

## General comments

- inspect molecular formula  $C_m H_h O_o N_n X_x$ :  
Degree of unsaturation  $\mathbf{m} + \mathbf{1} - \mathbf{0.5}(\mathbf{h} + \mathbf{x} - \mathbf{n})$
- 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

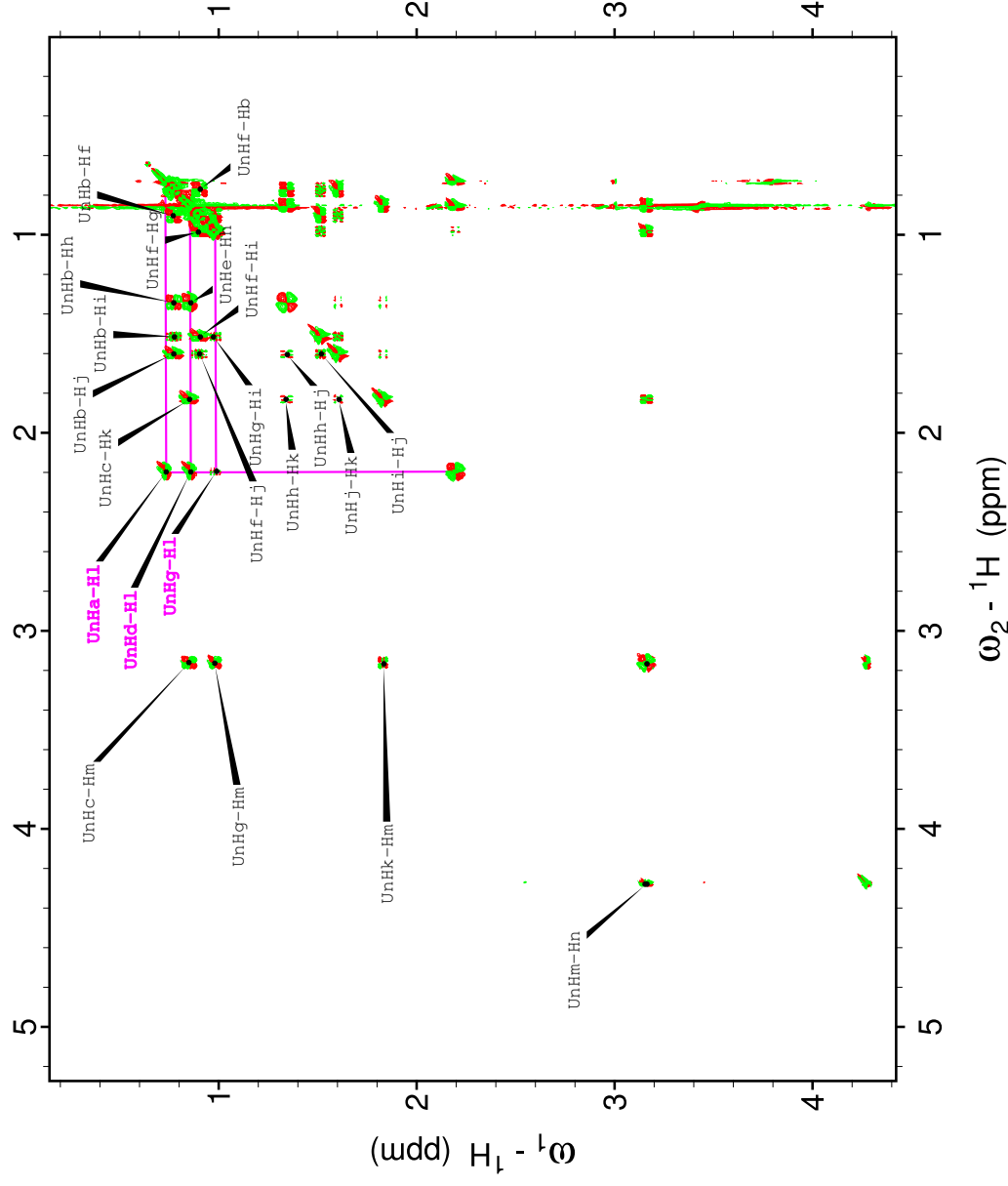
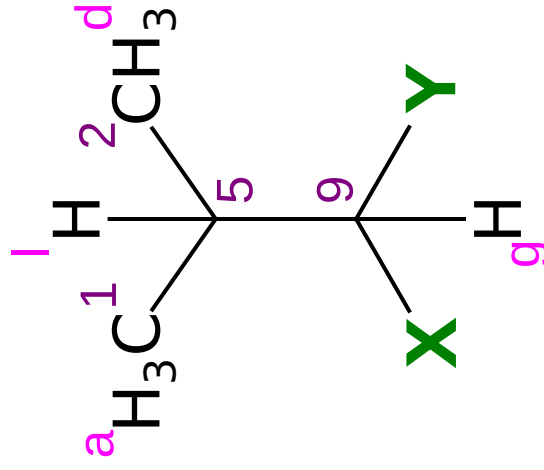
Task 1:

**Unknown substance  $C_{10}H_{20}O$**



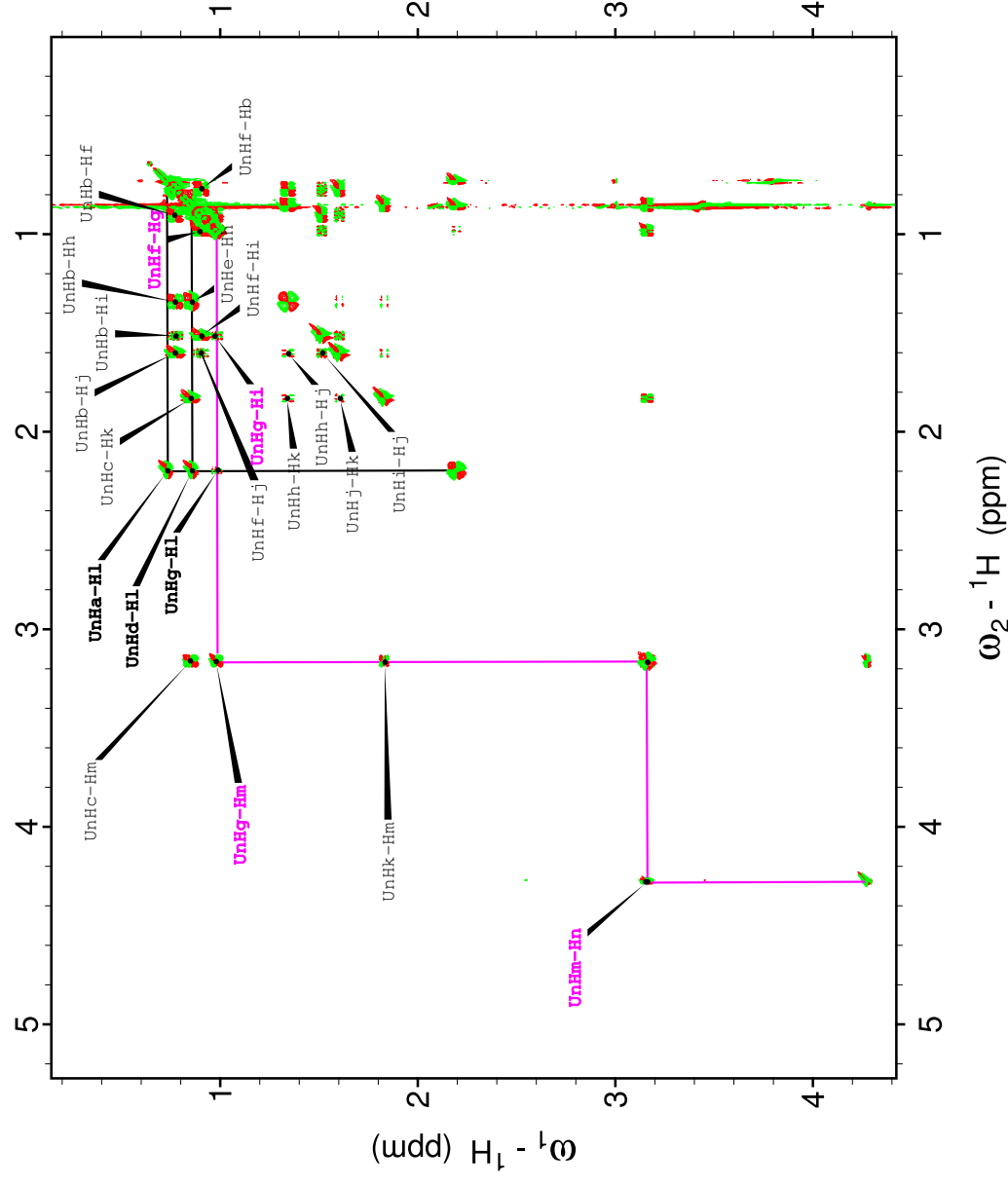
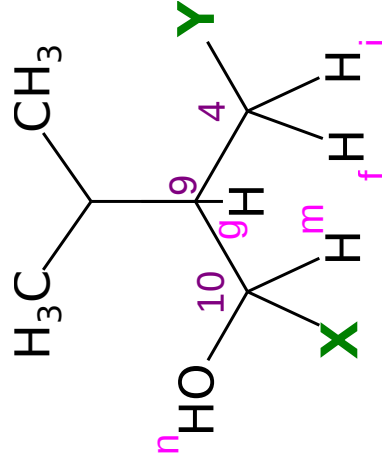
# Task 1: *J*-connectivity of C<sub>10</sub>H<sub>20</sub>O

- methyls 1a,2d connected to CH 5l
- remaining crosspeak of CH 5l to CH 9g
- methyls 1a,2d diastereotopic  $\Rightarrow$  chiral carbon 9



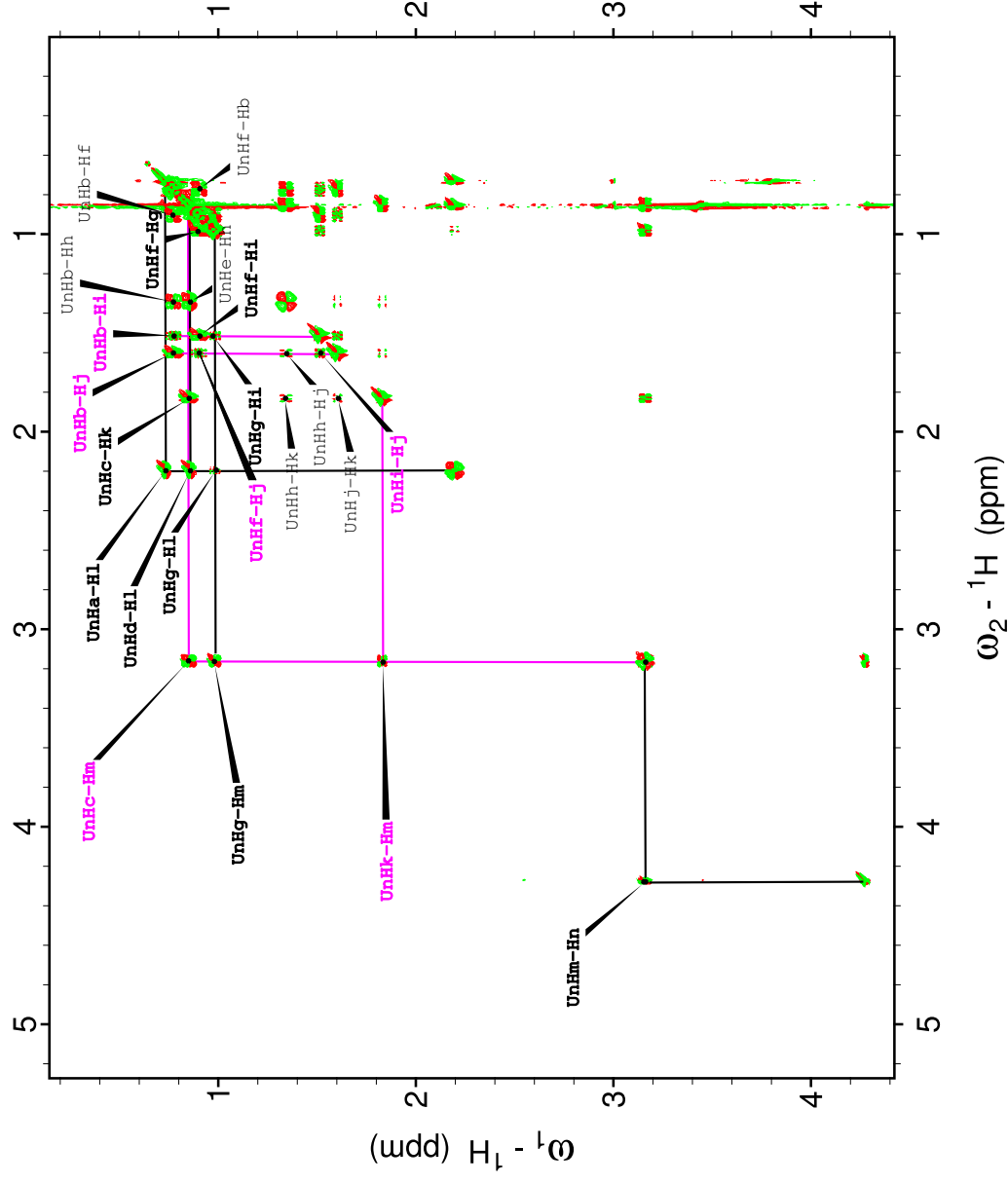
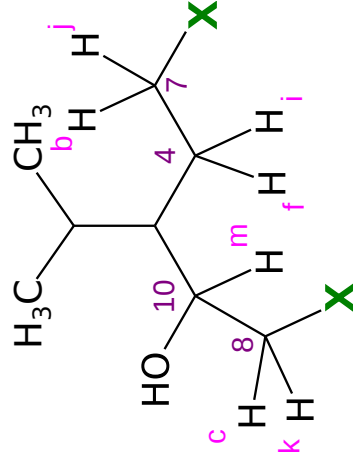
# Task 1: *J*-connectivity of C<sub>10</sub>H<sub>20</sub>O

- CH **9g** has crosspeaks with deshielded **10m** ⇒ OH group (**n**)
- CH **9g** has two crosspeaks with diastereotopic protons **4if**



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

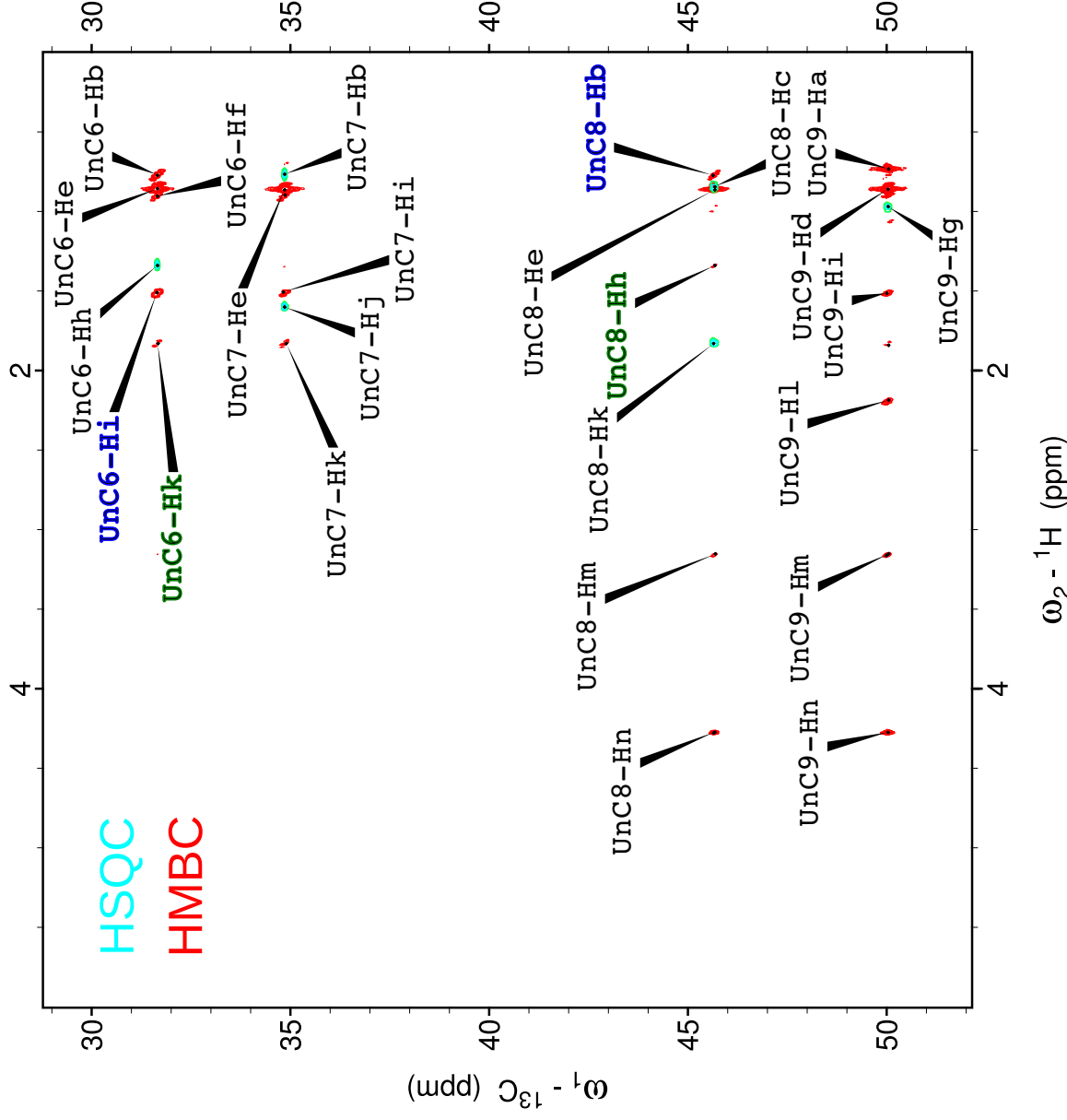
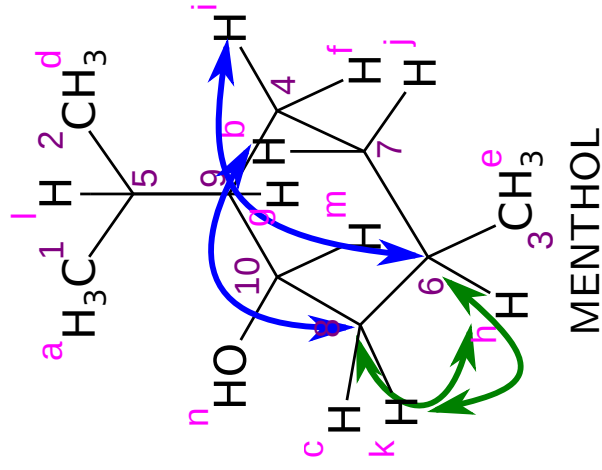
- CH 10m connected with  
CH<sub>2</sub> 8ck
- CH<sub>2</sub> 4if connected with  
CH<sub>2</sub> 7bj





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

- $CH_2$  **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 ( $^{3/4}J_{HC}$ )

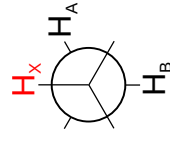




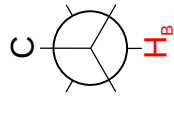
# Task 1:

## Stereochemistry of menthol $C_{10}H_{20}O$

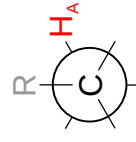
- $1 \leftrightarrow 2$ : homonuclear/heteronuclear couplings
  - large couplings preserved in 1D slices of HSQC: axial H - 2 visible interactions (geminal and vicinal)  $\times$  equatorial H - only geminal
  - 1D TOCSY: selective decoupling  $\Rightarrow$  simplification of complex multiplets
  - DQF-COSY: analysis of phase sensitive spectrum
- $1 \leftrightarrow 3$ : NOE contacts (axial strong)



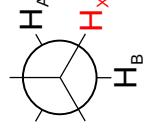
$${}^3J_{X,A} = \text{small}$$
$${}^3J_{X,B} = \text{large}$$



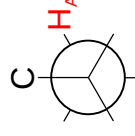
$${}^3J_{H,C} = 6-8 \text{ Hz}$$



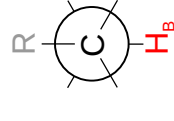
$${}^2J_{H,C} = 5-7 \text{ Hz}$$



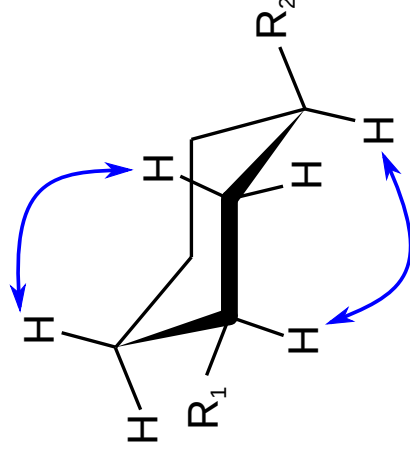
$${}^3J_{X,A} = \text{small}$$
$${}^3J_{X,B} = \text{small}$$



$${}^3J_{H,C} = 1-3 \text{ Hz}$$



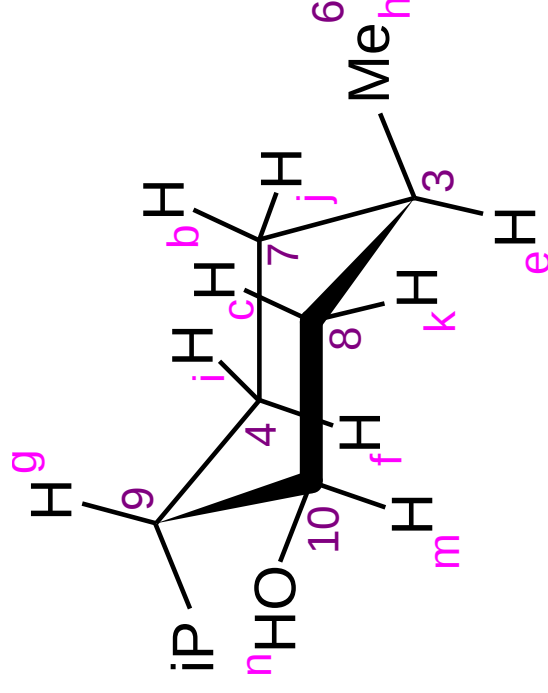
$${}^2J_{H,C} = 0-2 \text{ Hz}$$



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## Stereochemistry of menthol $C_{10}H_{20}O$

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  - 1D TOCSY: selective decoupling  $\Rightarrow$  simplification of complex multiplets
  - DQF-COSY: analysis of phase sensitive spectrum
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Task 1:

**Unknown substance  $C_{21}H_{26}O_7$**