

C8953
NMR strukturní analýza
seminář

Identification of an unknown compound

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Task 0: Classification of an unknown substance

Assign the general name to displayed substances:

CARBOHYDRATE

PEPTIDE

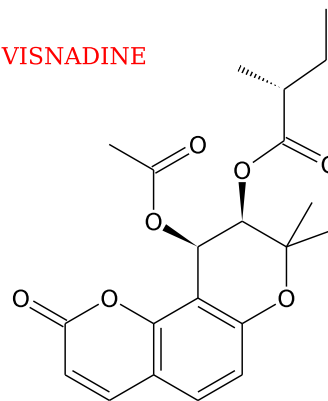
STEROID

TERPENE

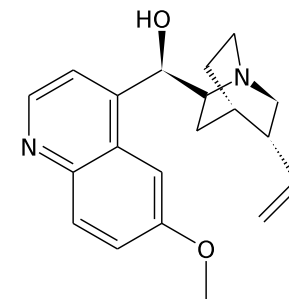
ALKALOID

COUMARINE

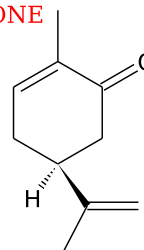
VISNADINE



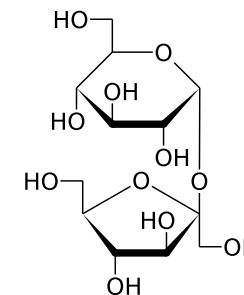
QUININE



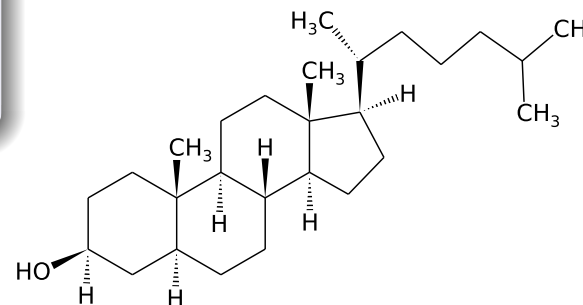
CARVONE



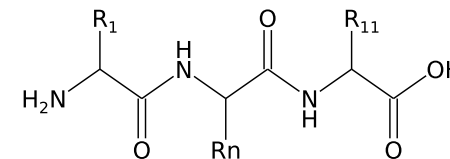
SACHAROSA



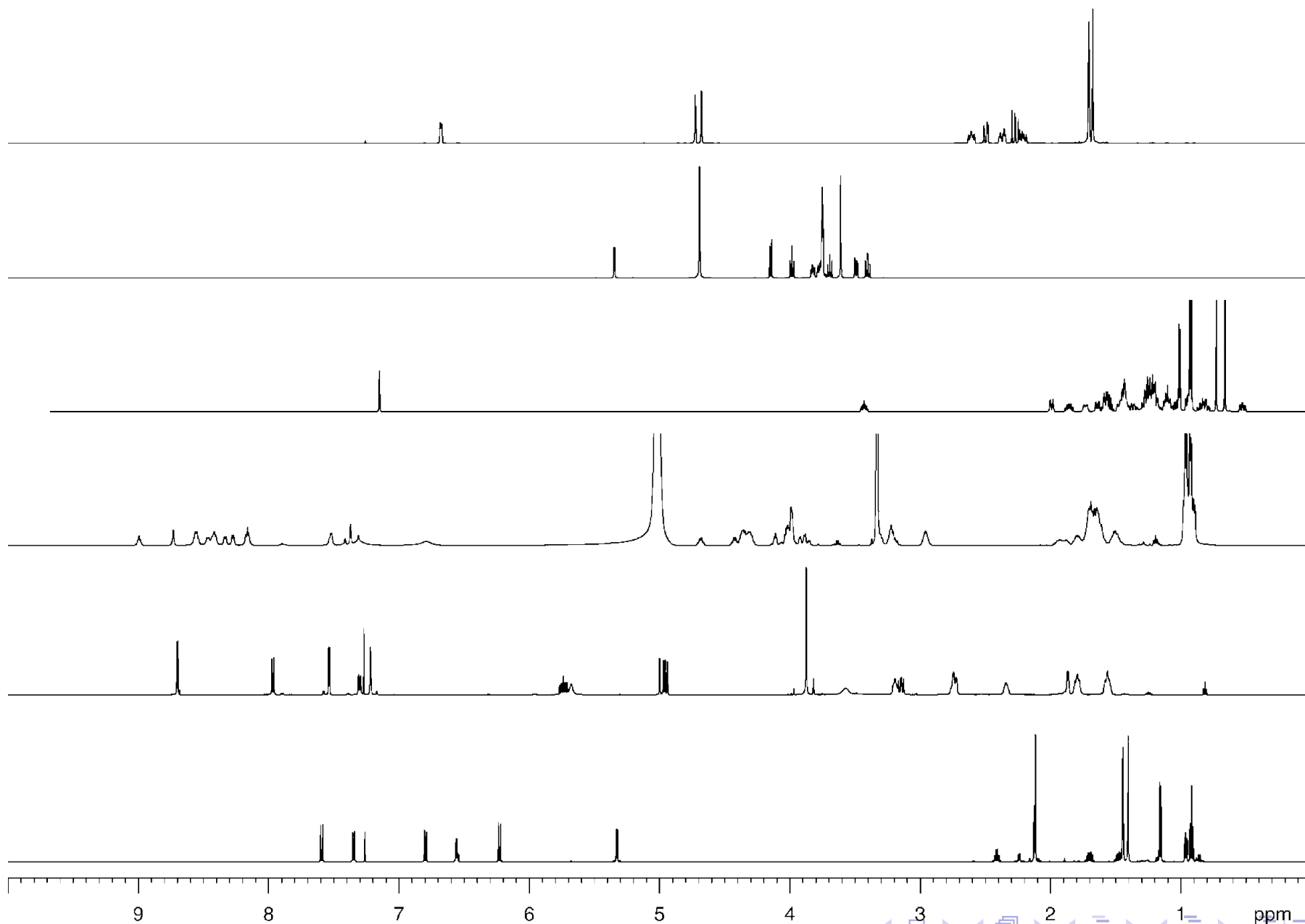
DIHYDROCHOLESTEROL



SGGLRLHLGLS



Task 0: Classification of an unknown substance



Task 1: $C_{10}H_{13}NO$

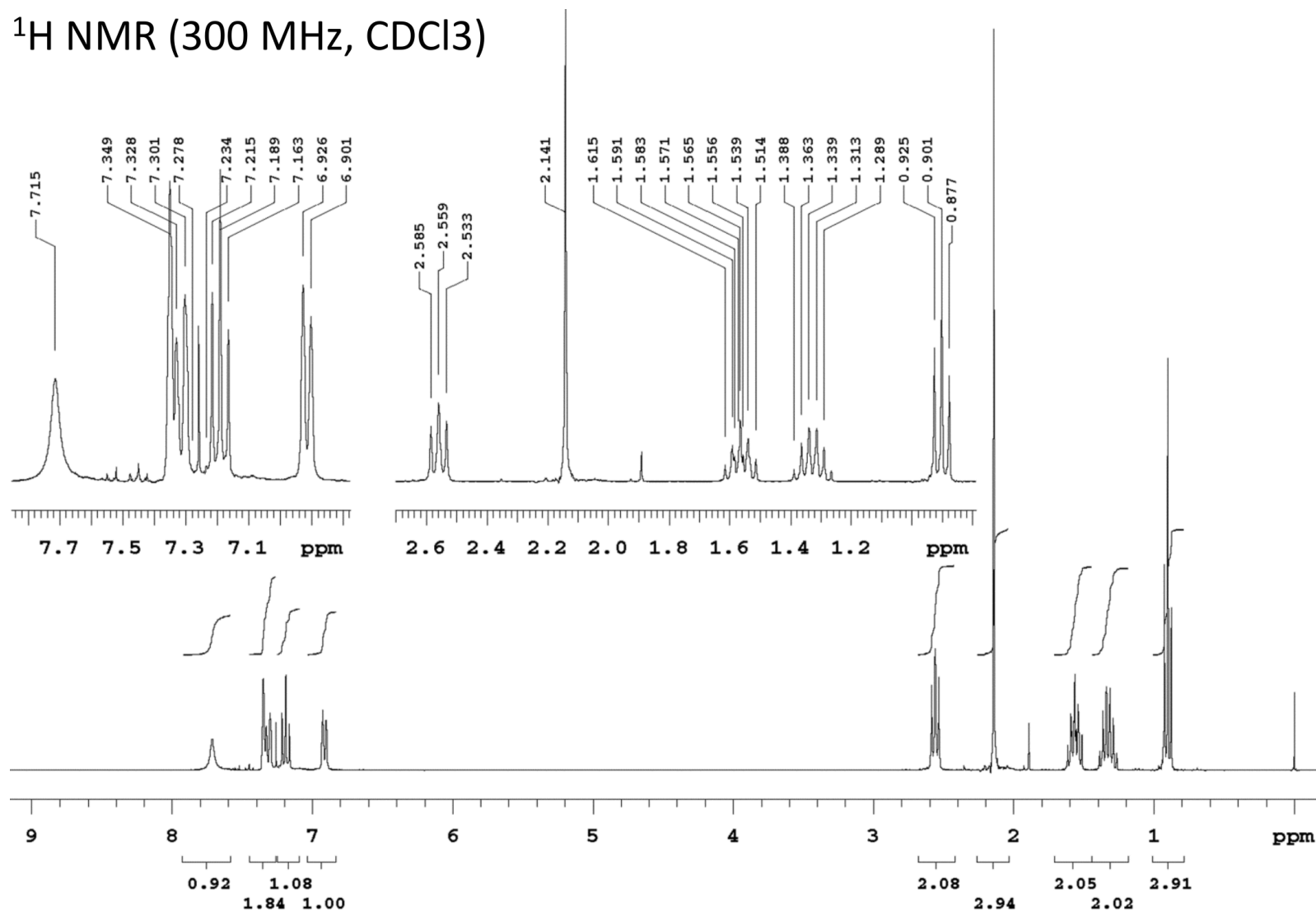
δ [ppm]	Multiplicity	Integral
1.05	triplet	3
1.75	singlet	3
3.70	quartet	2
7-7.60	complex multiplet	5

Task 2: $C_{11}H_{15}NO_2$

δ [ppm]	Multiplicity	J (Hz)	Integral
1.30	triplet	7	3
3.00	singlet	-	6
4.25	quartet	7	2
6.65	dublet	8	2
7.80	dublet	8	2

Task 3: $C_{12}H_{17}NO$ - $^1H/COSY$

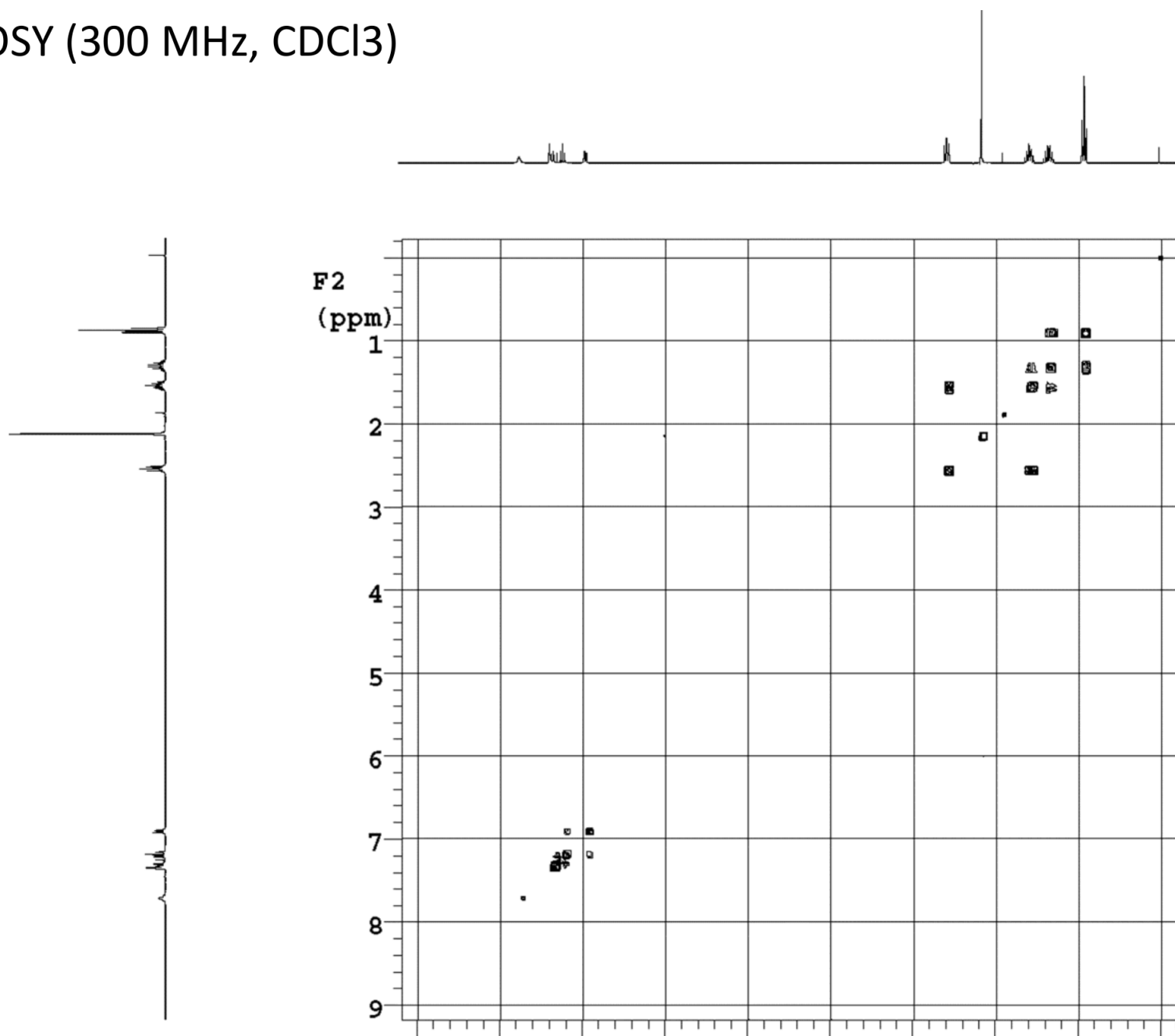
1H NMR (300 MHz, $CDCl_3$)



SOLUTION

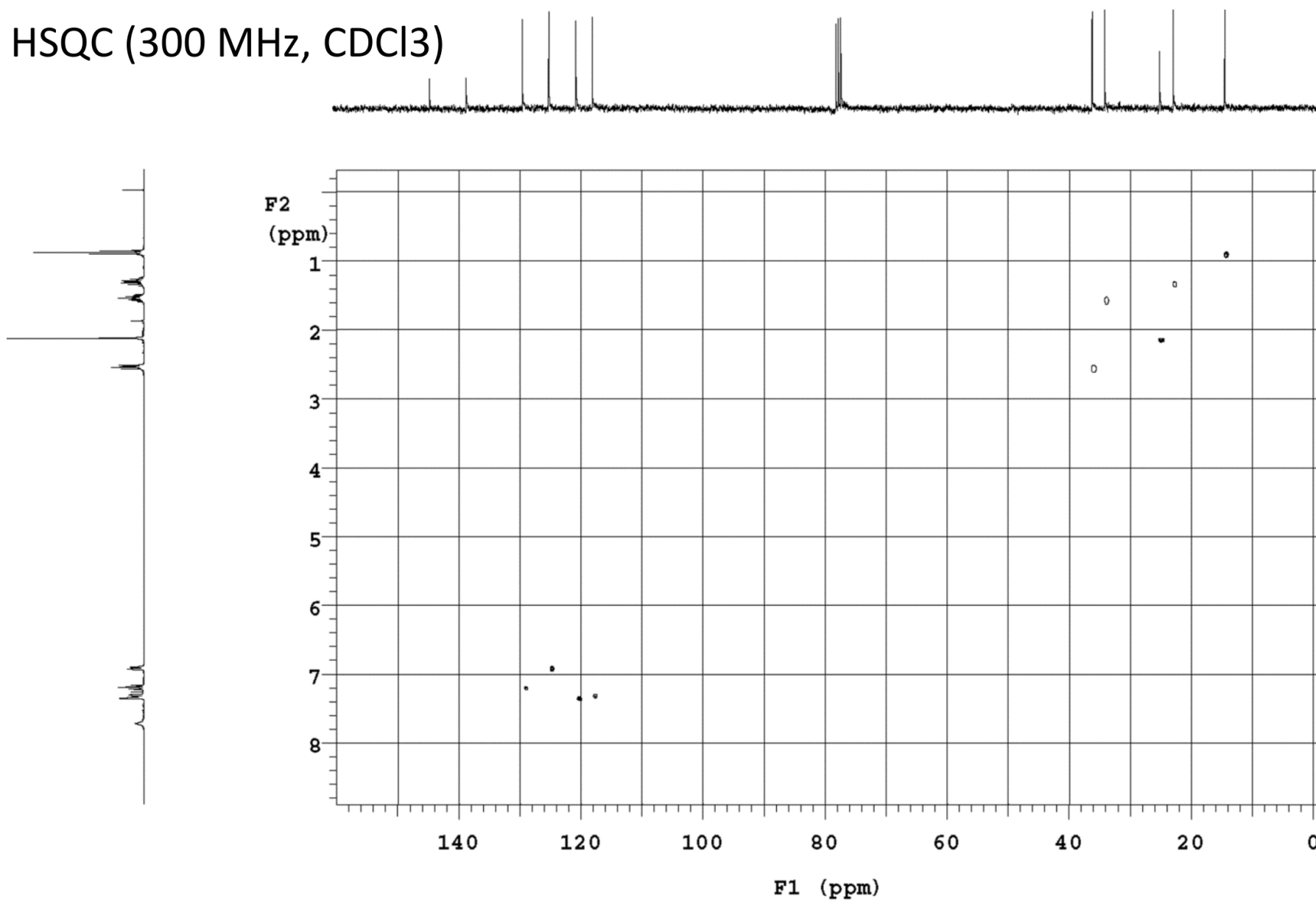
Task 3: $C_{12}H_{17}NO$ - $^1H/COSY$

COSY (300 MHz, $CDCl_3$)



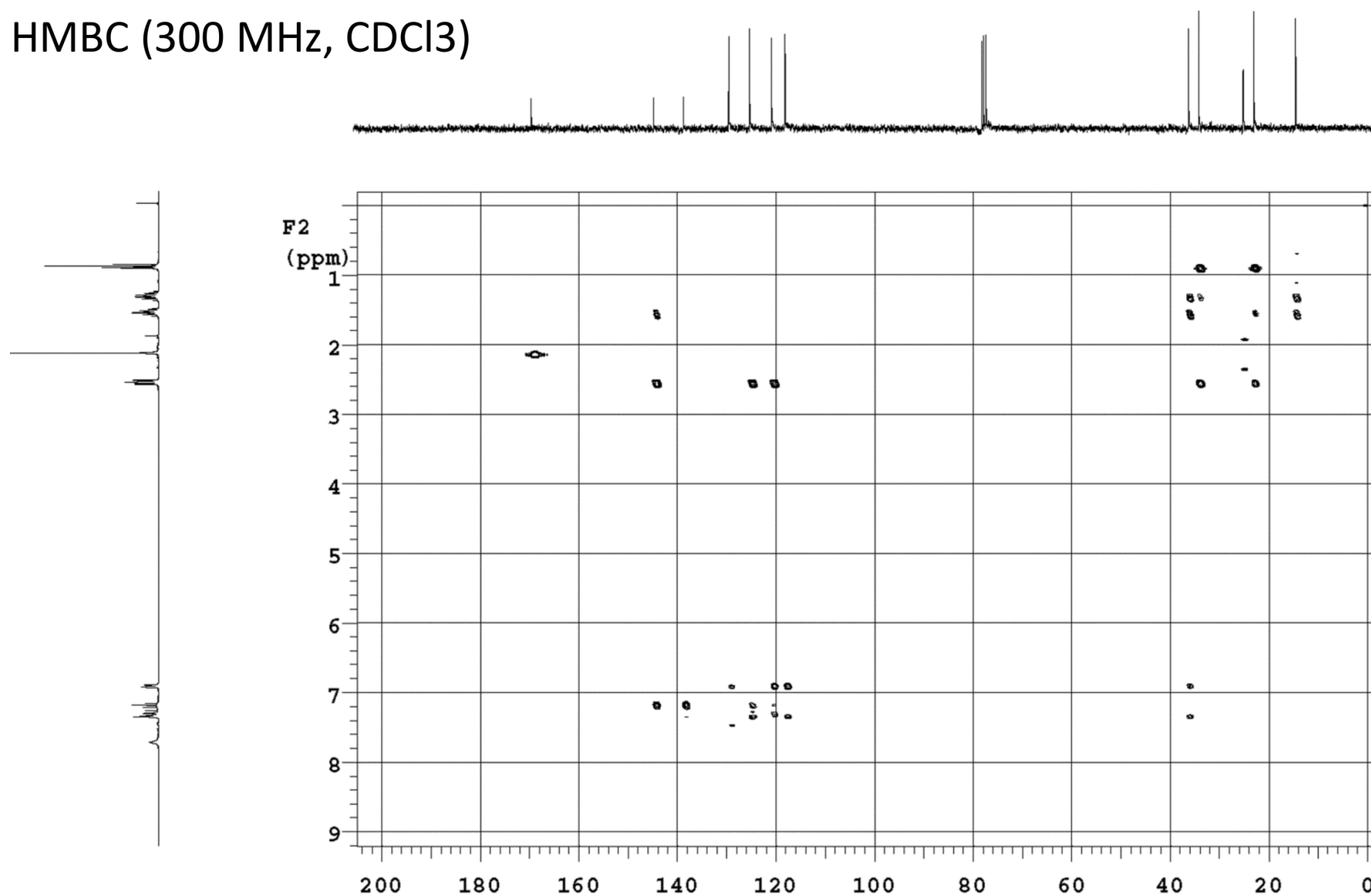
Task 3: $C_{12}H_{17}NO$ - 1H - ^{13}C /HSQC, HMBC

HSQC (300 MHz, $CDCl_3$)



Task 3: $C_{12}H_{17}NO$ - 1H - ^{13}C /HSQC, HMBC

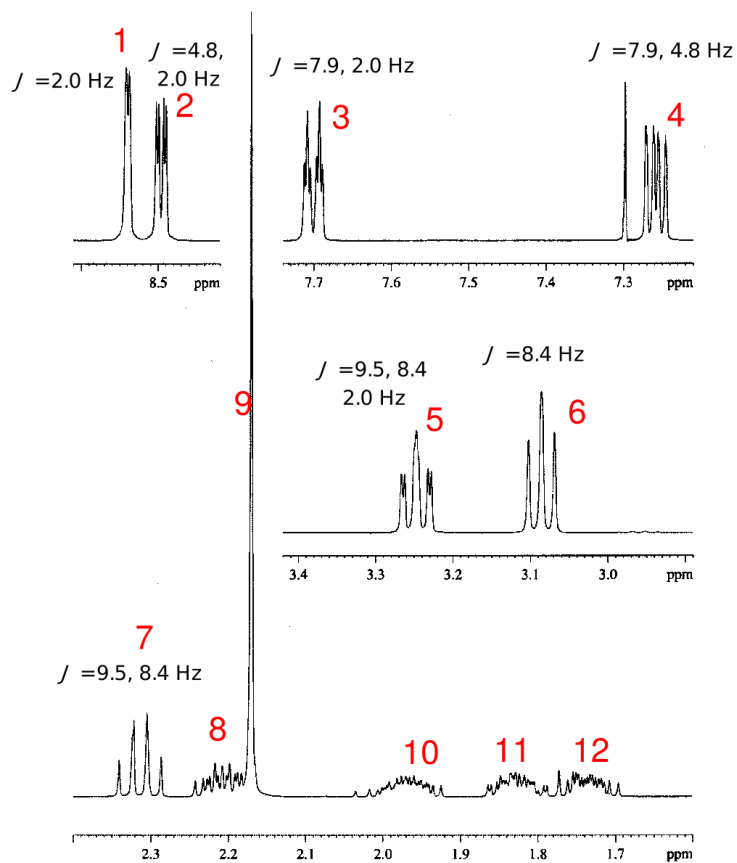
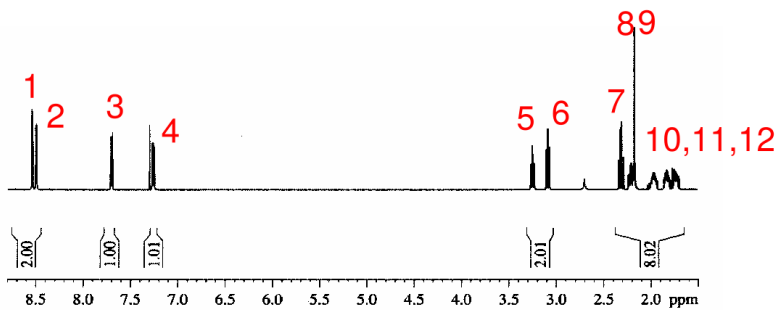
HMBC (300 MHz, $CDCl_3$)



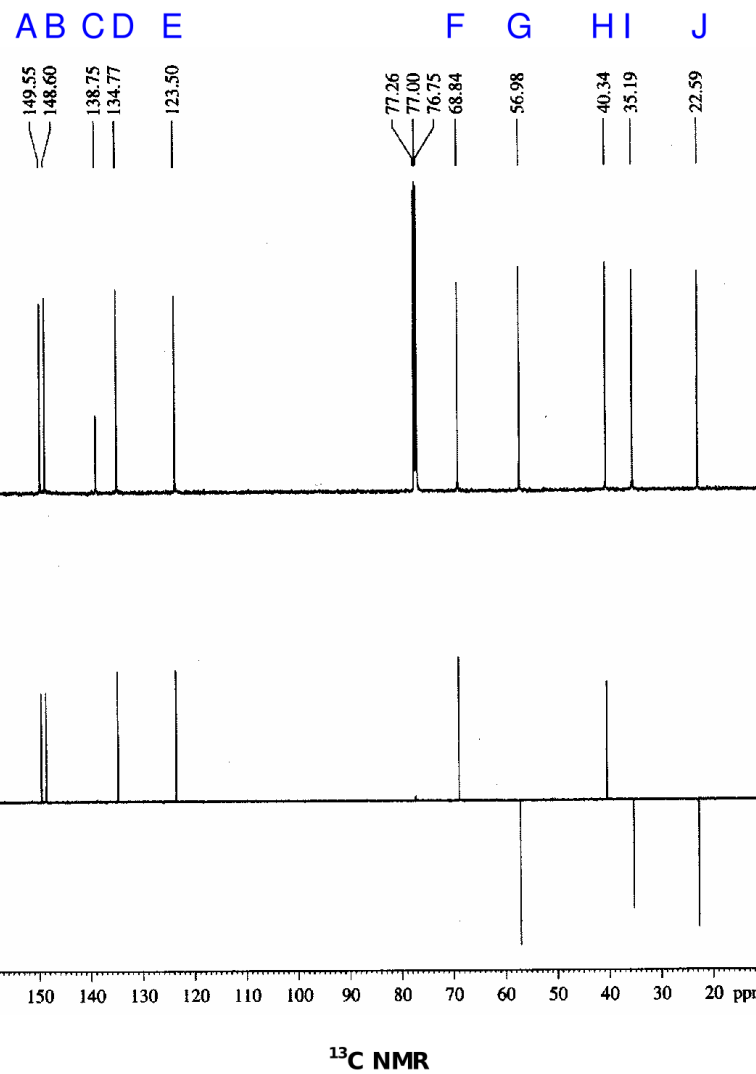
General comments

- inspect molecular formula $C_mH_hO_oN_nX_x$:
Degree of unsaturation $m + 1 - 0.5(h + x - n)$
- identify signals of CH_3 and exchangeable protons in 1D 1H spectrum
- arbitrary numbering (e.g., from lower to higher value of chemical shift) of resolved resonances in all spectra
- identification of the individual spin systems using DQF-COSY
- resolve geminal protons using HSQC
- connect molecular fragments/isolated spins using HMBC, NOESY
- specify the stereochemistry (relative configuration) by means of J - and NOE interaction
- in 1D spectrum bottom blue numbers are integrals, labels in violet frames contains the arbitrary label (A-N), multiplet specification (use with caution, automatically determined), and position of a signal in ppm
- UnHa-UnHb in 2D refers to correlation of protons a and b of unknown compound Un

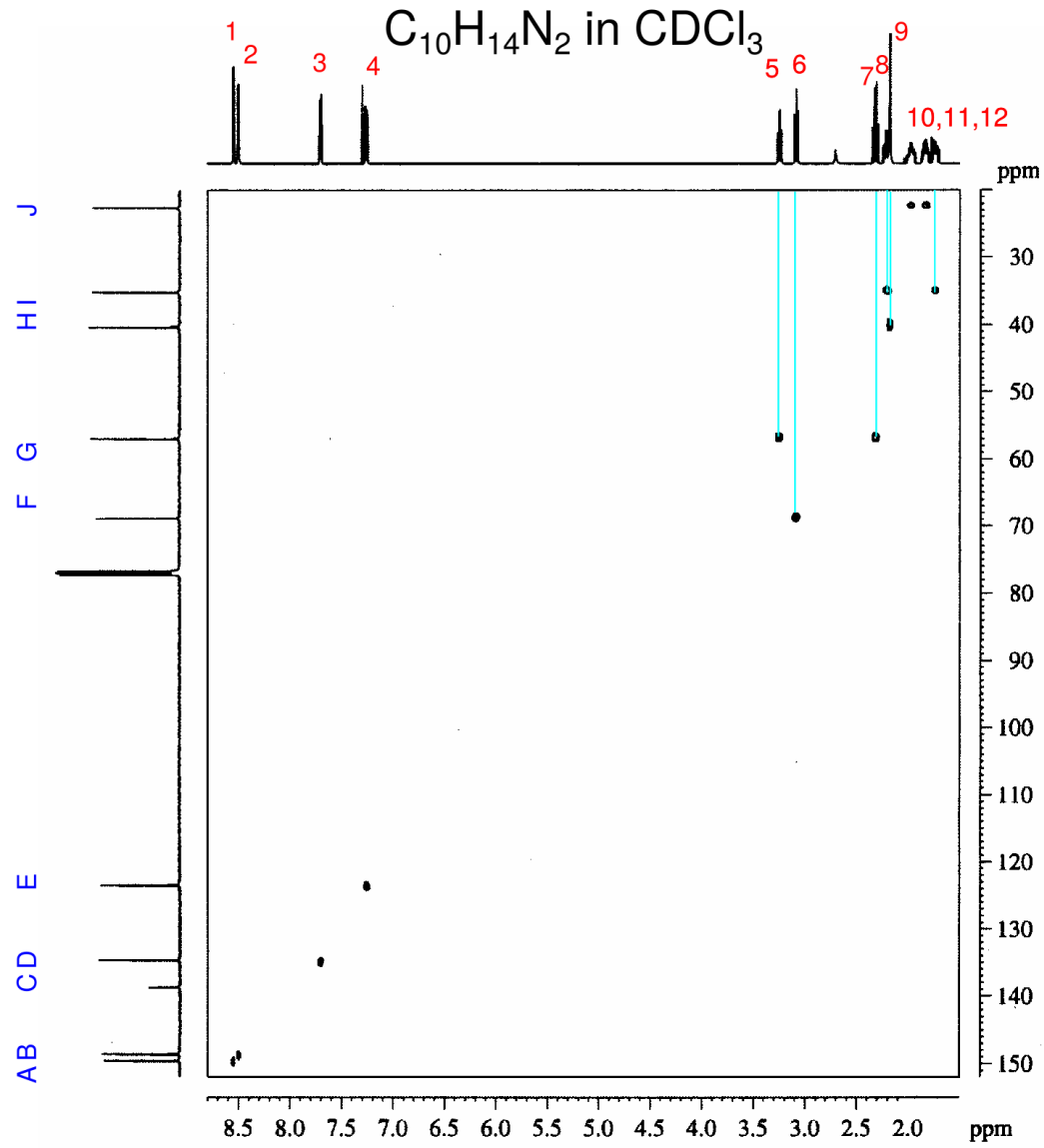
C₁₀H₁₄N₂ in CDCl₃



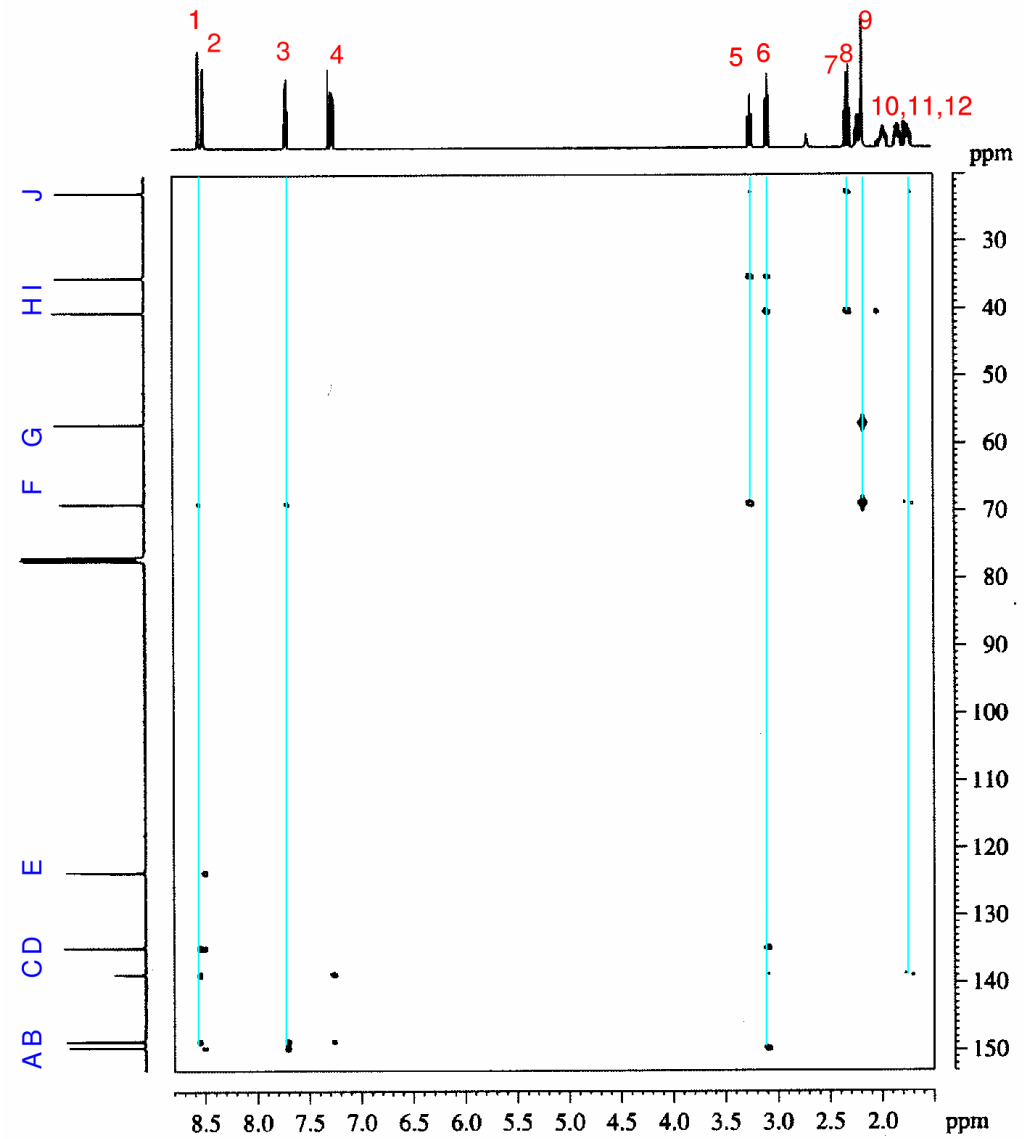
¹H NMR



¹³C NMR

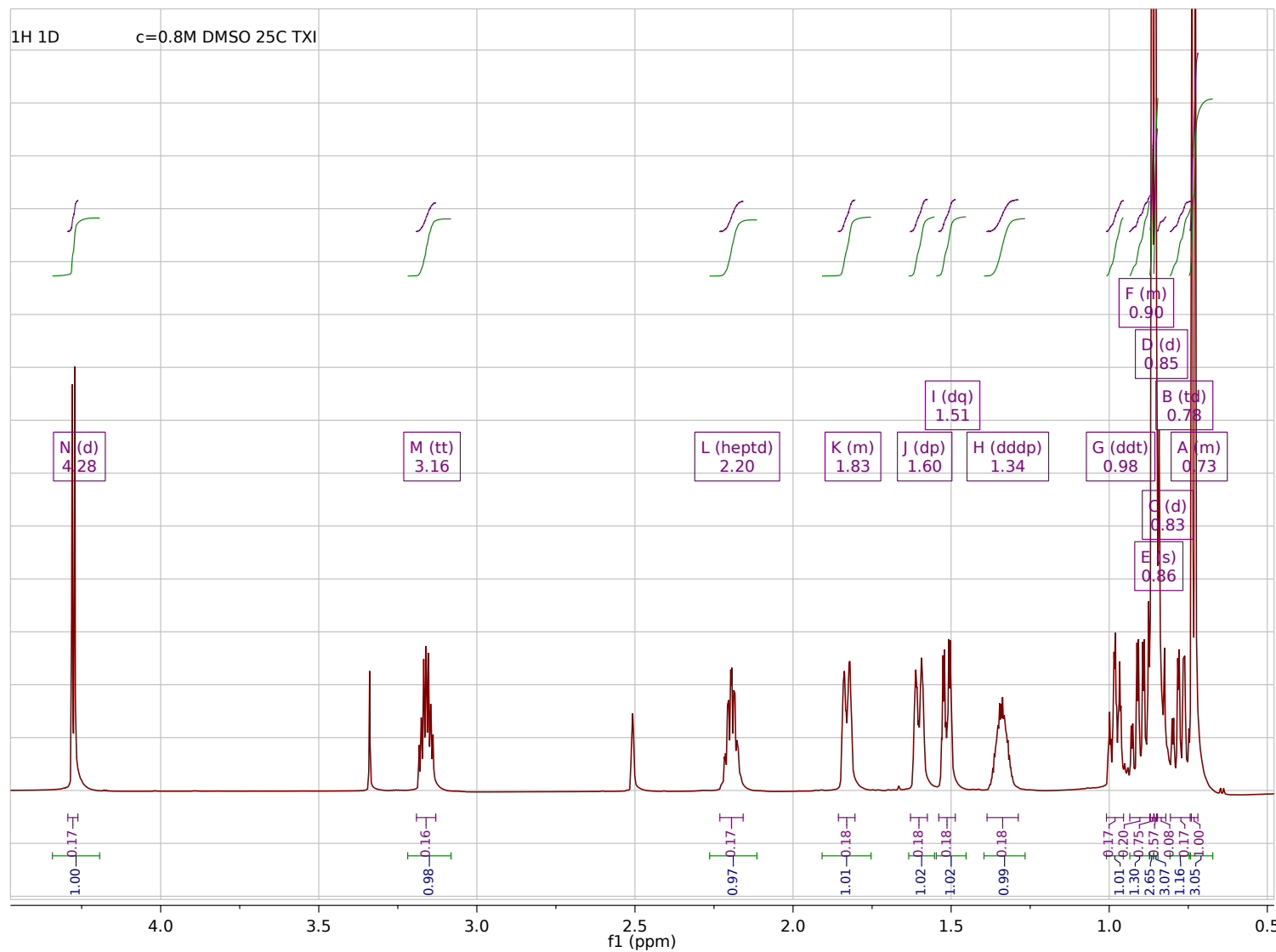


$^{13}C, ^1H$ HSQC



$^{13}C, ^1H$ HMBC

1D ^1H of $\text{C}_{10}\text{H}_{20}\text{O}$

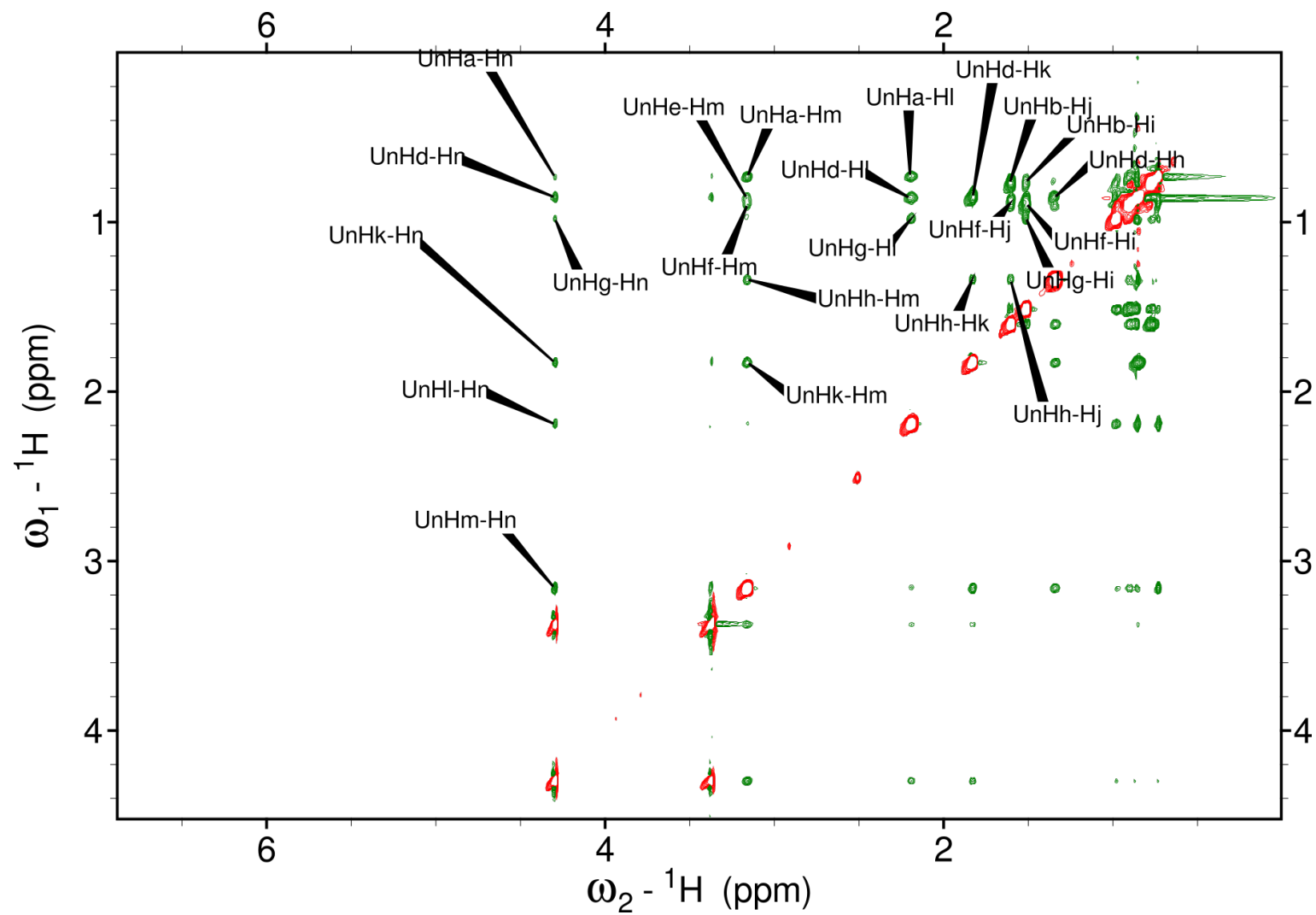
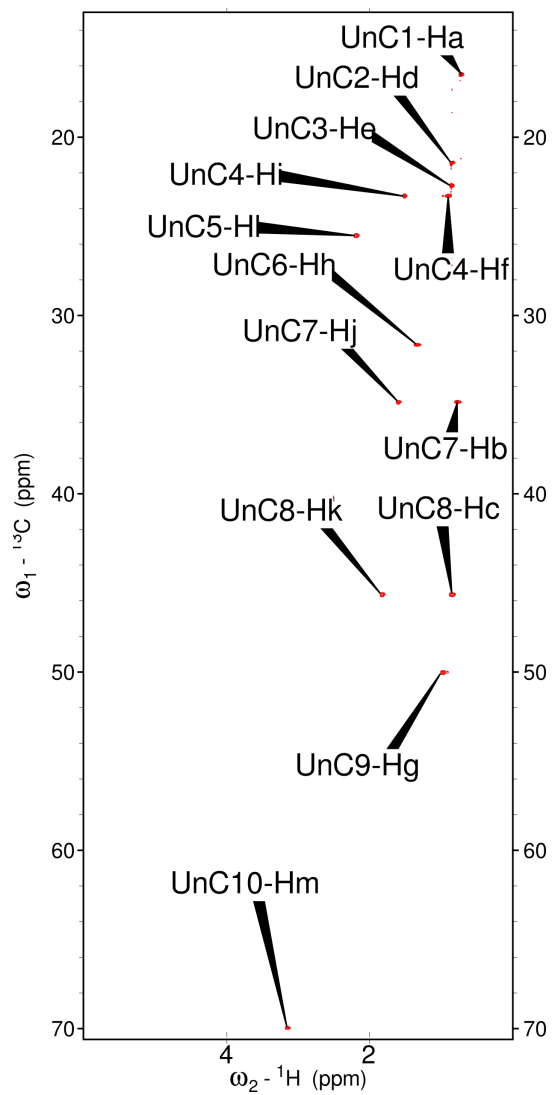


1H 1D

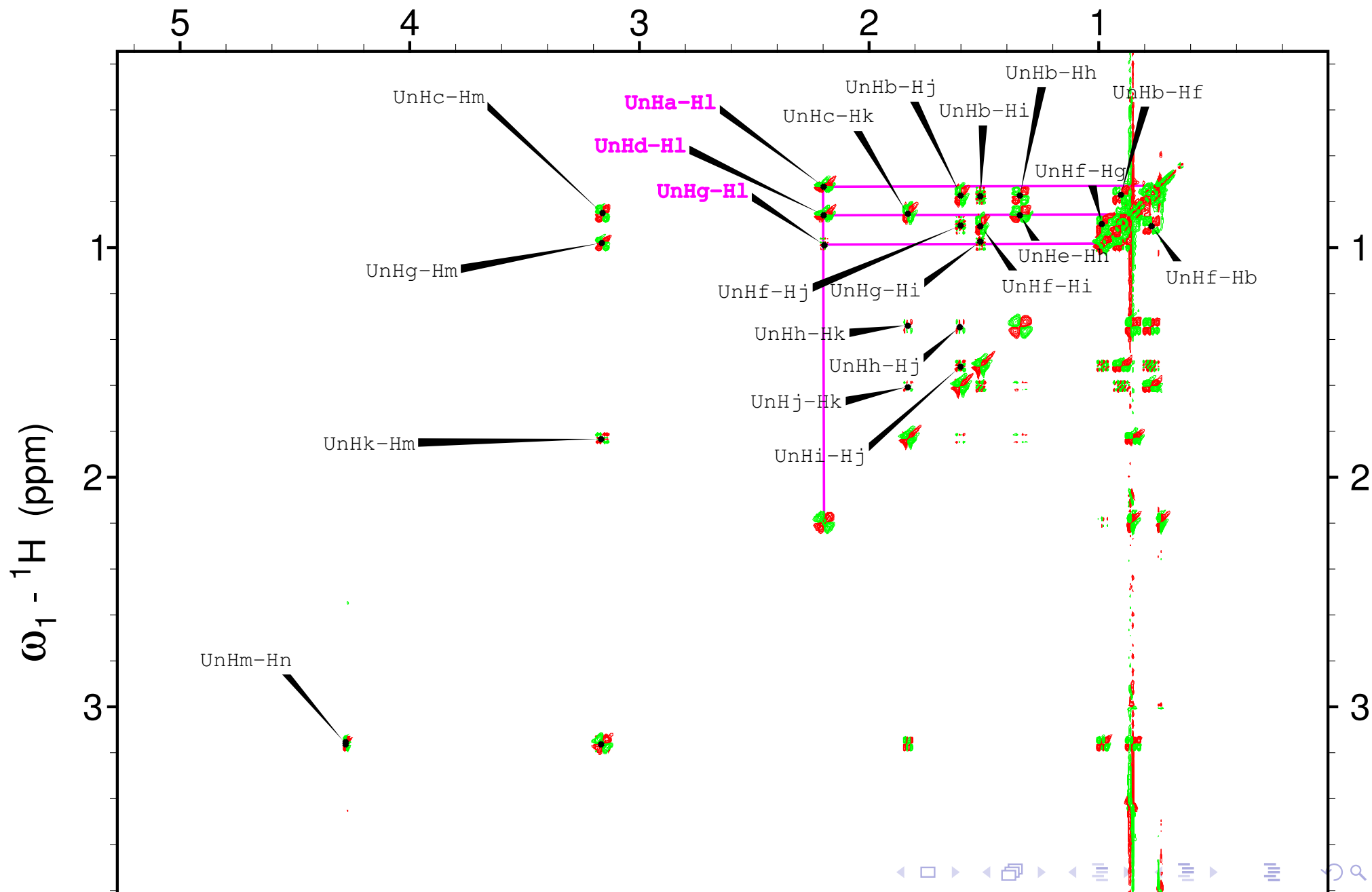
c=0.8M DMSO 25C TXI



^1H - ^{13}C HSQC and NOESY



Task 1: J -connectivity of $C_{10}H_{20}O$



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- CH_2 **8ck** weakly coupled with CH_2 **7bj \Rightarrow closing ring**
- protons **b** and **k** coupled to CH **6h** which is connected to methyl **3e**
- other expected crosspeaks in DQ-COSY crowded/overlapped, found topology confirmed in HMBC ($^{3/4}J_{HC}$)

