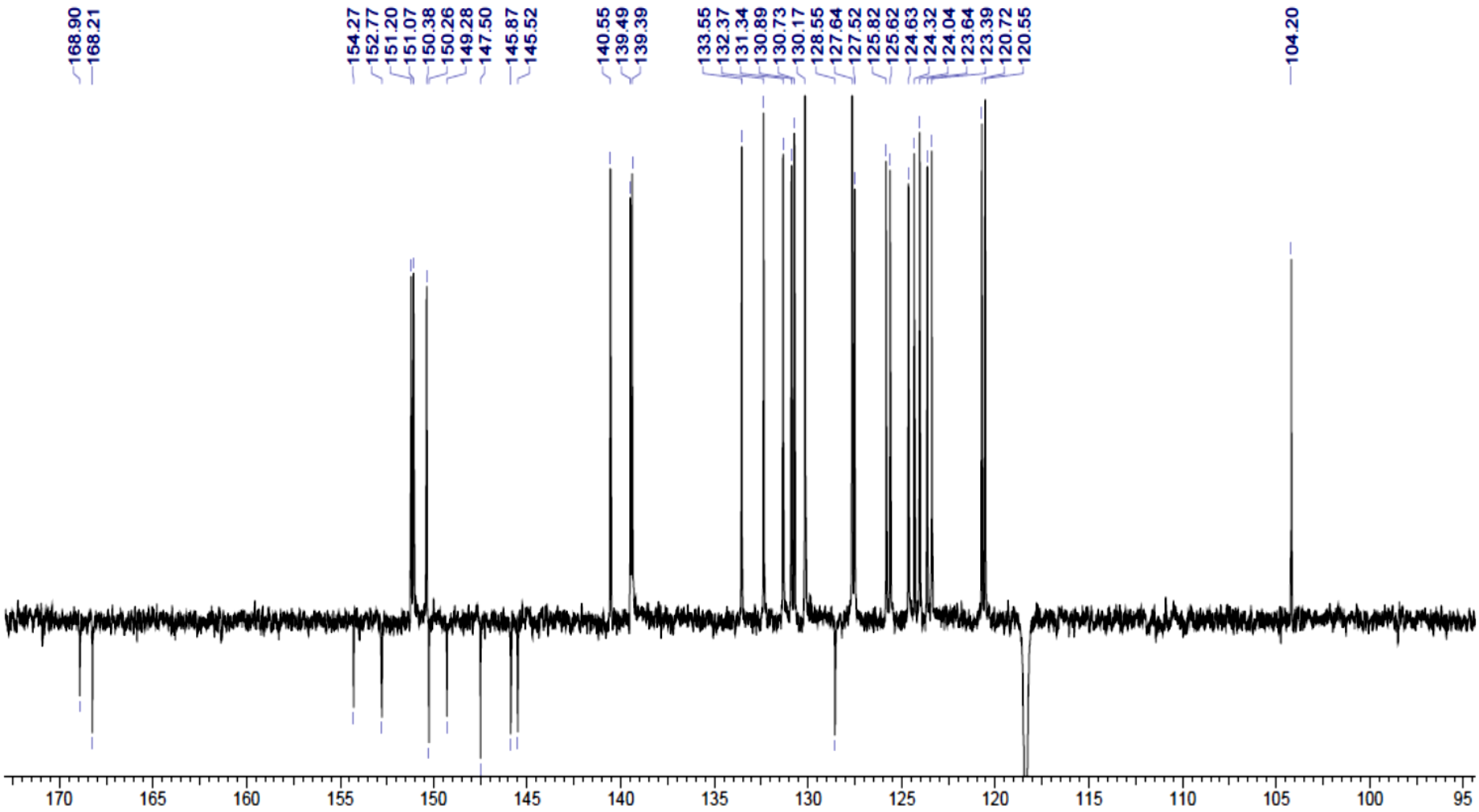
^{1}H NMR spectrum of **2cHL$_3$**



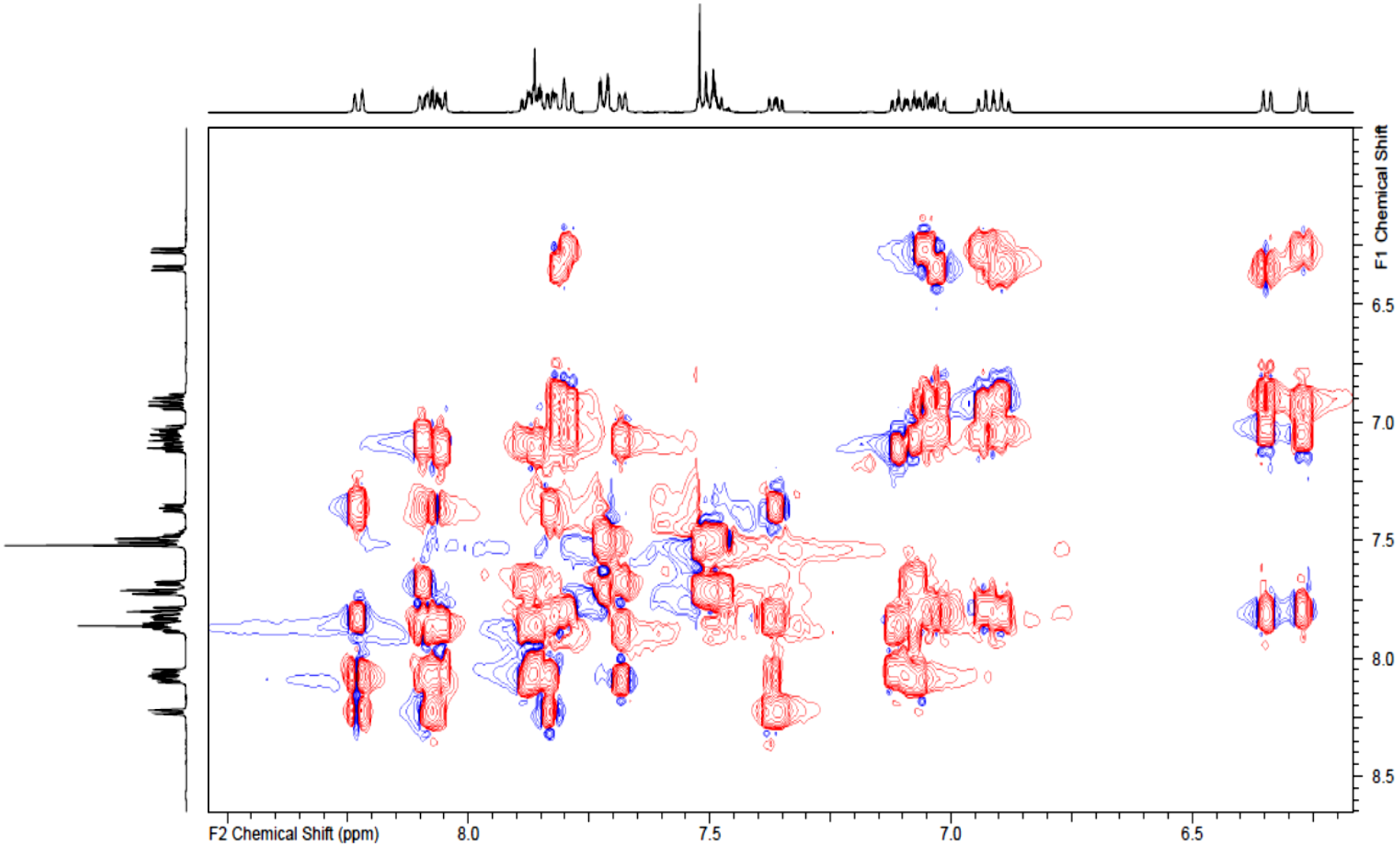
^{13}C NMR APT spectrum of **2cHL$_3$**



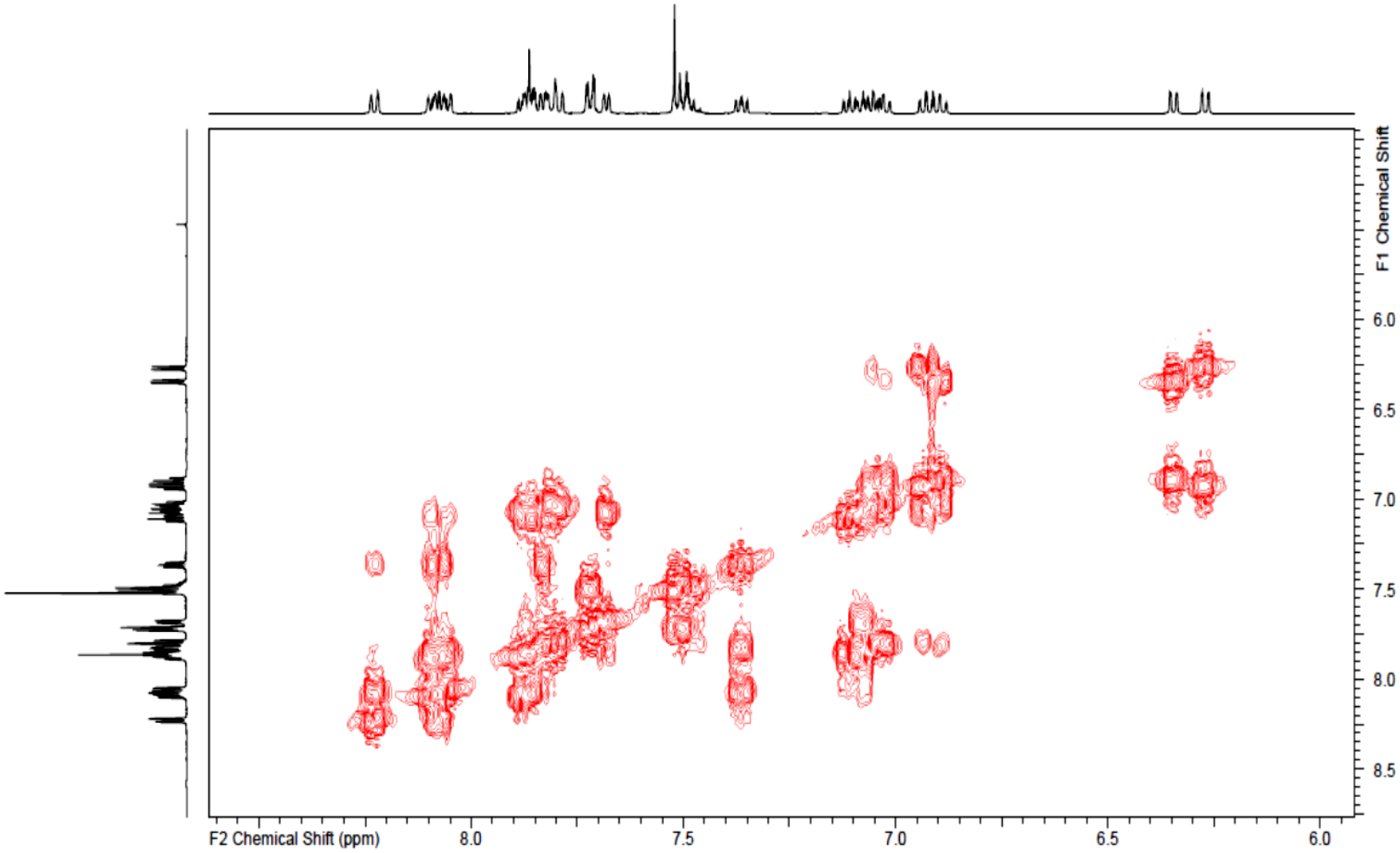
Section of ^{13}**C NMR APT** spectrum of **2cHL$_3$**



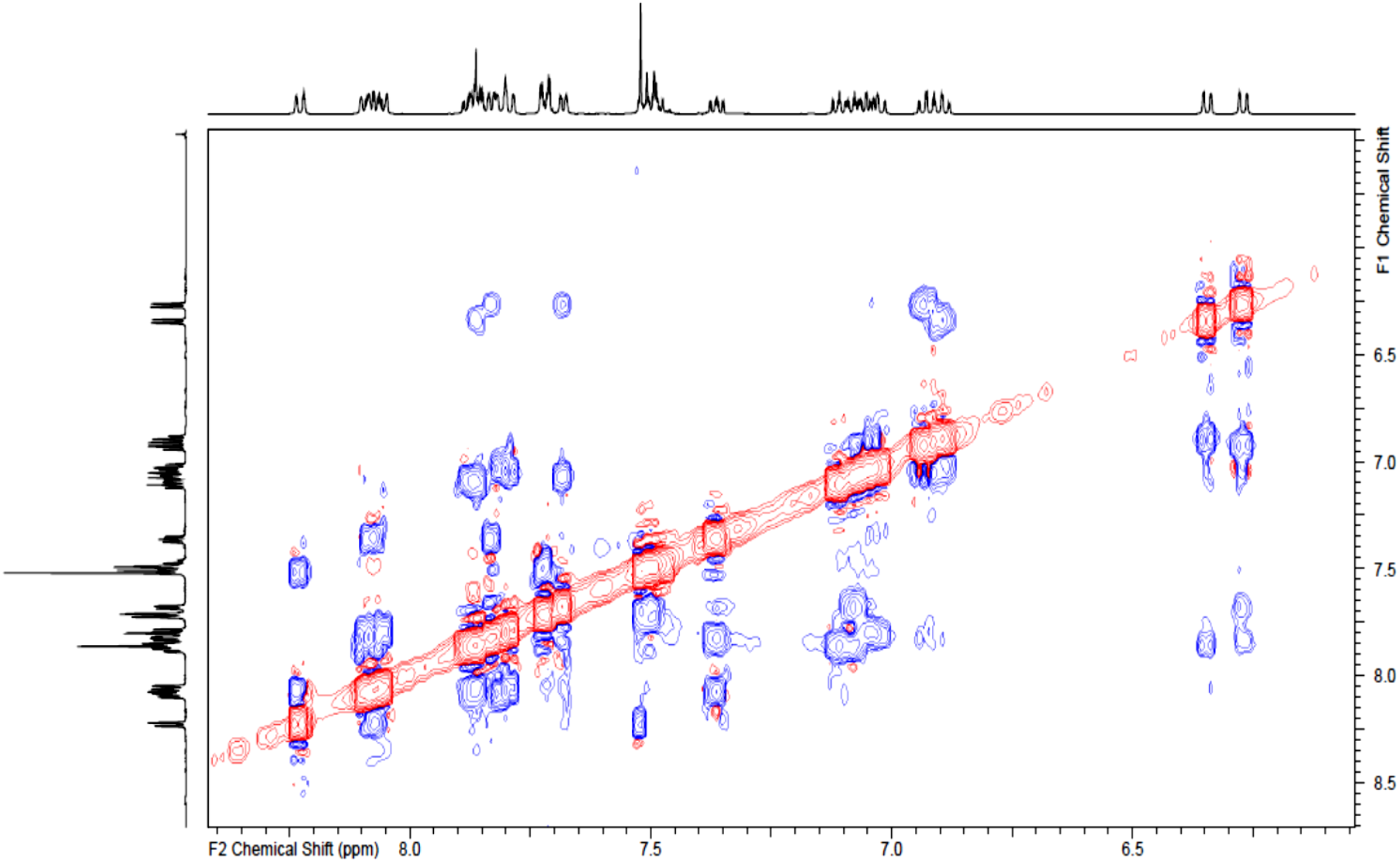
Section of the **TOCSY** spectrum of **2cHL$_3$**



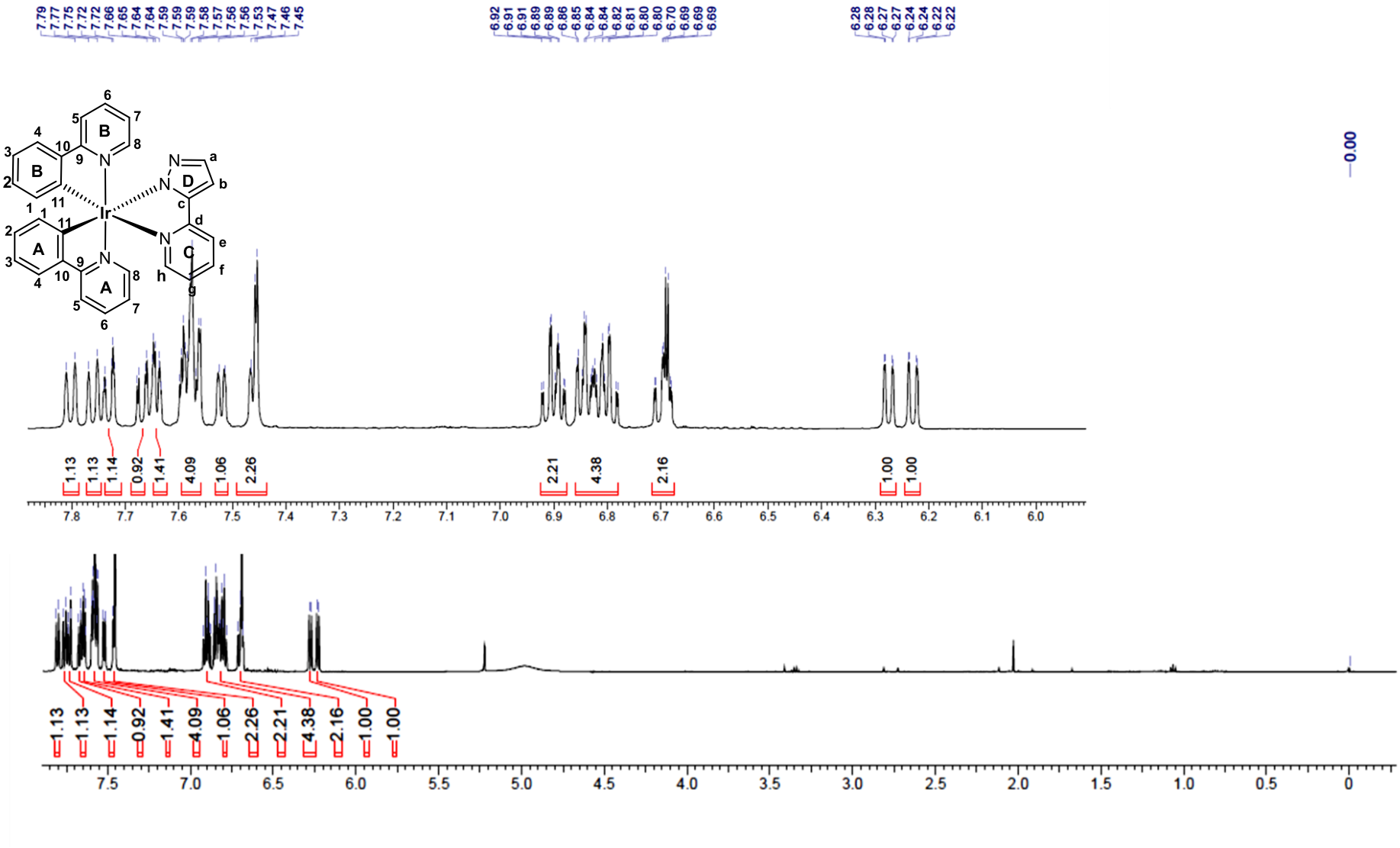
Section of the **COSY** spectrum of **2cHL$_3$**



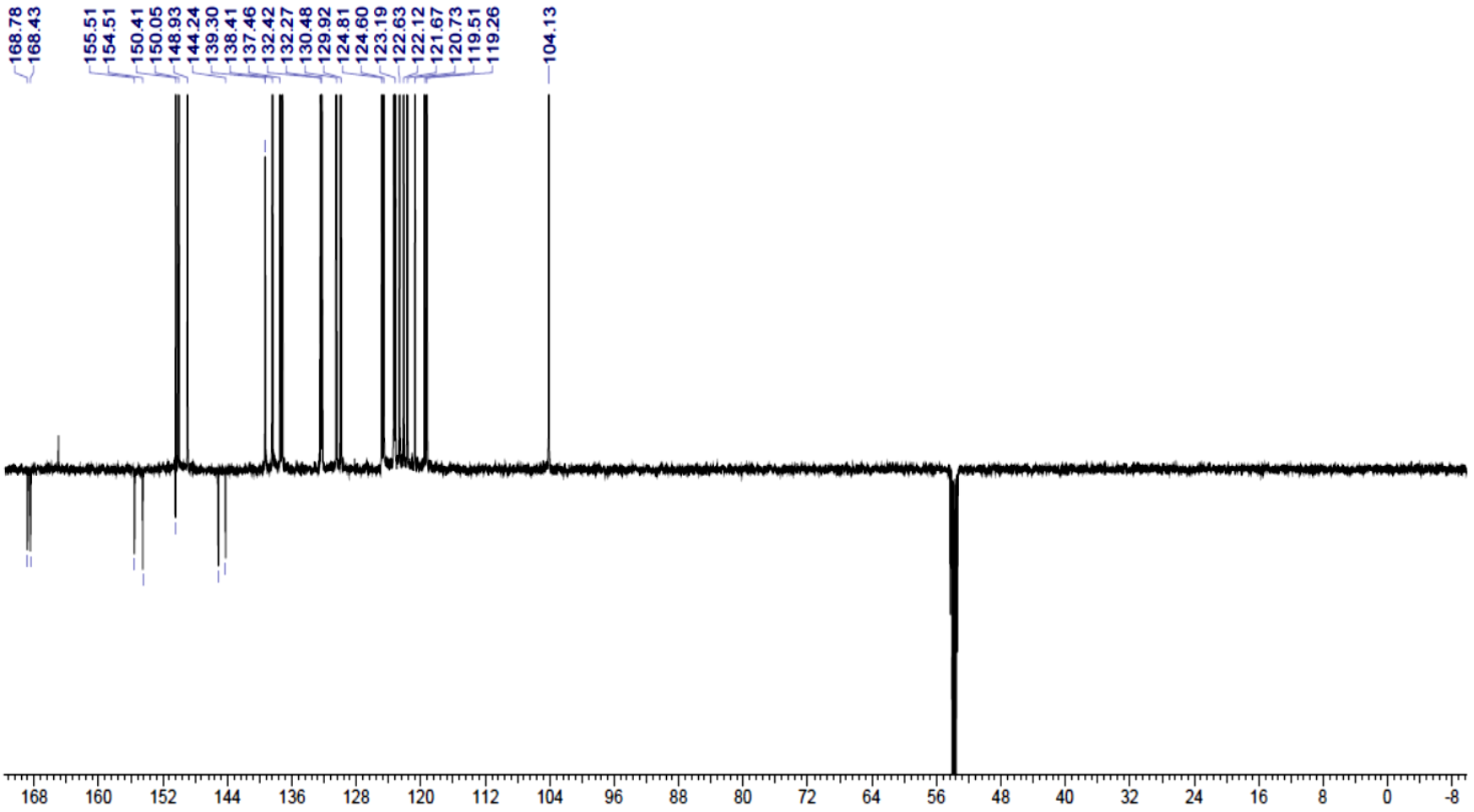
Section of the **NOESY** spectrum of **2cHL$_3$**



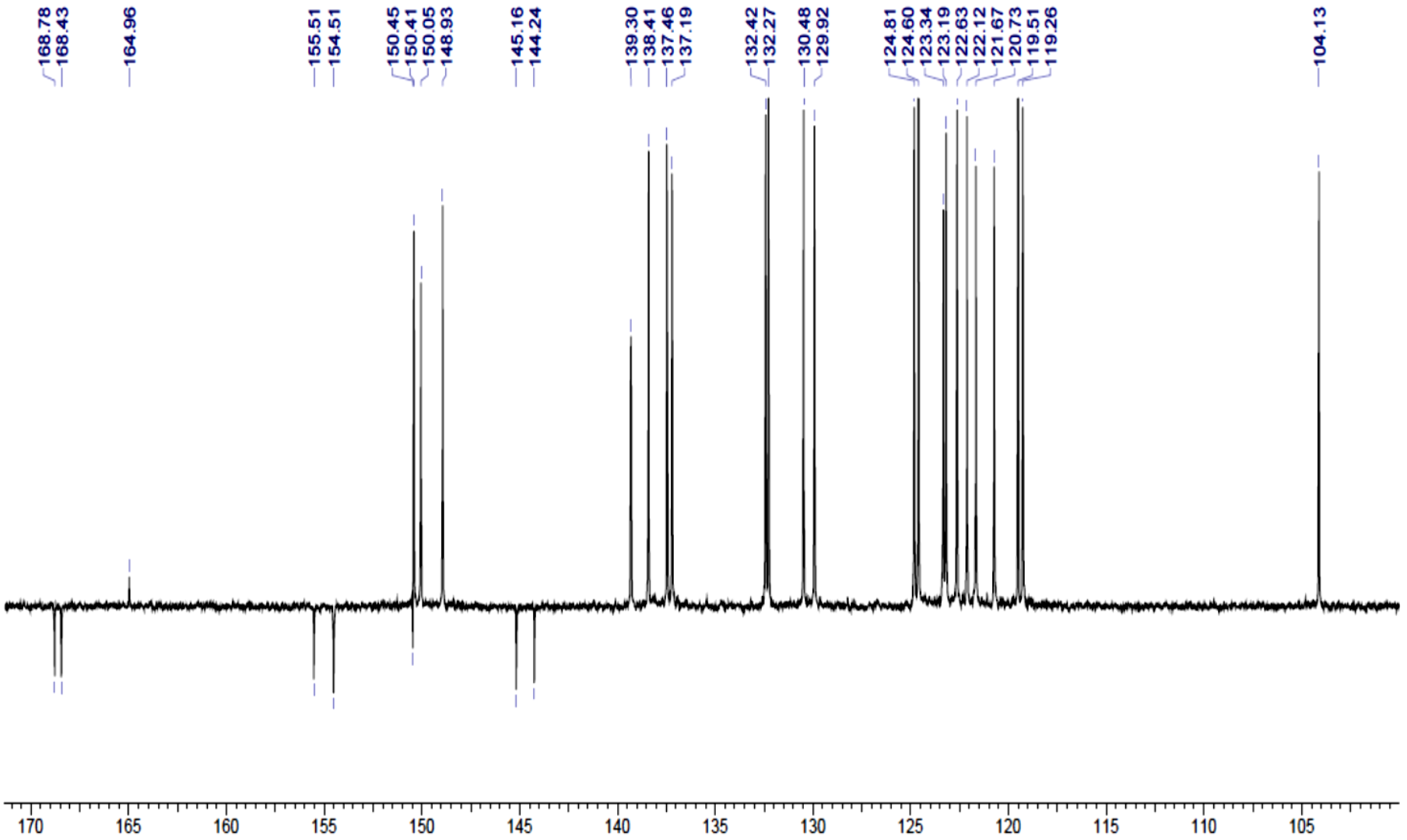
^{1}H NMR spectrum of **3cL$_1$**



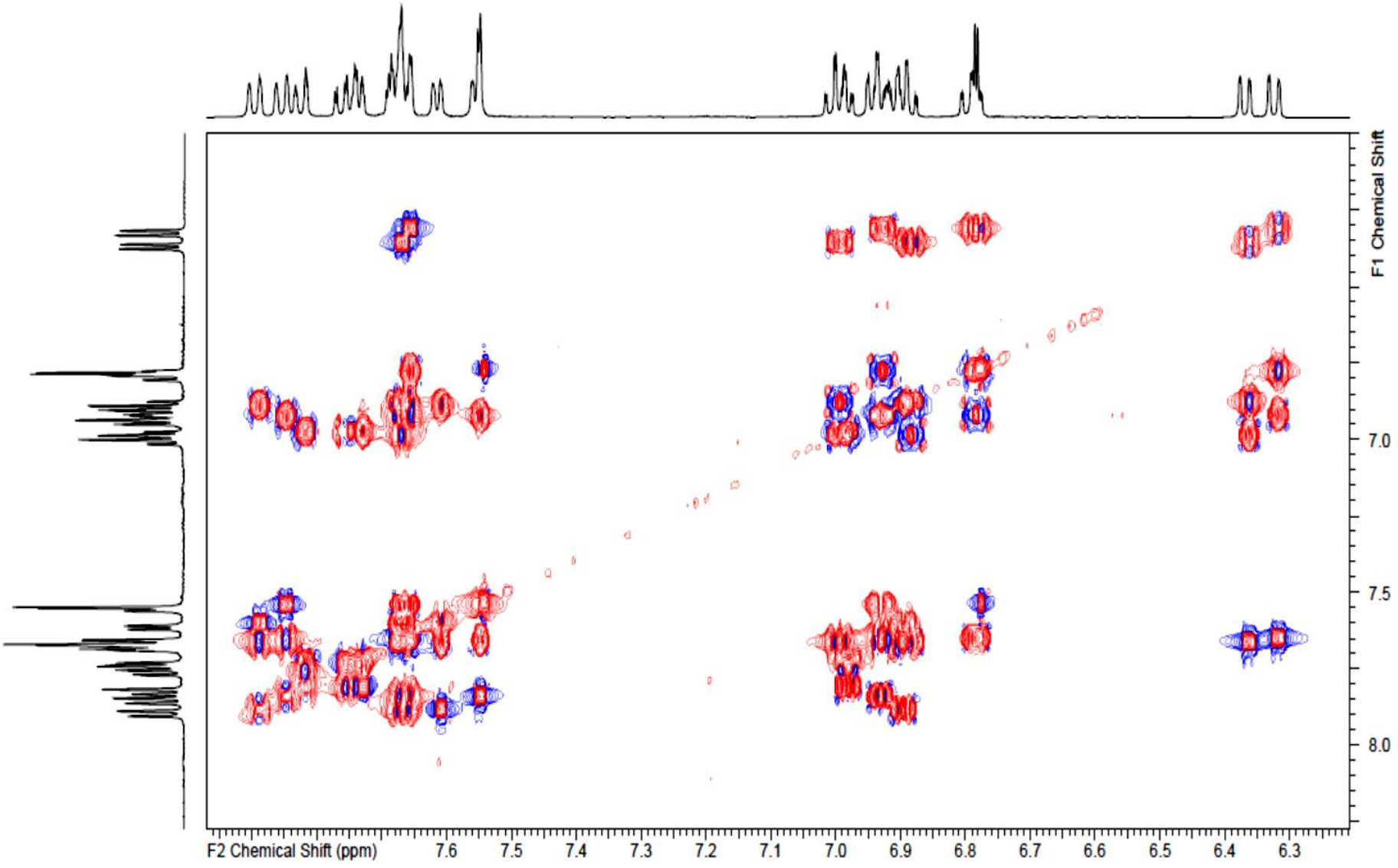
^{13}C NMR DEPT spectrum of **3cL$_1$**



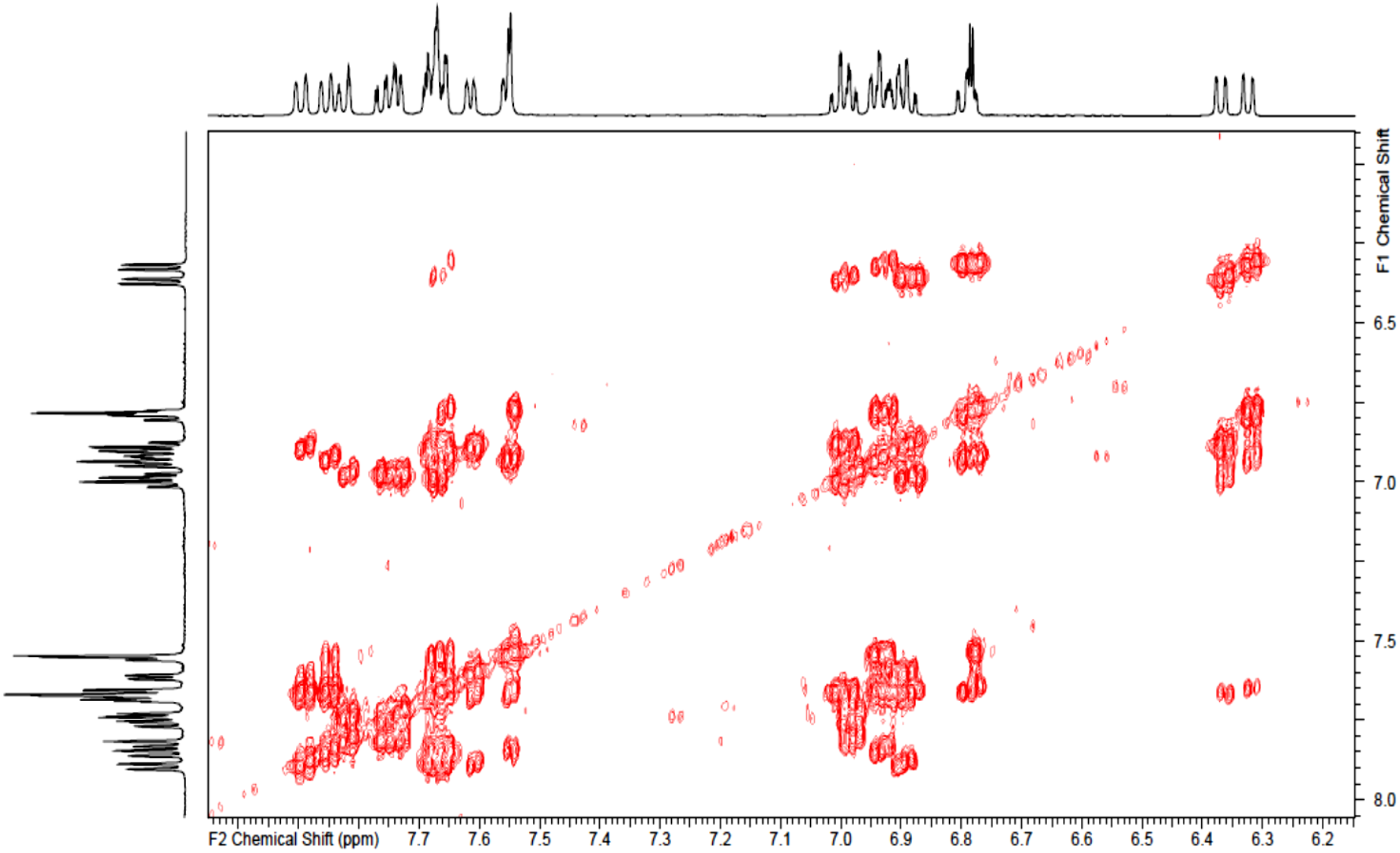
Section of ^{13}**C NMR DEPT** spectrum of **3cL$_1$**



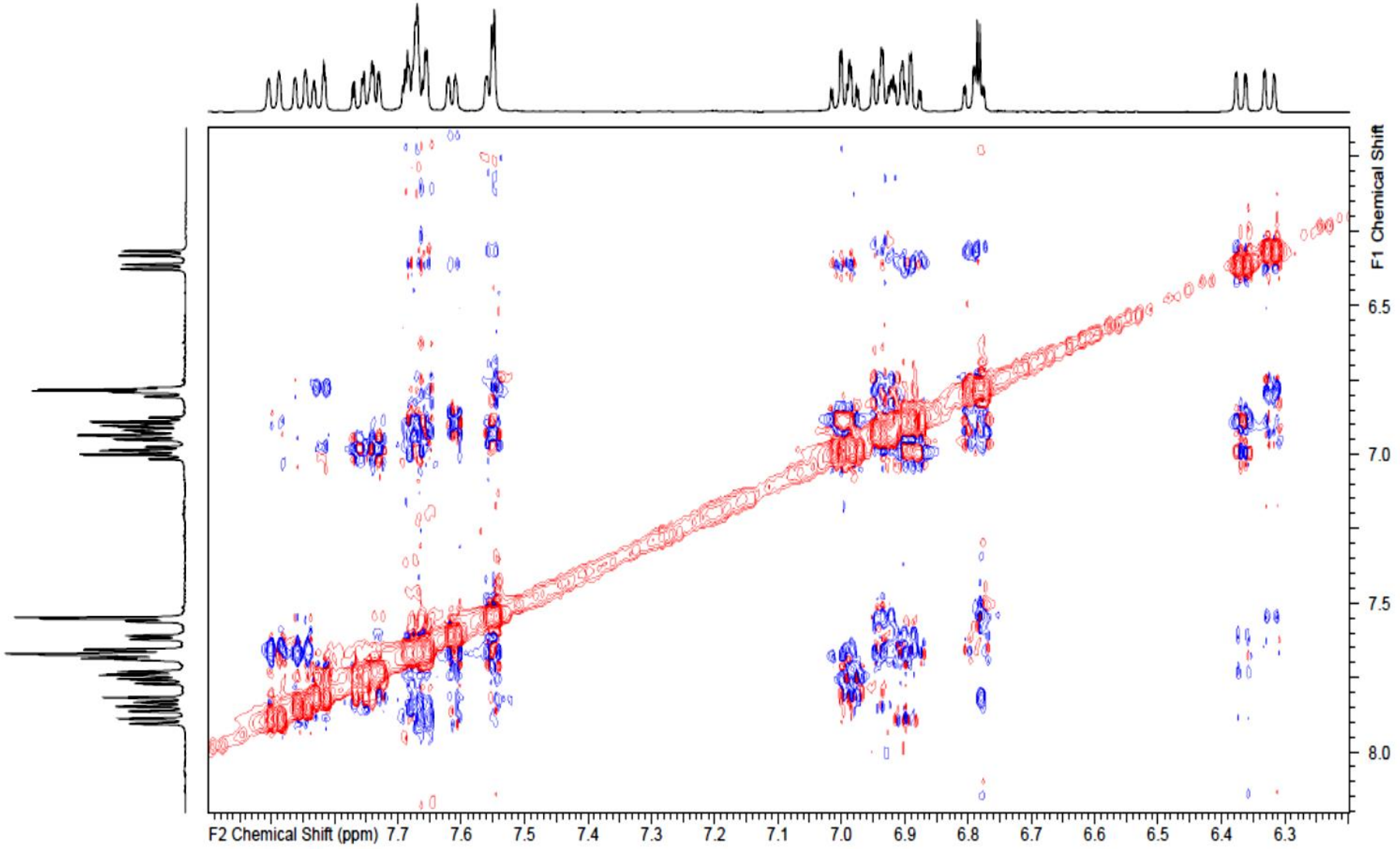
Section of the **TOCSY** spectrum of **3cL$_1$**



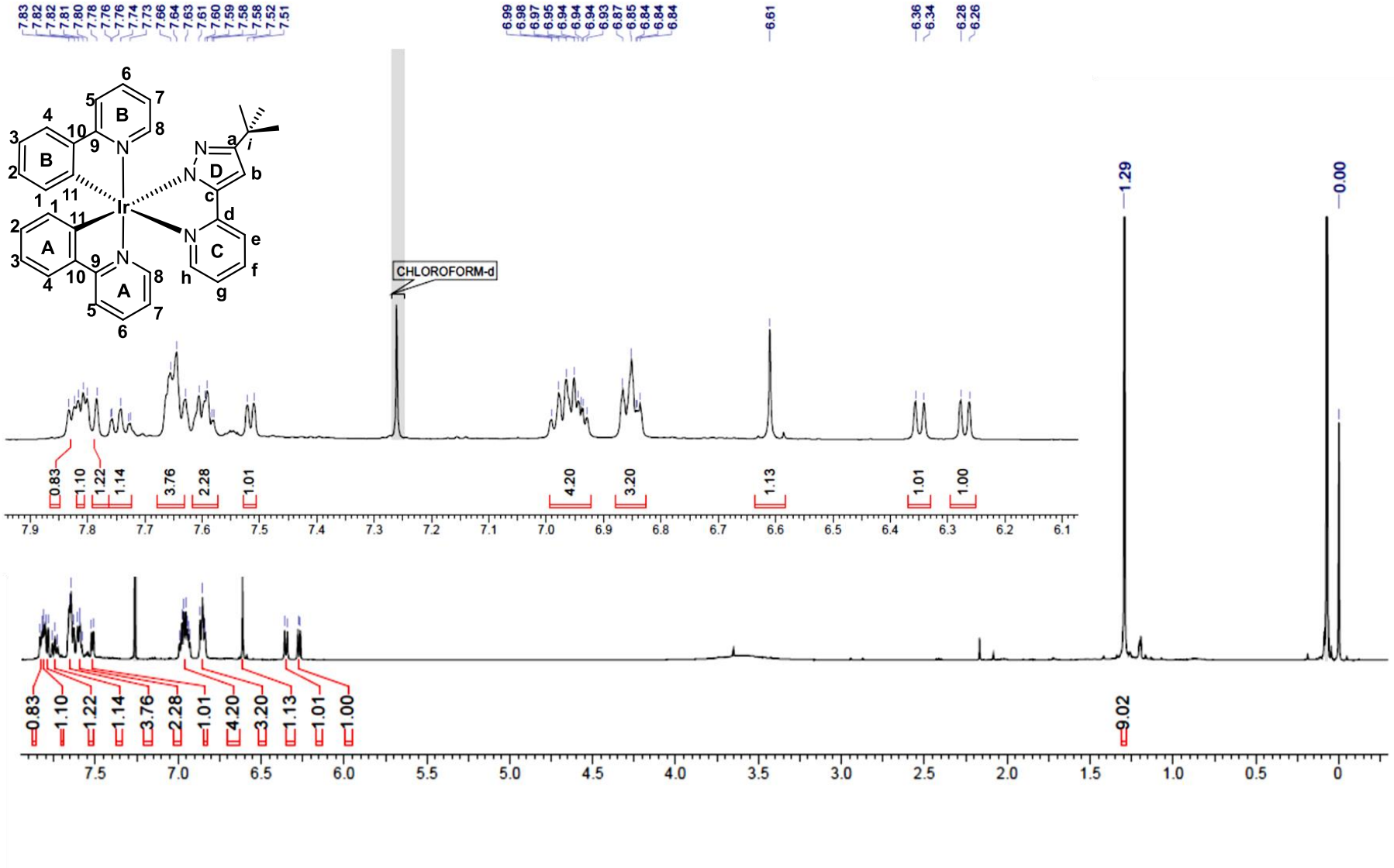
Section of the **COSY** spectrum of **3cL$_1$**



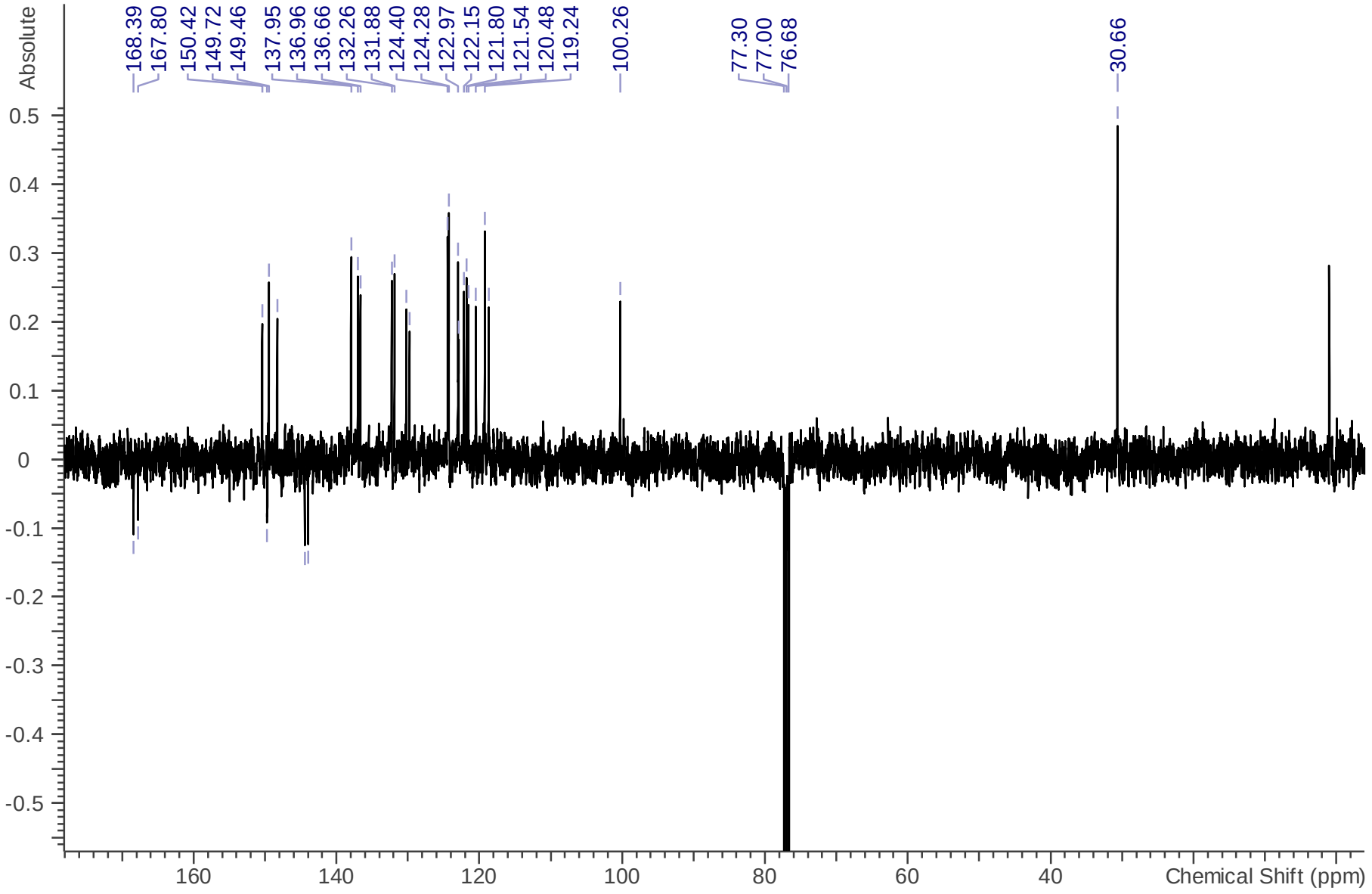
Section of the **NOESY** spectrum of **3cL$_1$**



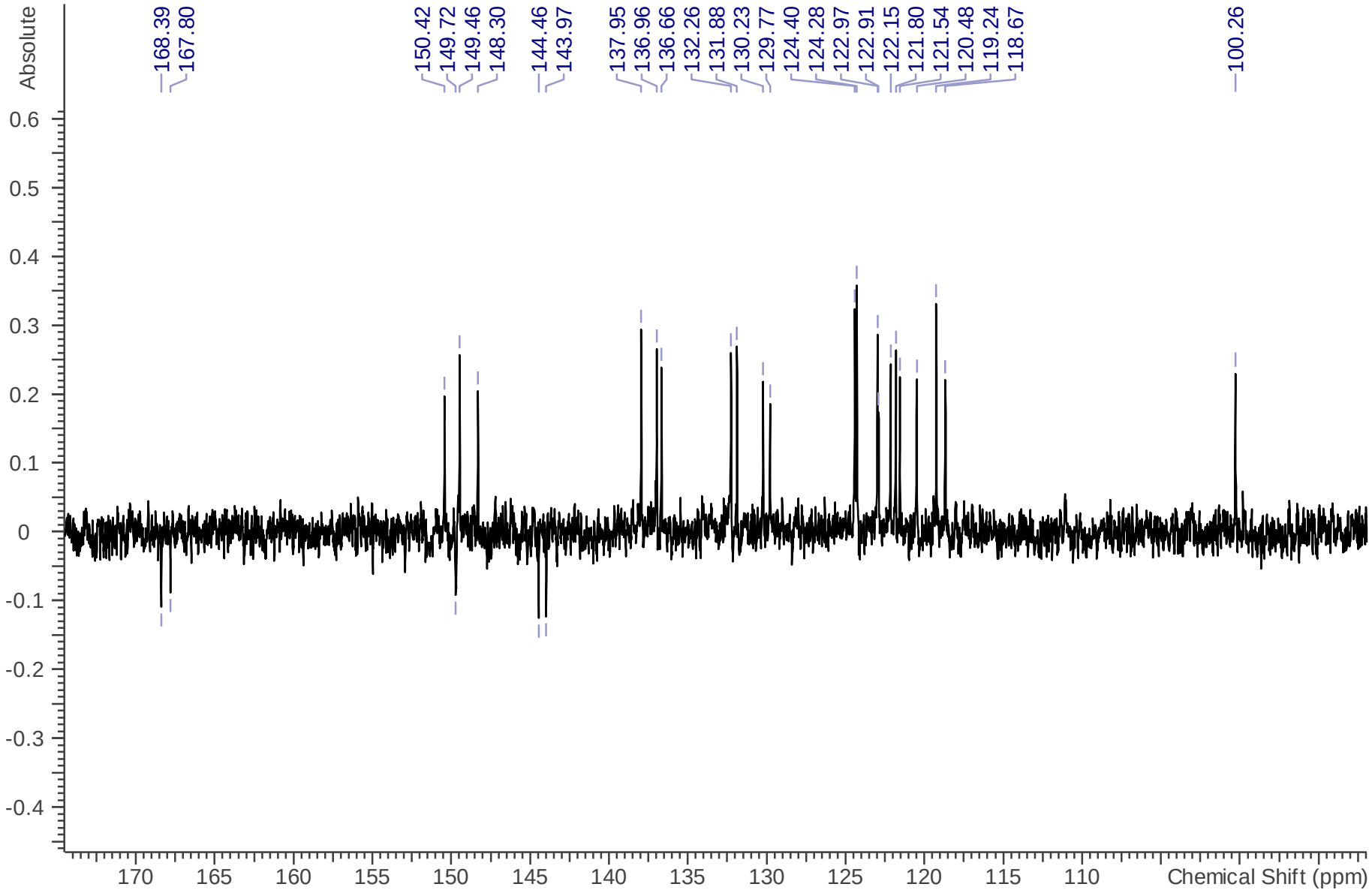
^{1}H NMR spectrum of **3cL$_2$**



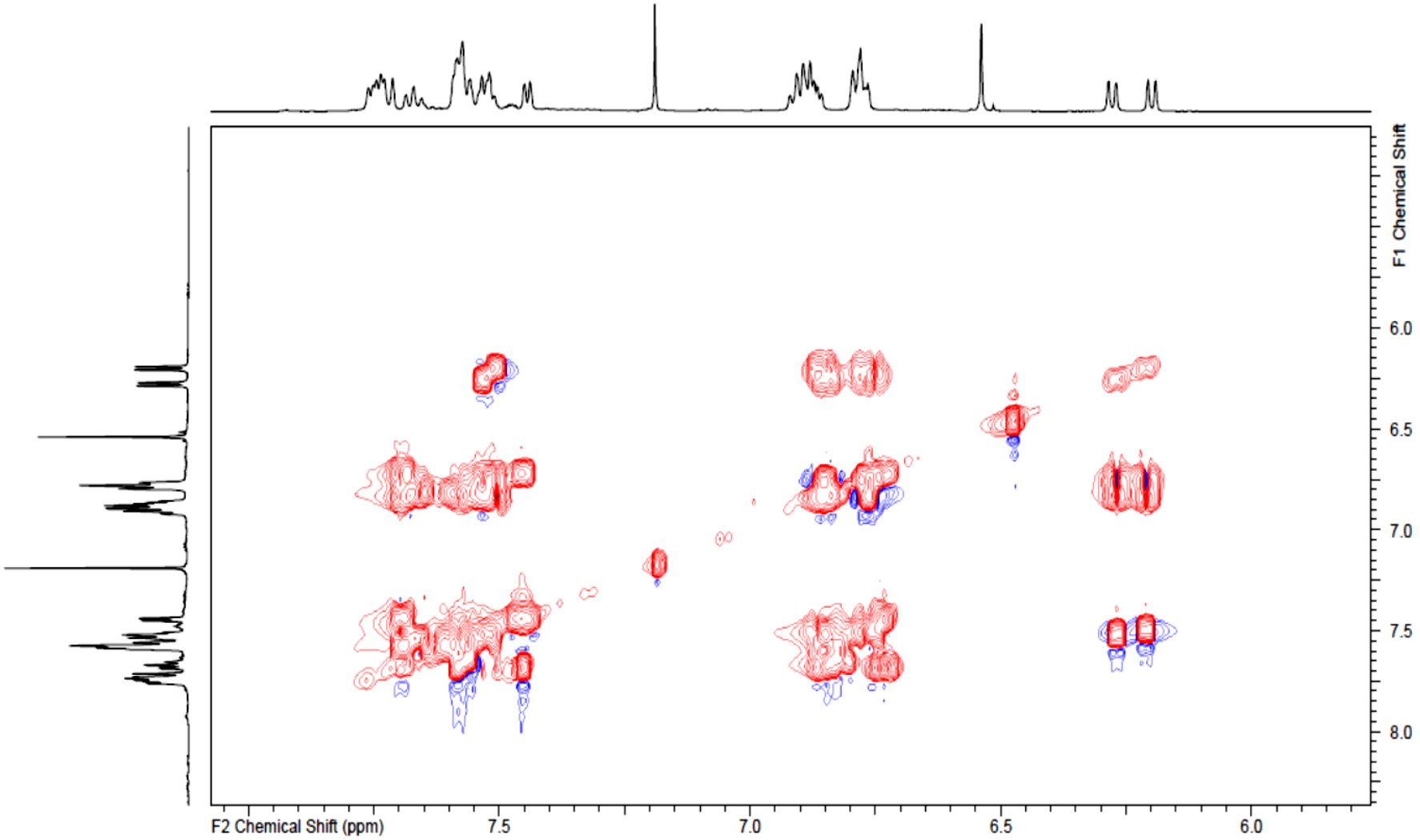
^{13}C NMR APT spectrum of **3cL$_2$**



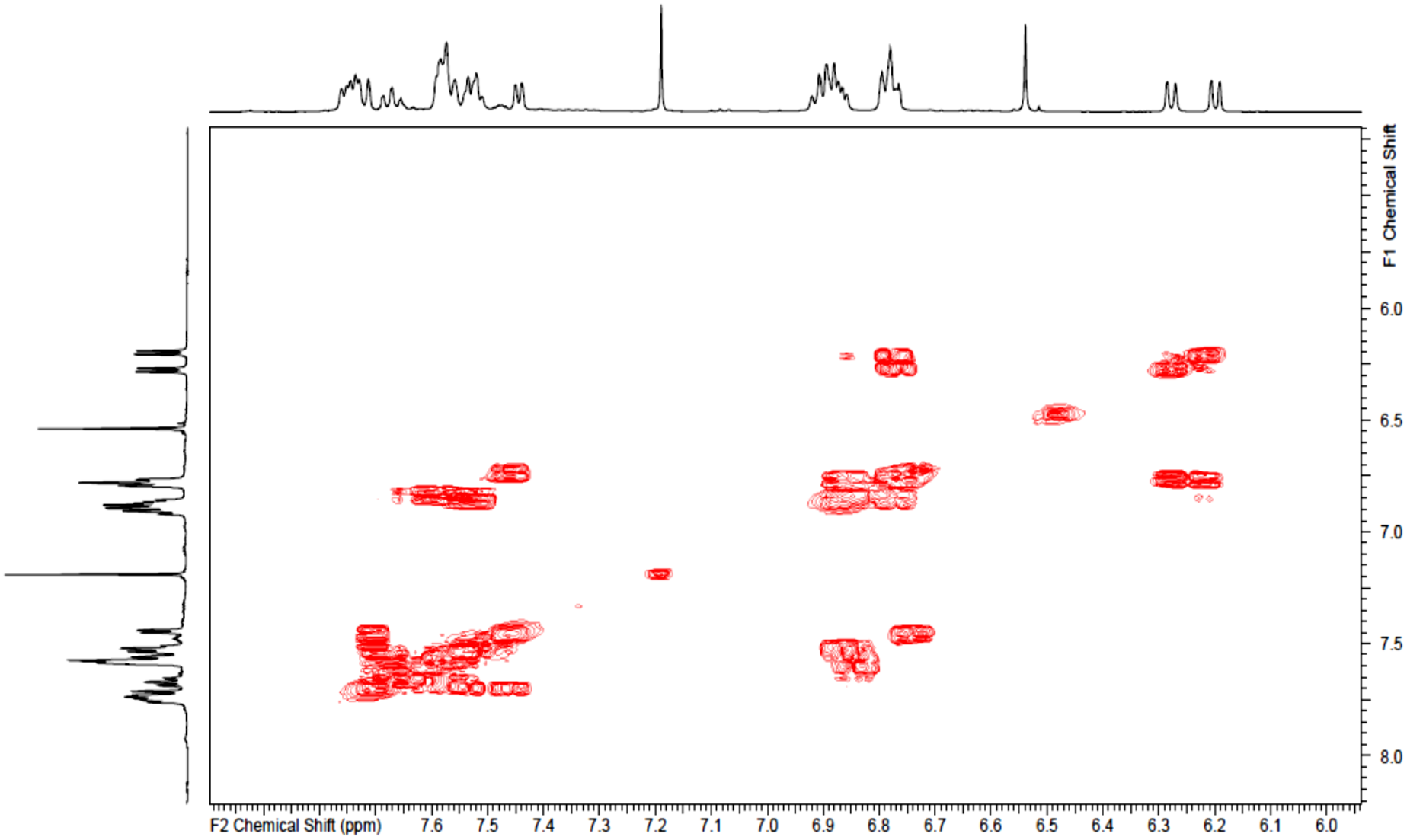
Section of ^{13}**C NMR APT** spectrum of **3cL$_2$**



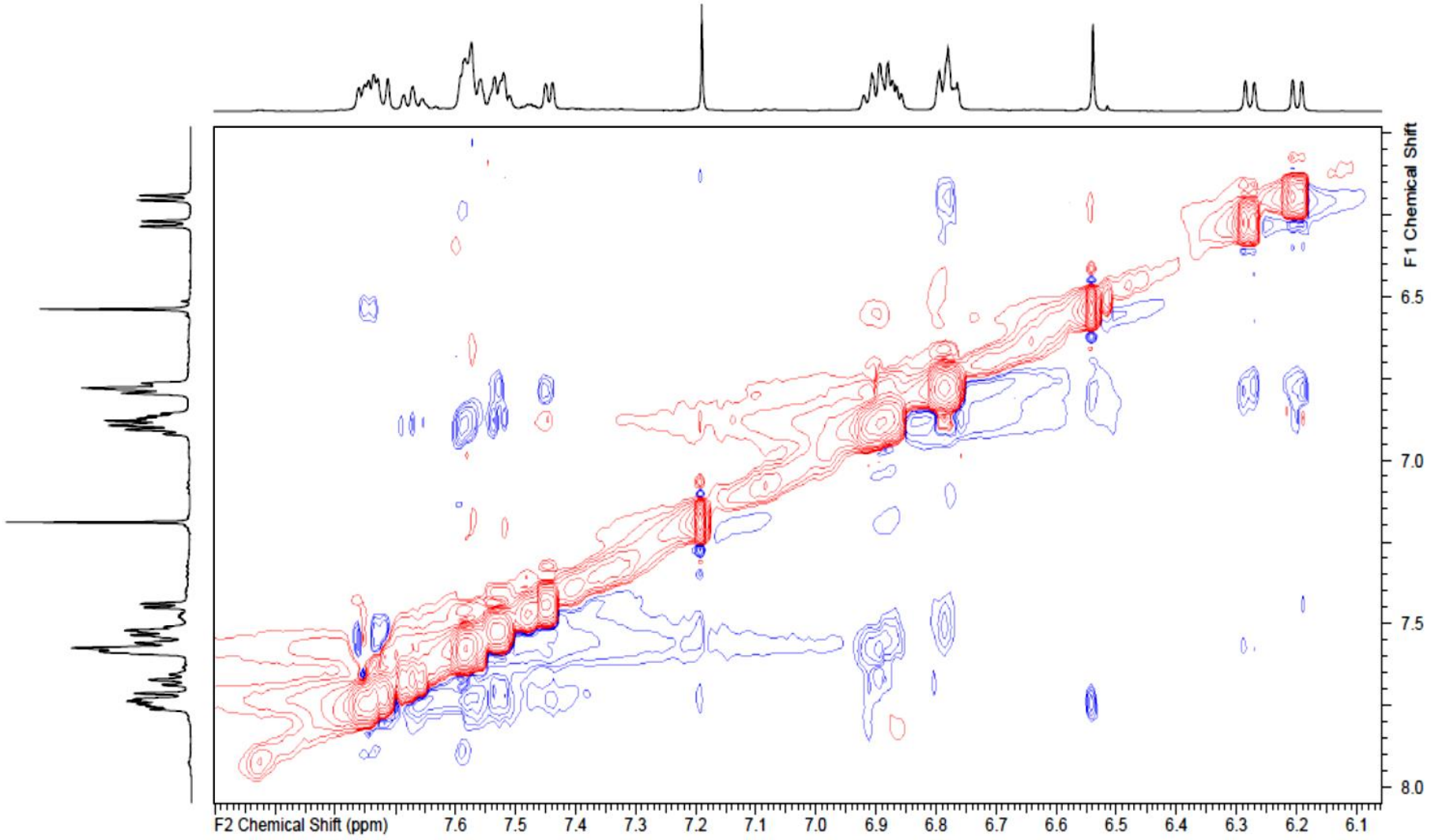
Section of the **TOCSY** spectrum of **3cL$_2$**



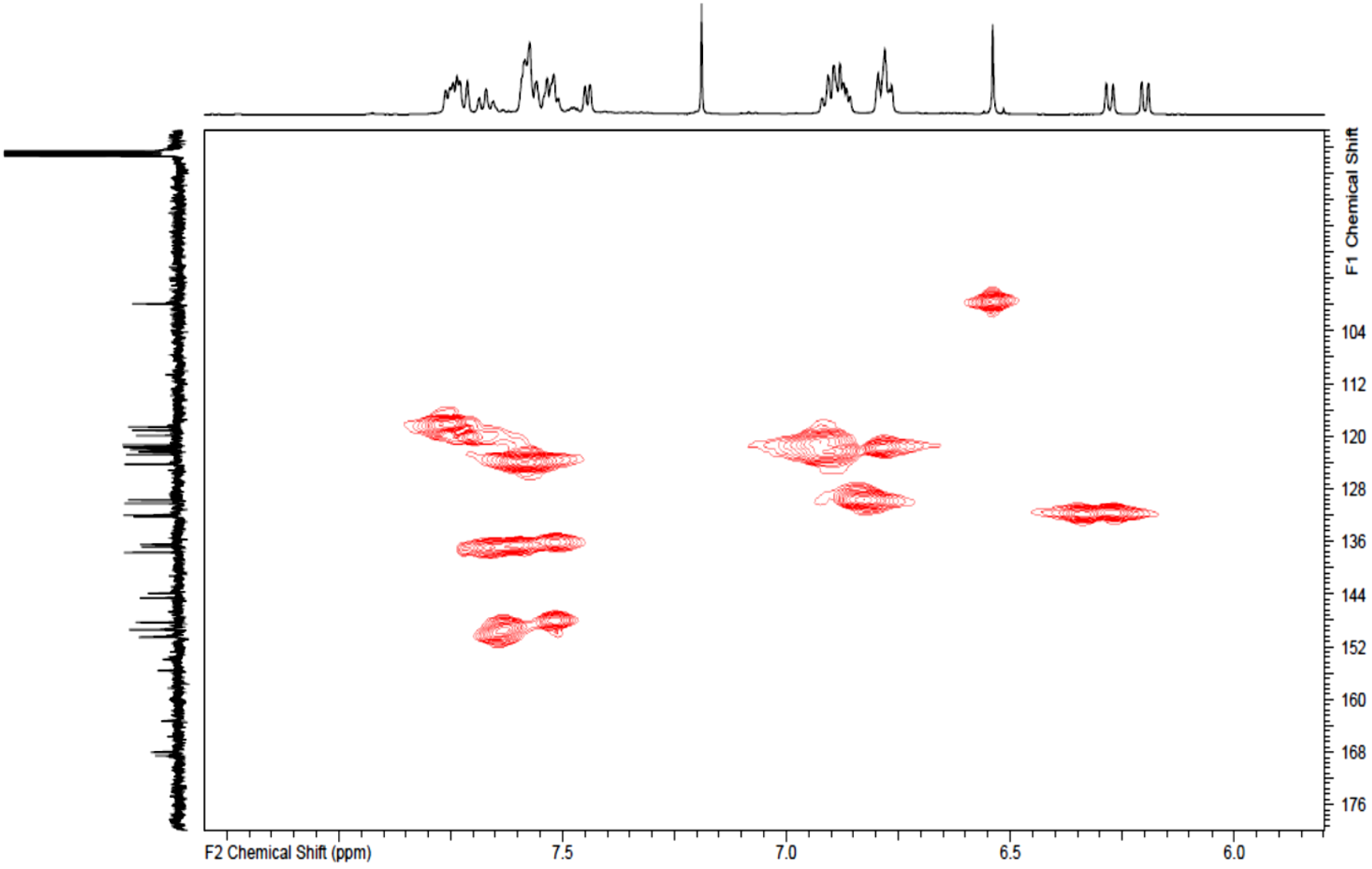
Section of the **COSY** spectrum of **3cL**$_2$



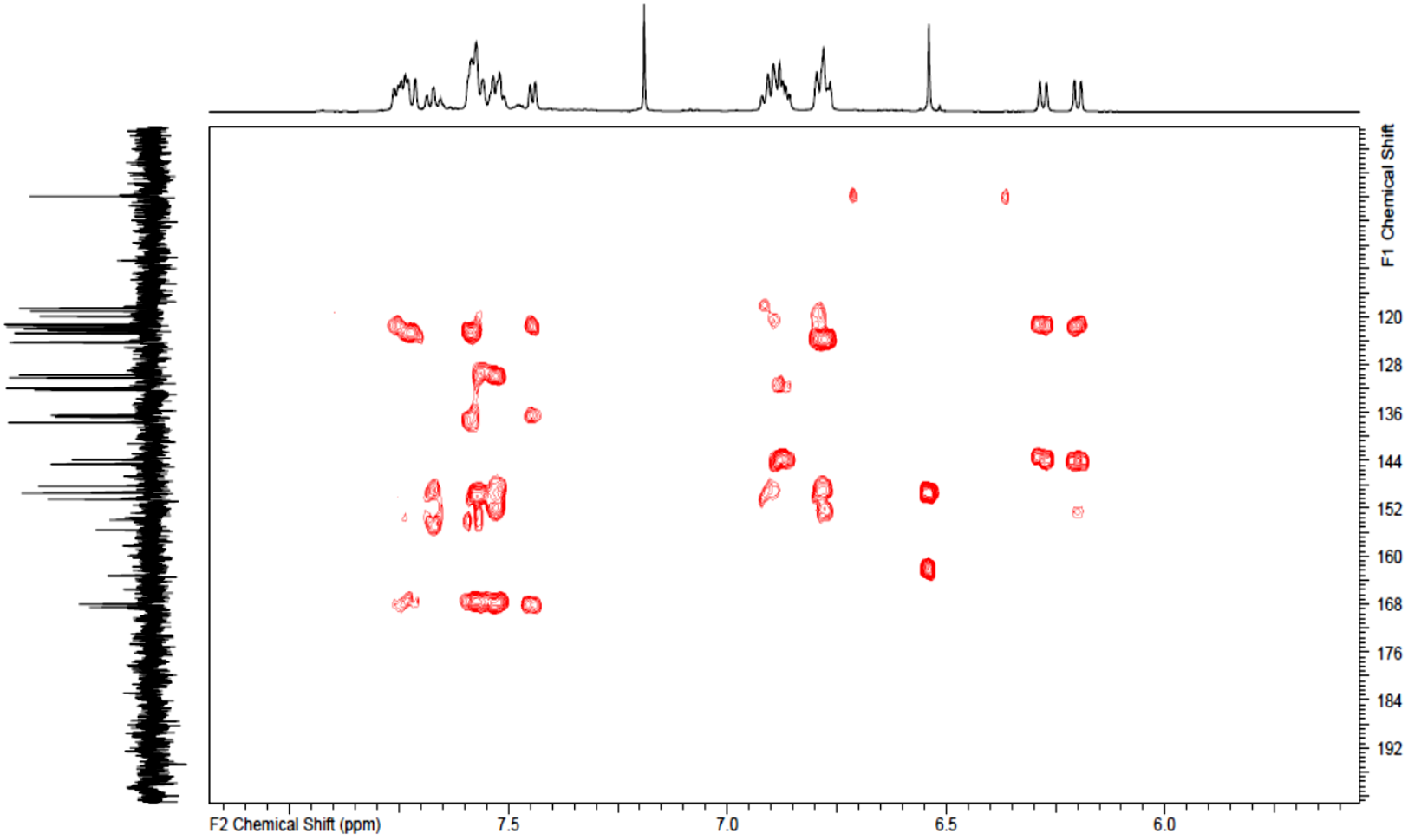
Section of the **NOESY** spectrum of **3cL$_2$**



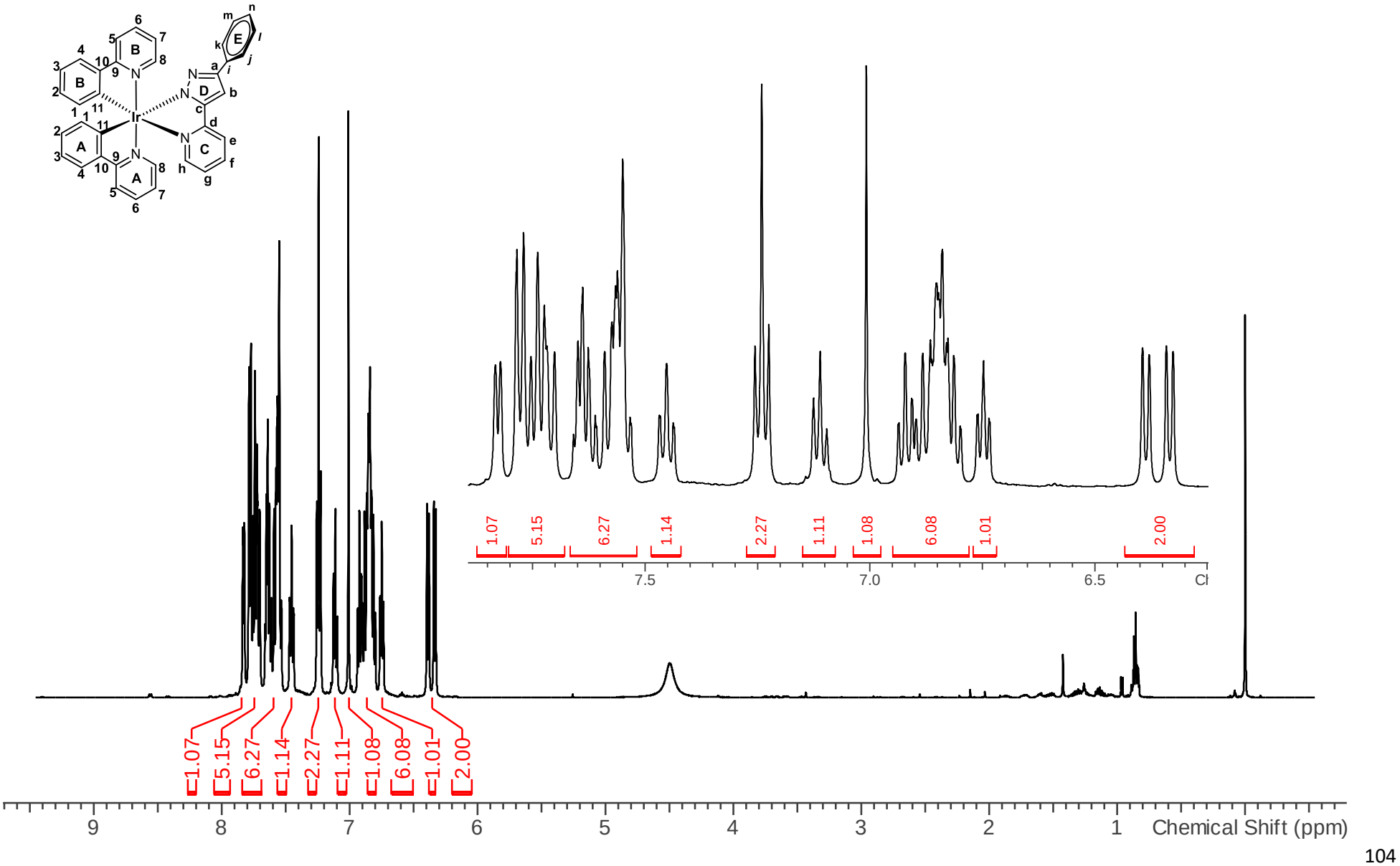
Section of the **HSQC** spectrum of **3cL$_2$**



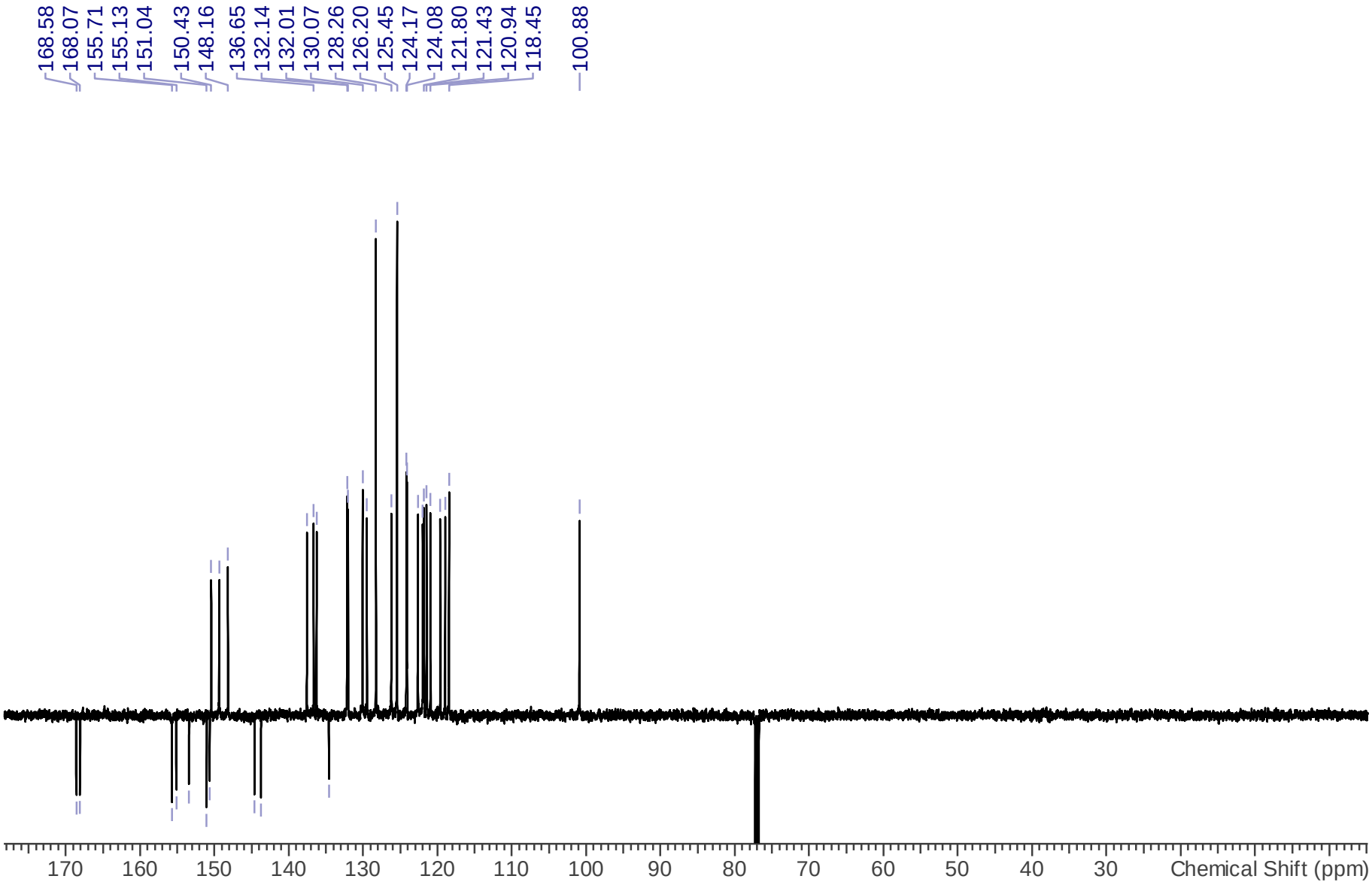
Section of the **HMBC** spectrum of **3cL$_2$**



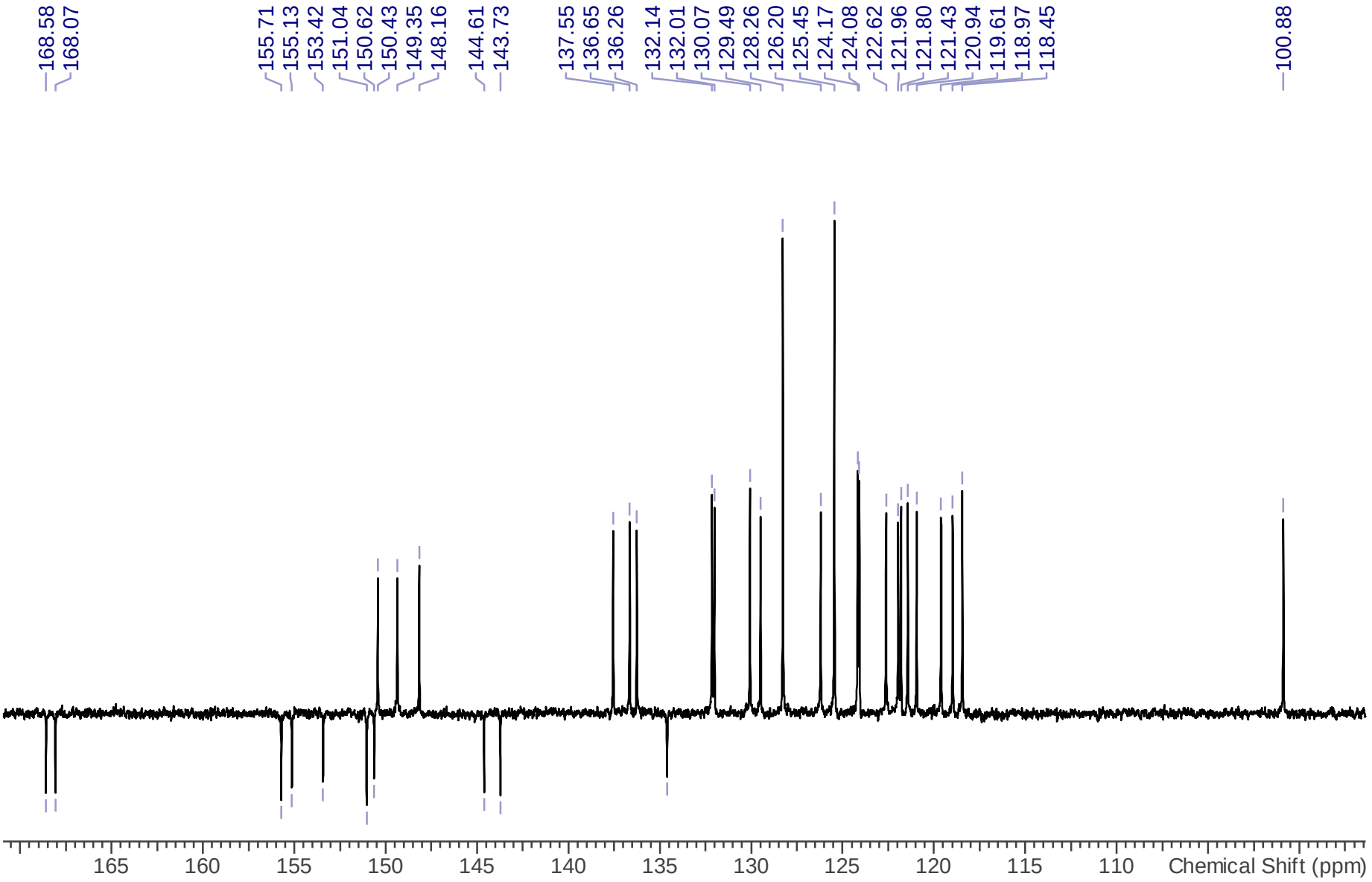
^{1}H NMR spectrum of **3cL$_3$**



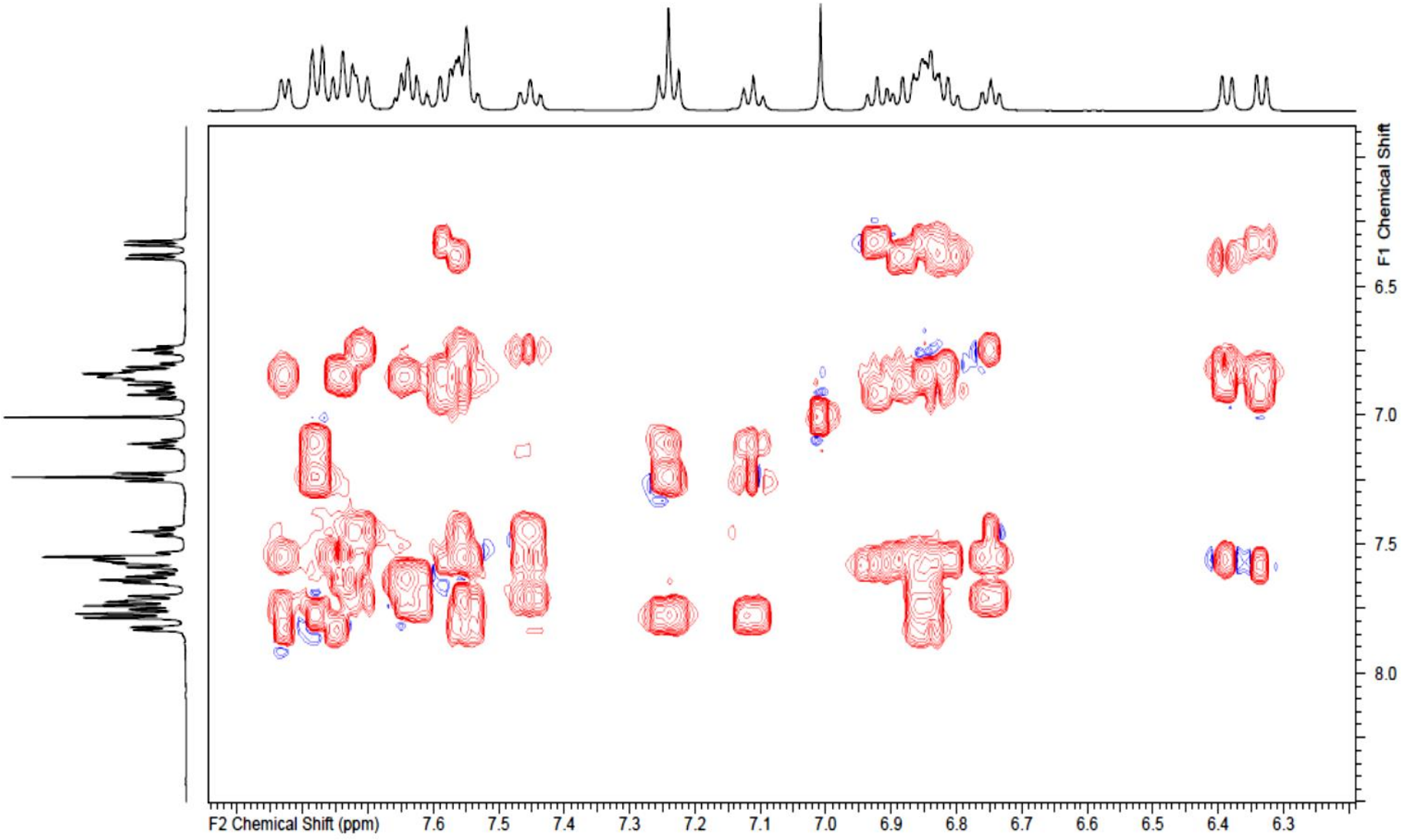
^{13}C NMR APT spectrum of **3cL$_3$**



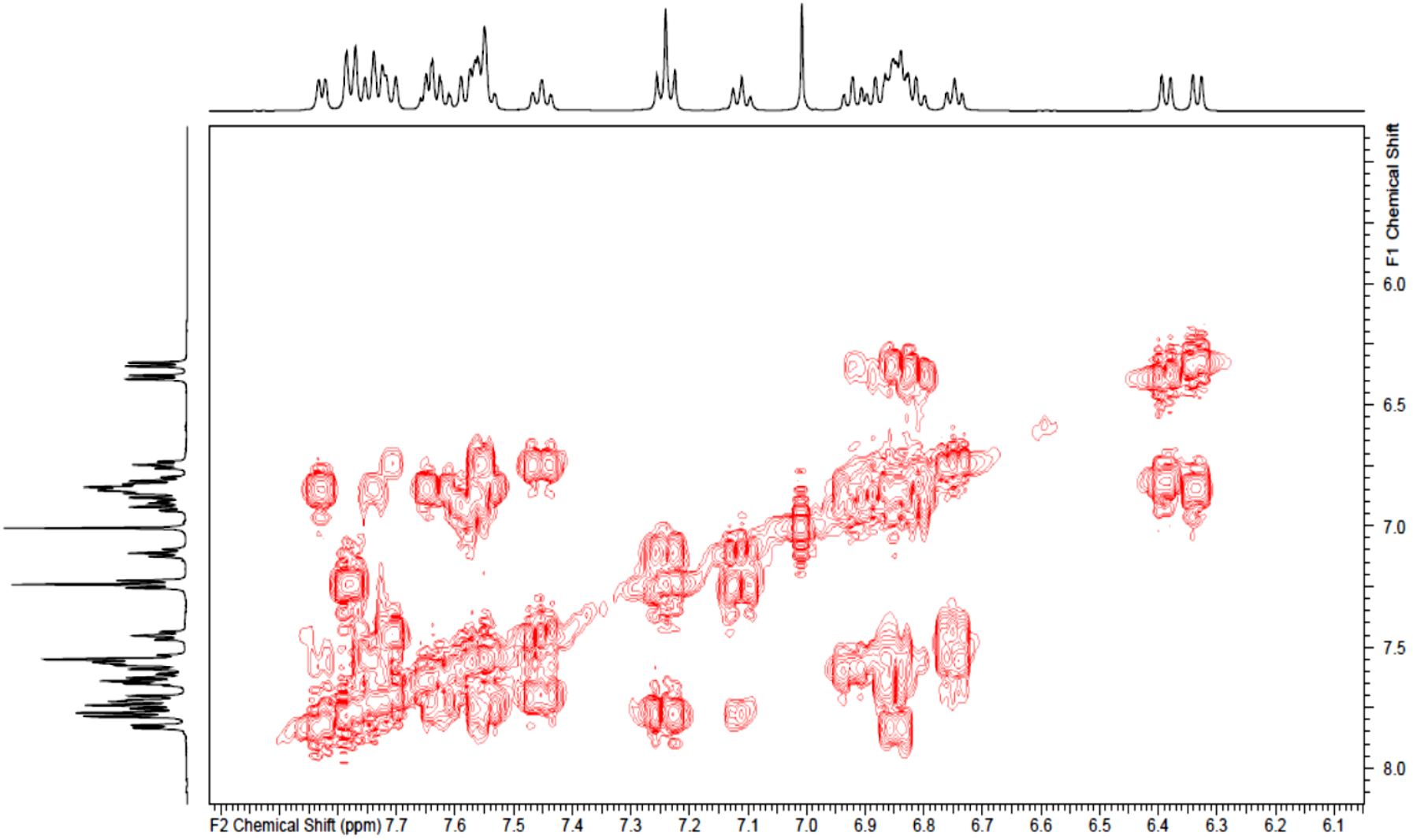
Section of ^{13}**C NMR APT** spectrum of **3cL$_3$**



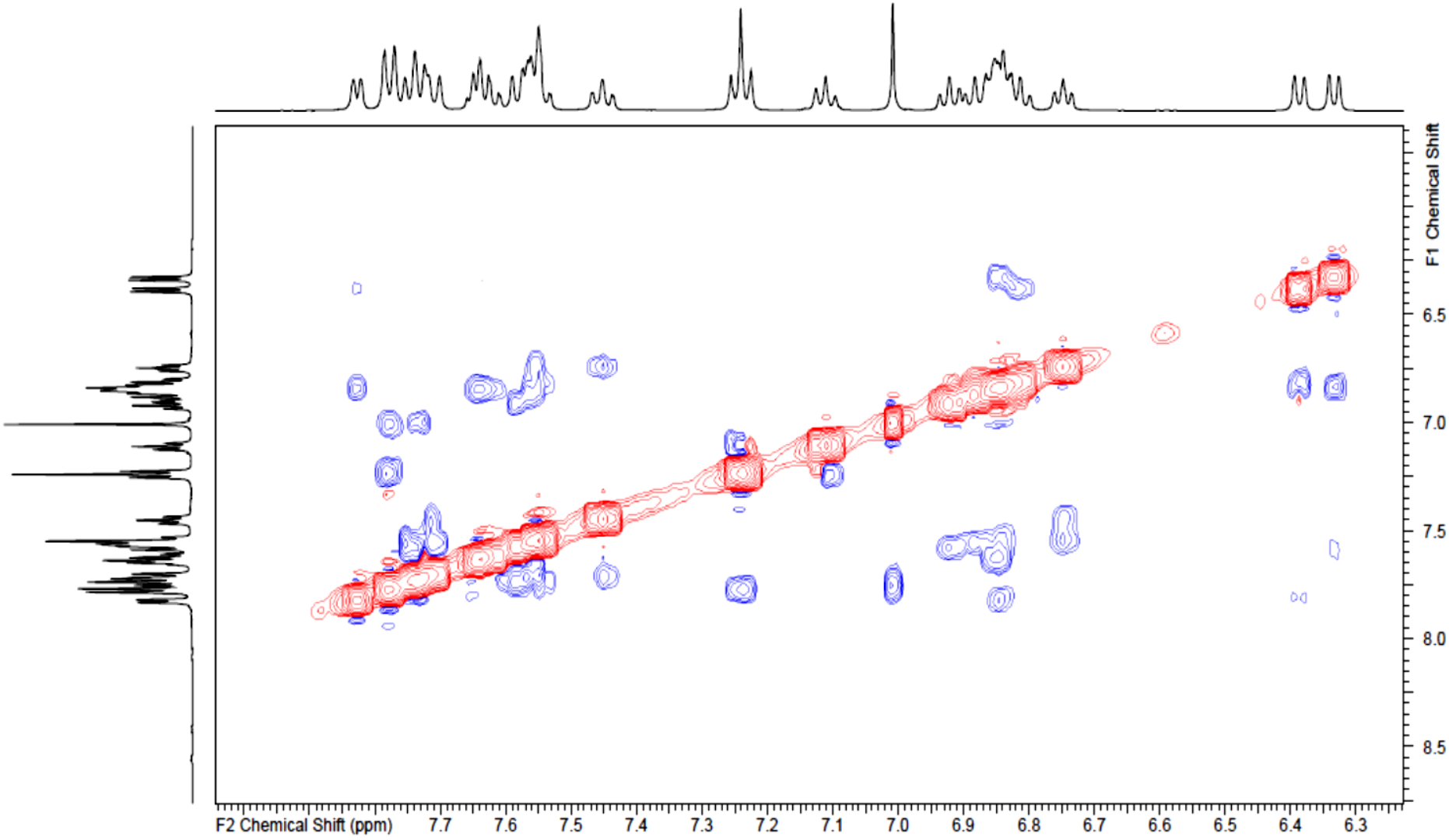
Section of the **TOCSY** spectrum of **3cL$_3$**



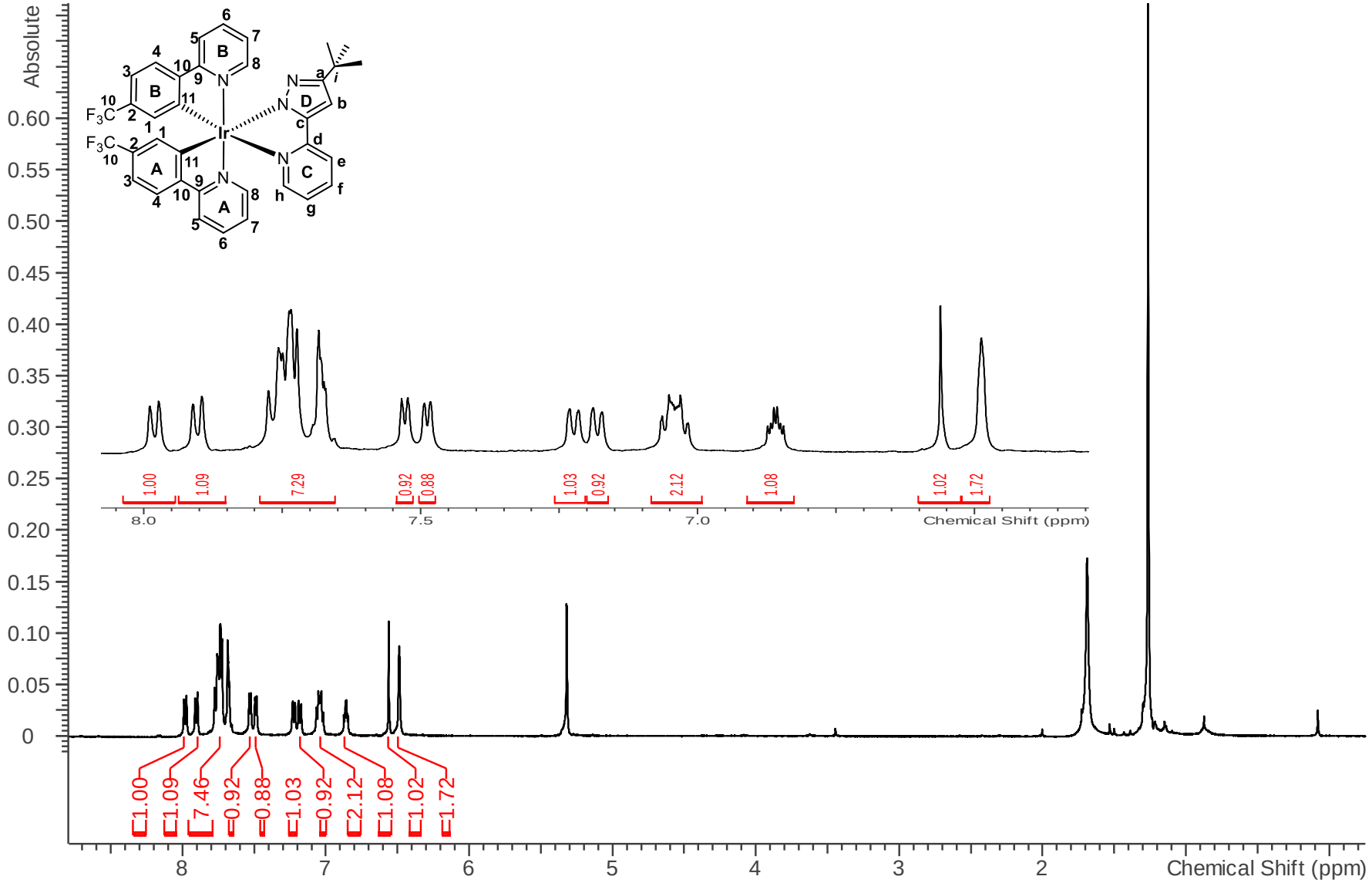
Section of the **COSY** spectrum of **3cL$_3$**



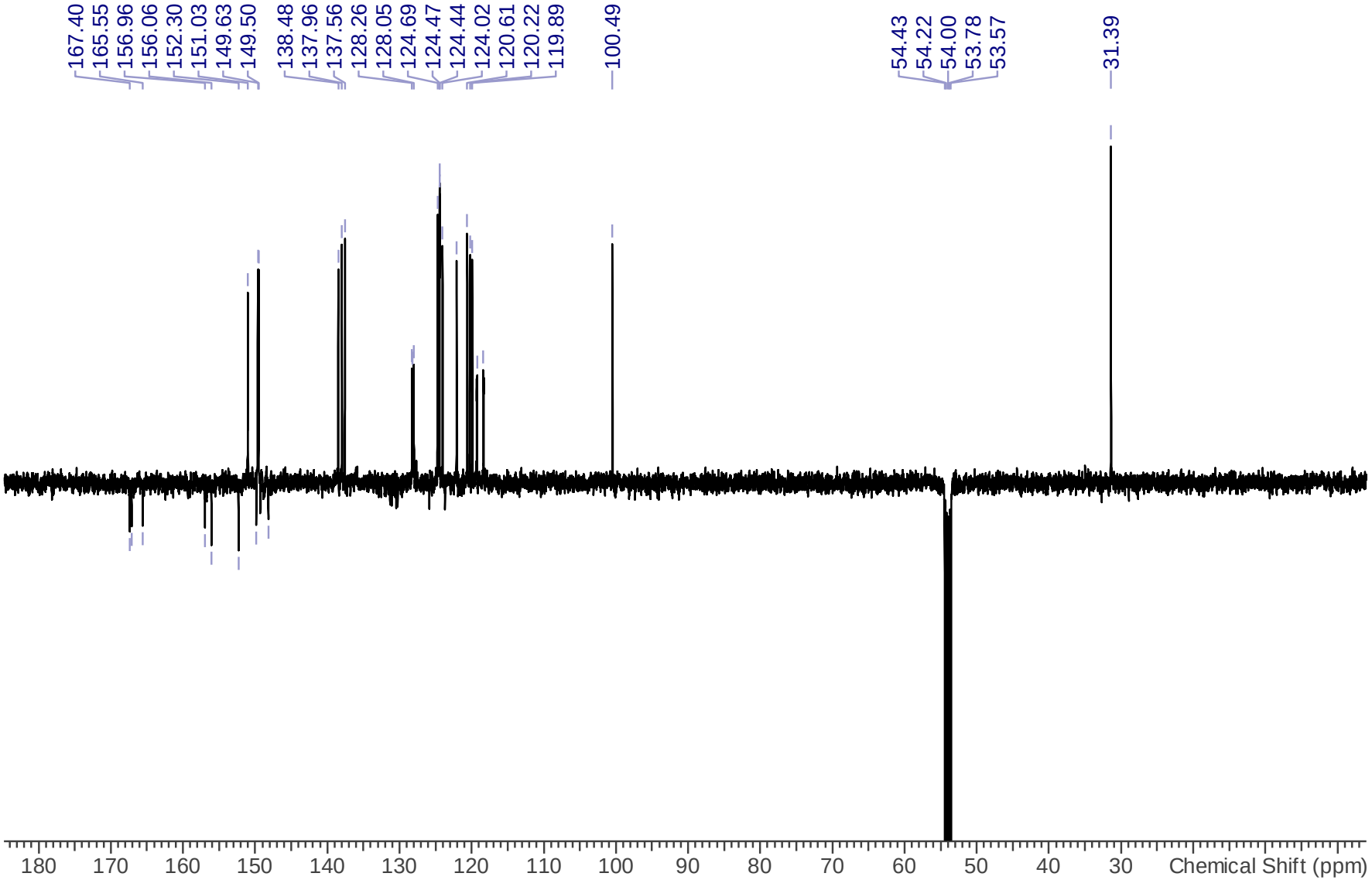
Section of the **NOESY** spectrum of **3cL**$_3$



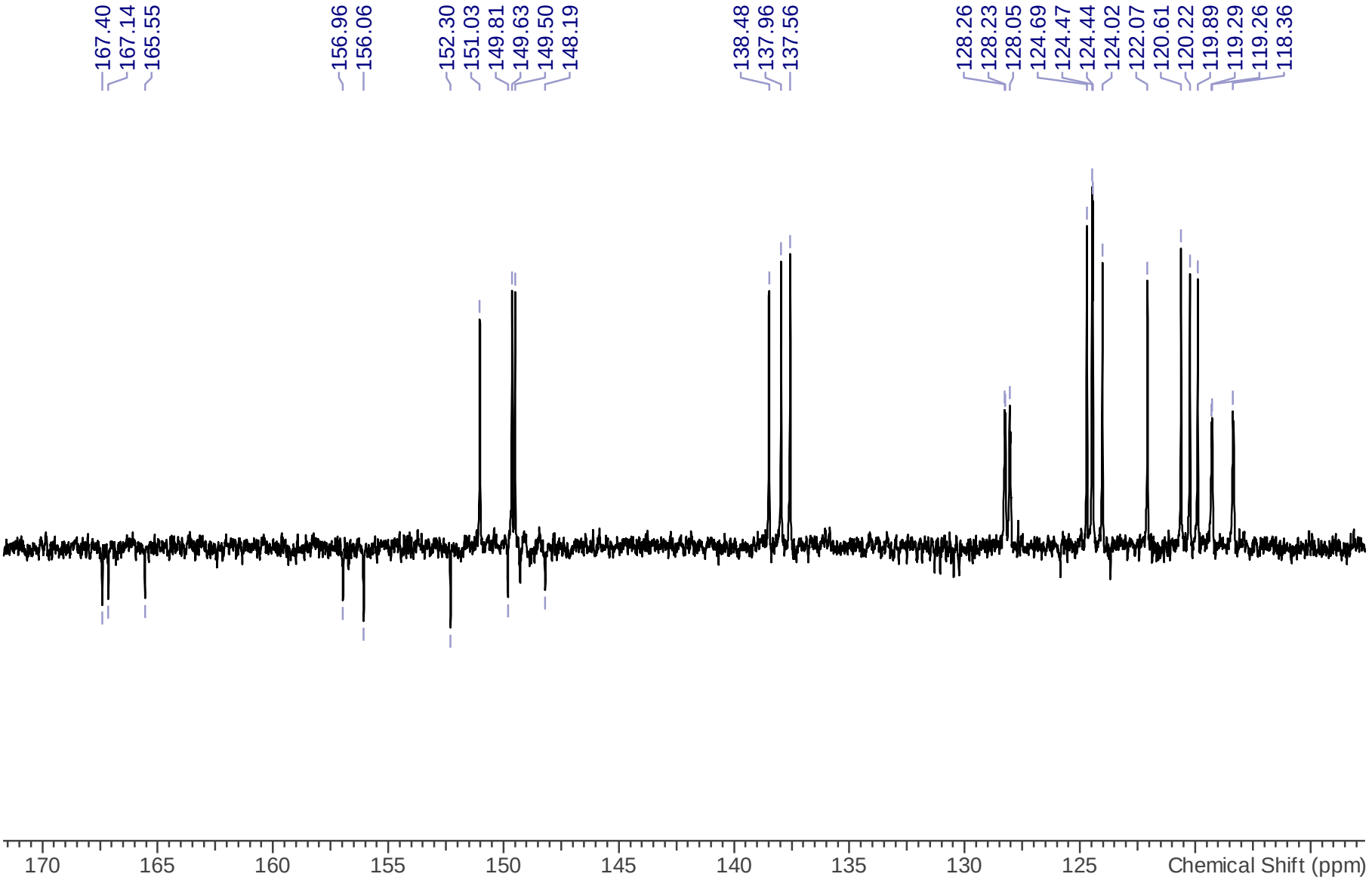
^{1}H NMR spectrum of **3dL$_2$**



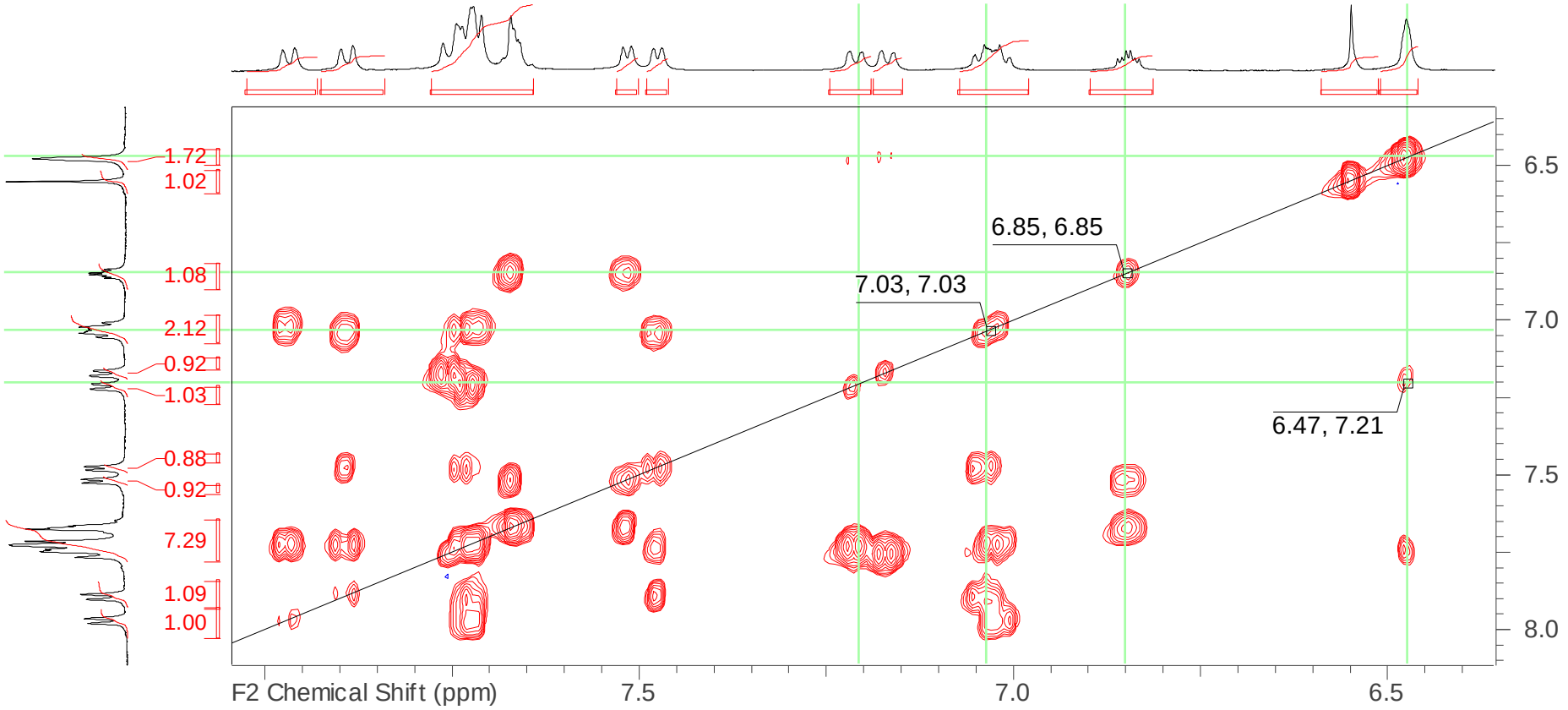
^{13}C NMR APT spectrum of **3dL$_2$**



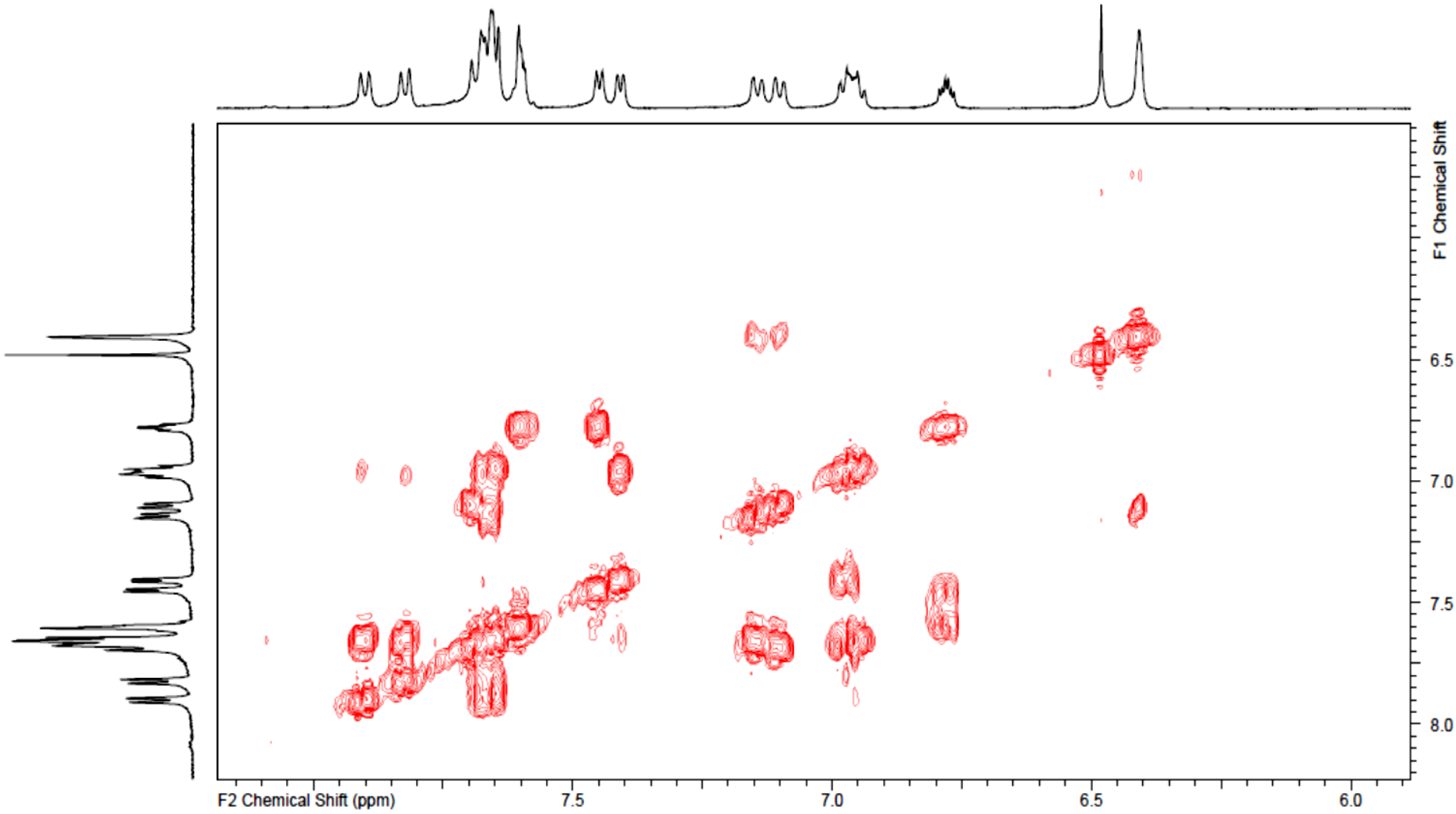
Section of ^{13}**C NMR APT** spectrum of **3dL$_2$**



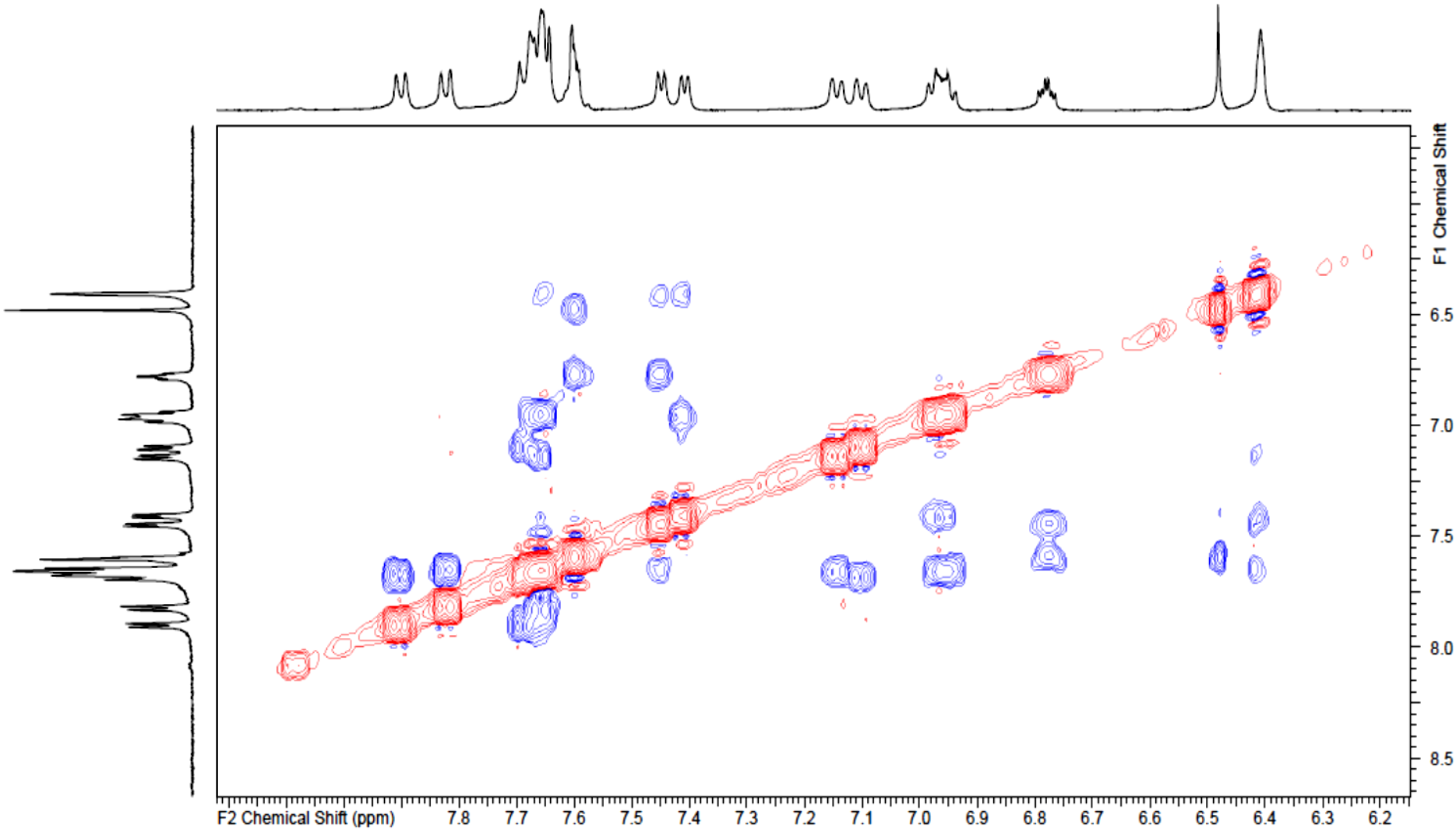
Section of the **TOCSY** spectrum of **3dL$_2$**



Section of the **COSY** spectrum of **3dL$_2$**



Section of the **NOESY** spectrum of **3dL$_2$**



^{19}F NMR spectrum of **3**