C8953
NMR structural analysis seminar

Elucidenting the 3D structure

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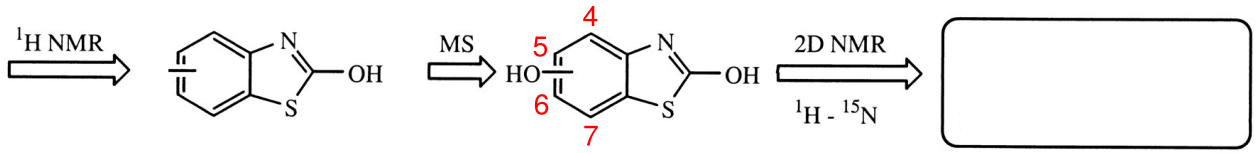
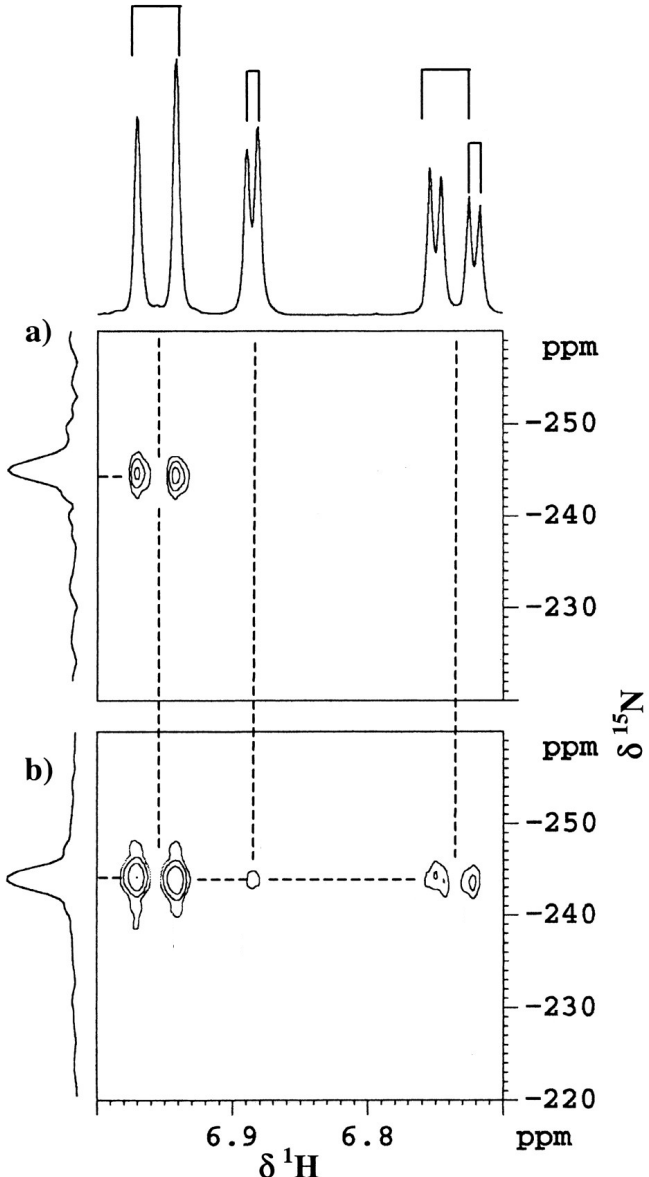
Isomerisms and NMR

- Functional groups (constitution) - chemical shift
- Position of substituents - HMBC, NOESY/ROESY
- Relative configuration on double bonds or rings - *J*-coupling, NOESY/ROESY
- Absolute configuration - application of Chiral Derivatizing Agents (CDA)

Benzothiazole Biodegradation: ^{1}H-^{15}N HMBC (*Appl. Environ. Microbiol.*, 2001, **67**)





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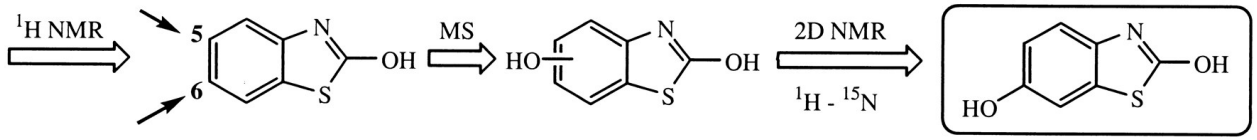
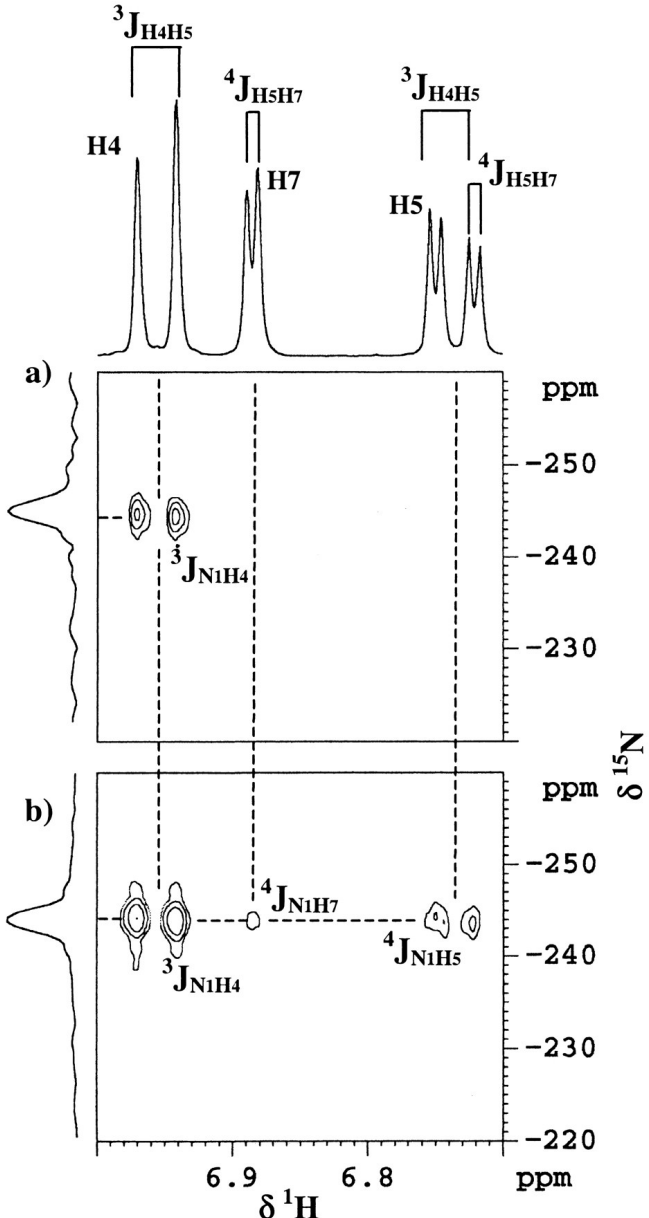
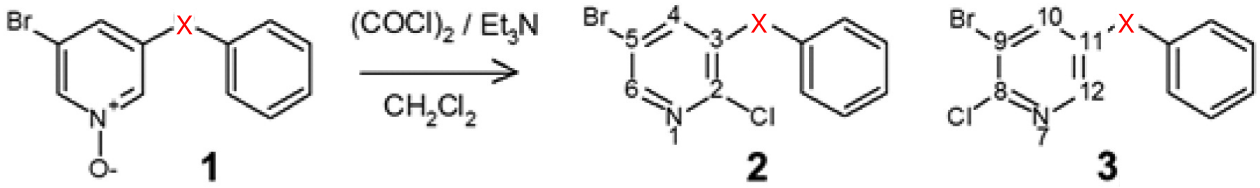
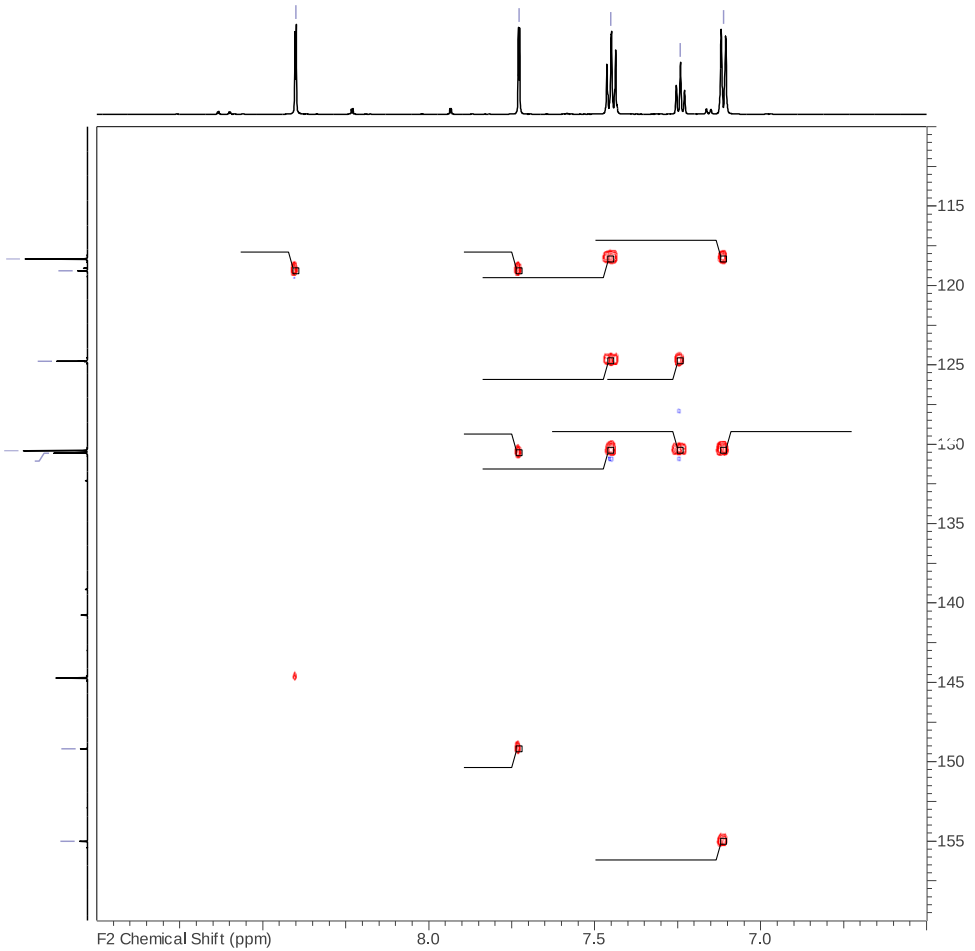




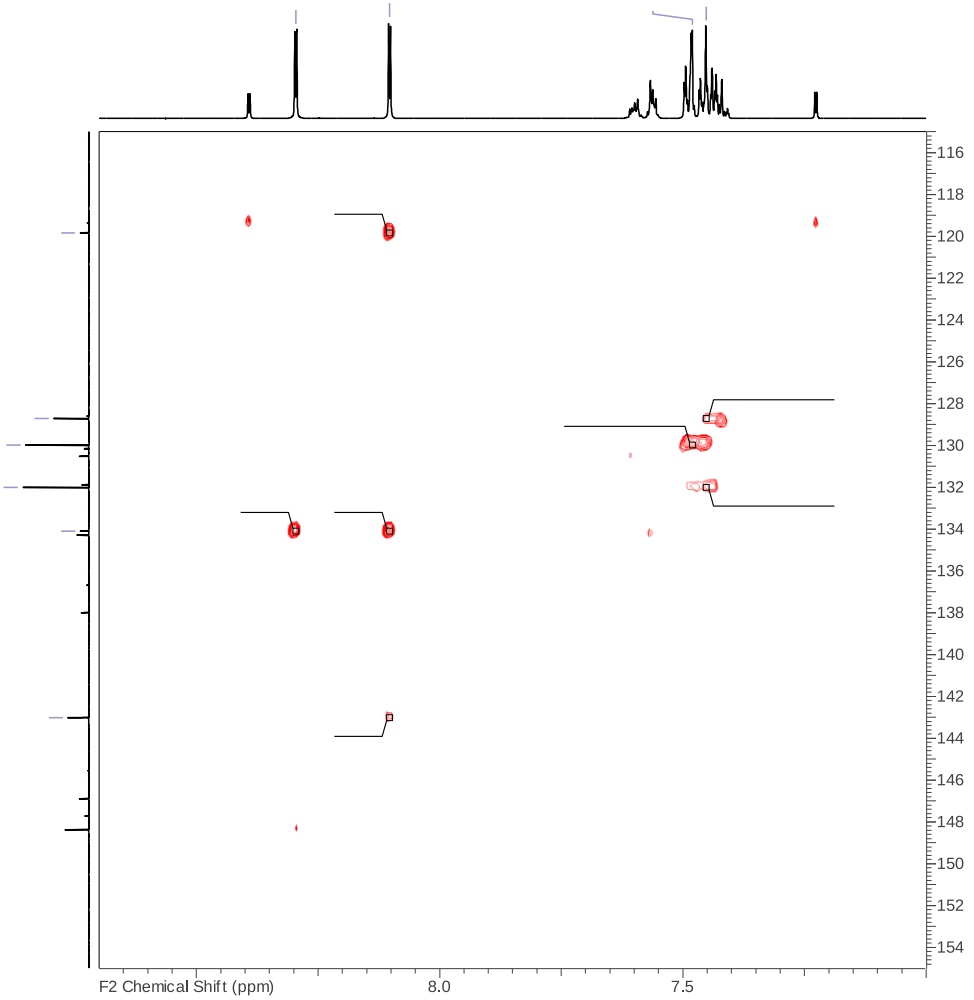
Regioselectivity in the Halogenation: 1,1-ADEQUATE (*Org. Lett.*, **2016**, 18, 19561959)



X = O



X = S



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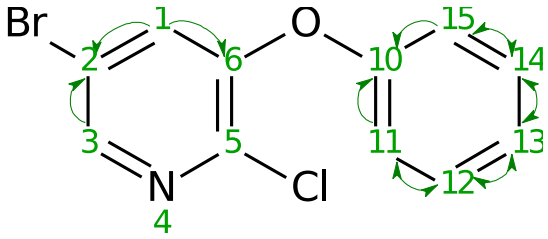
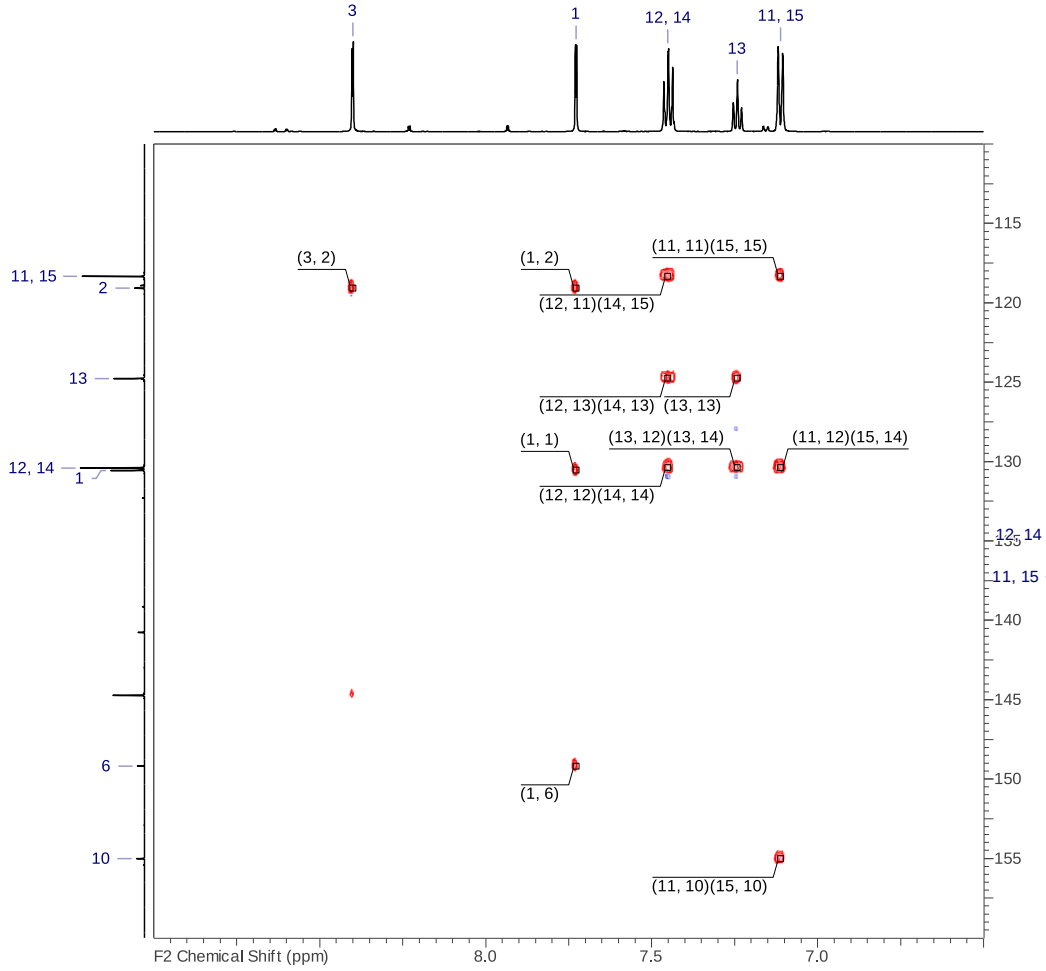
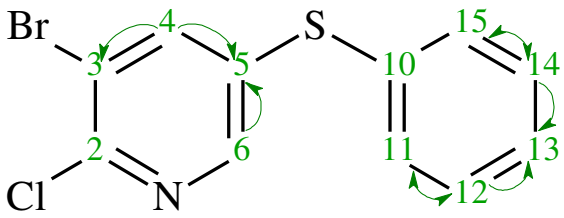
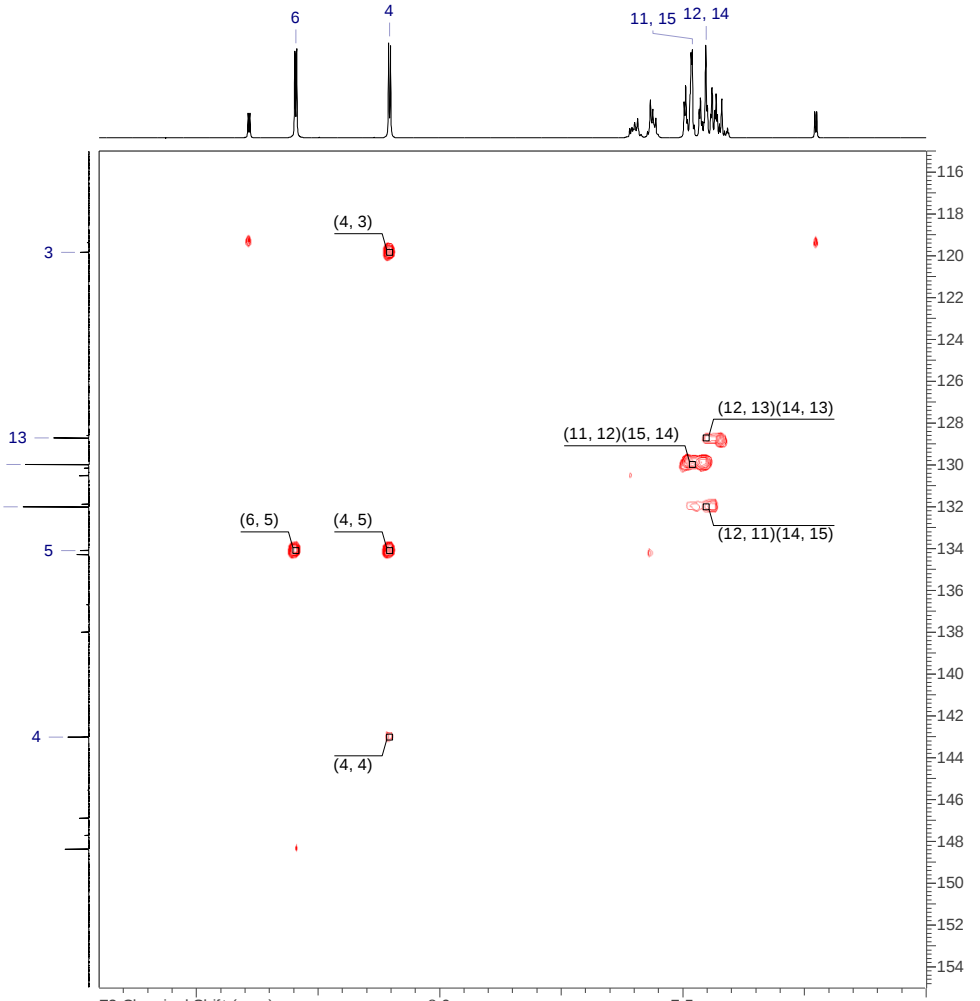


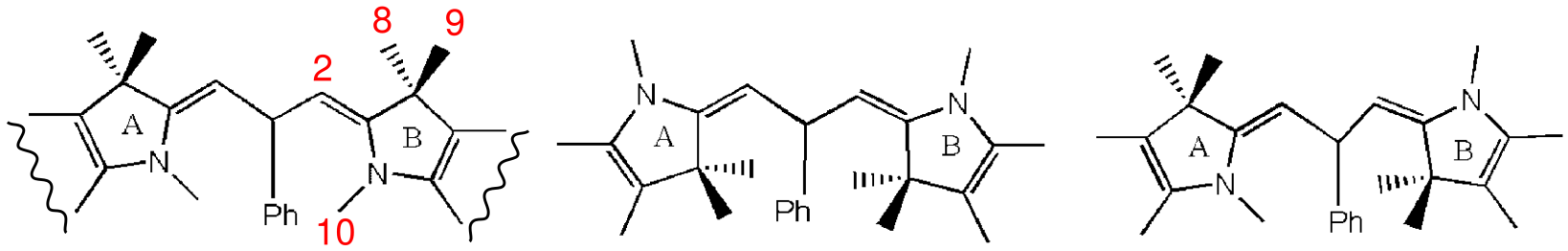






Configuration on double bonds (*Magn. Reson. Chem.* **2008**, 46, 872–877)

Describe the isomers of molecule shown bellow:



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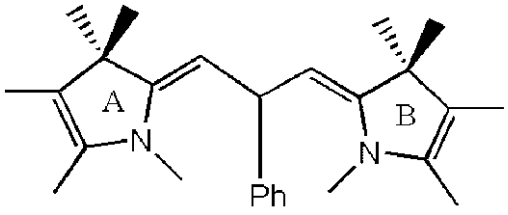
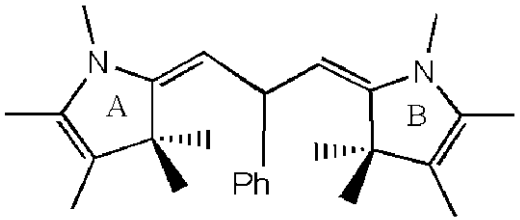
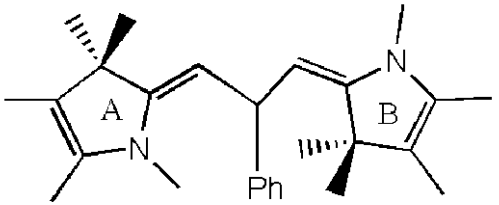


(Z,Z)

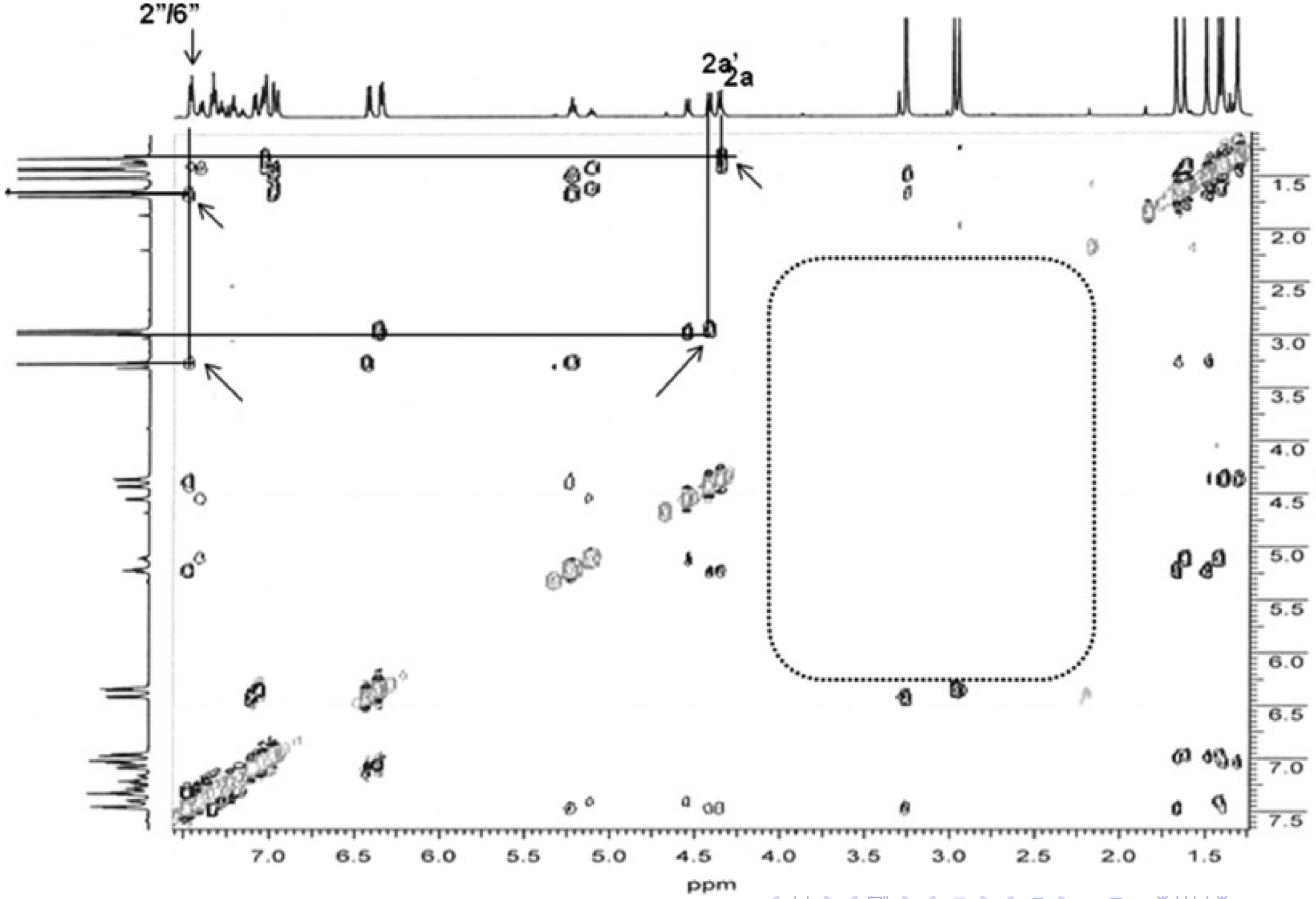


(E,E)

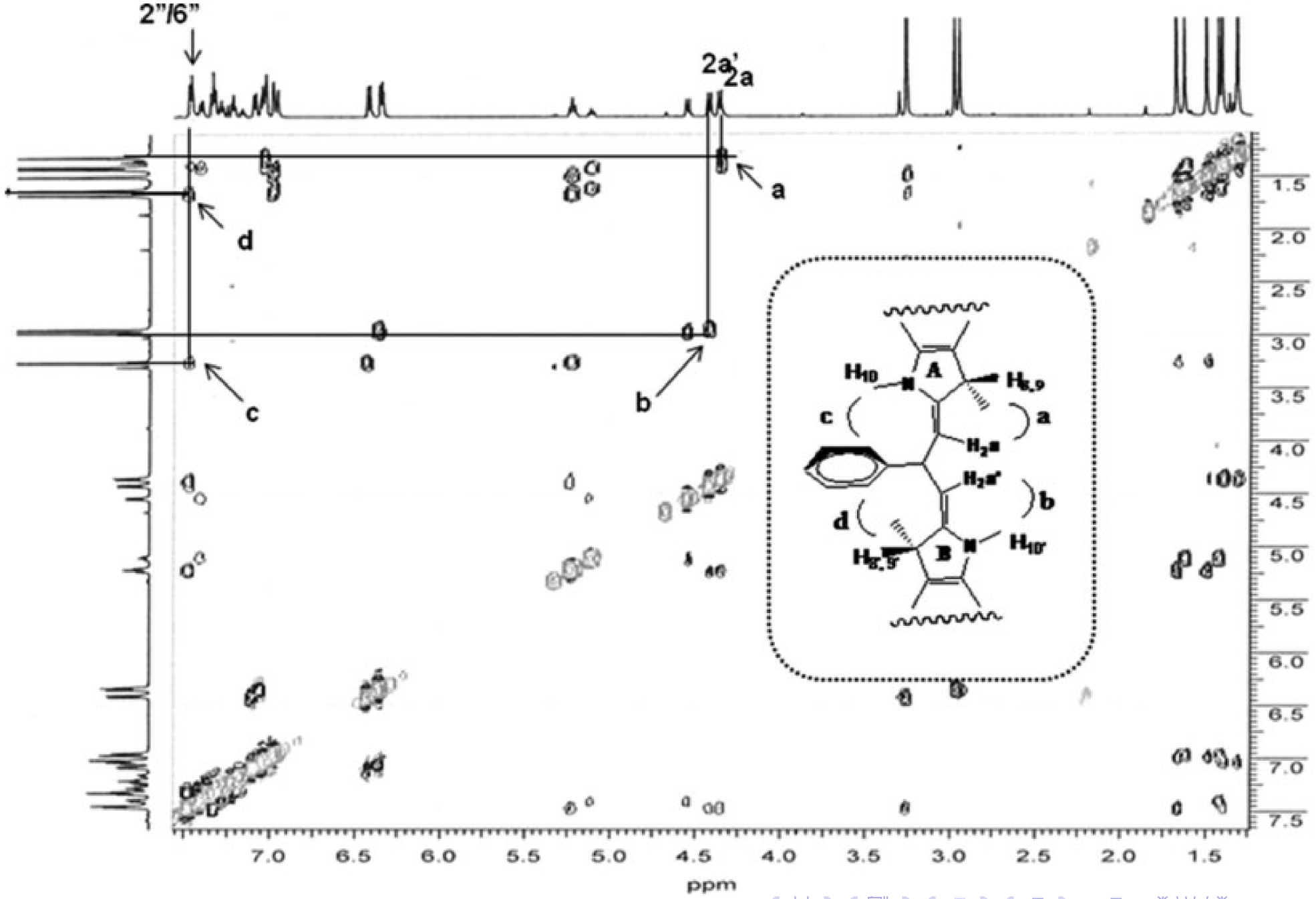


(Z,E)

Configuration on double bonds: NOESY

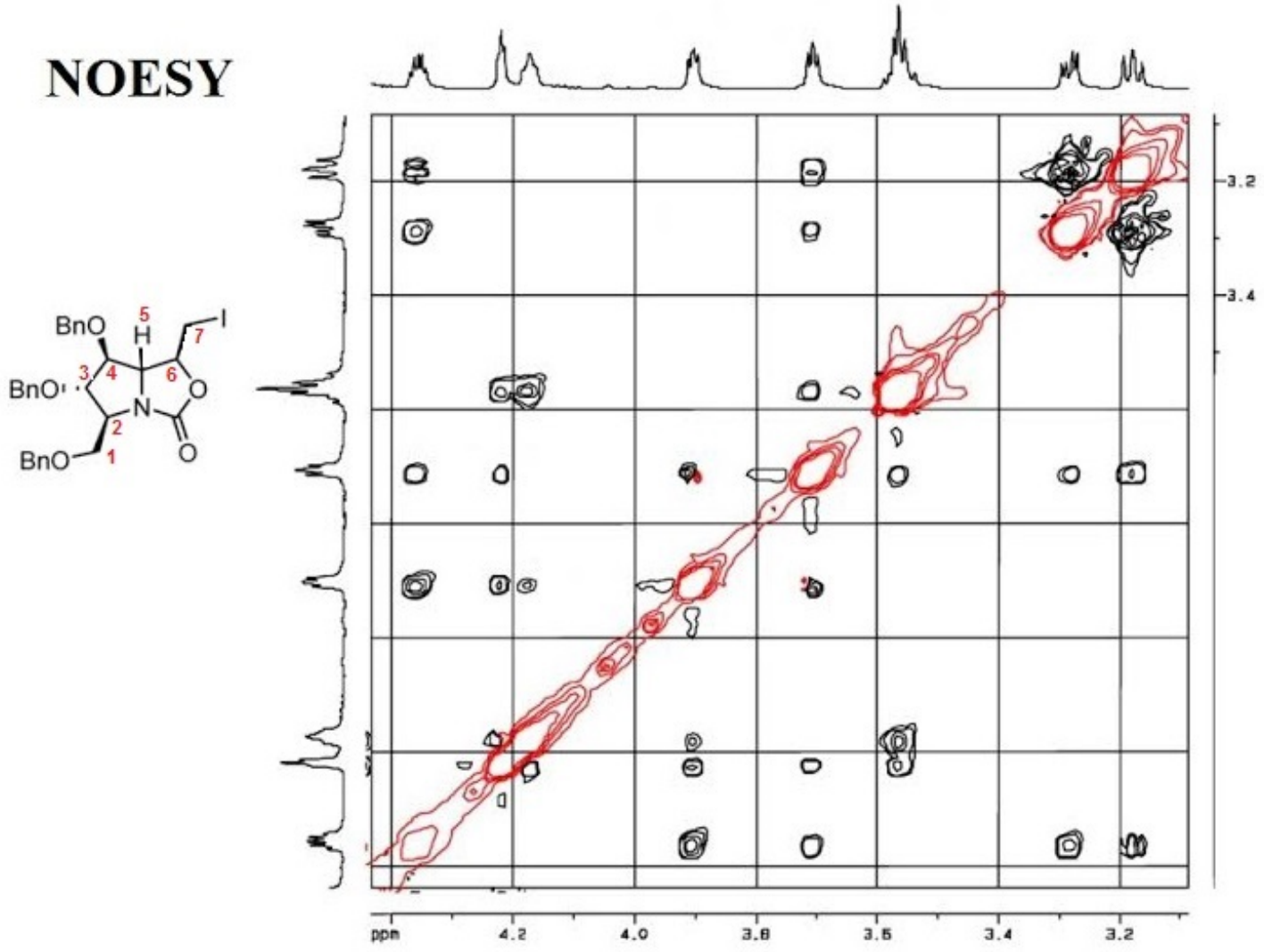


Configuration on double bonds: NOESY



Relative stereochemistry on a ring: NOESY

Provide the complete assignment of ^{1}H resonances and determine the orientation of H5 and H6.



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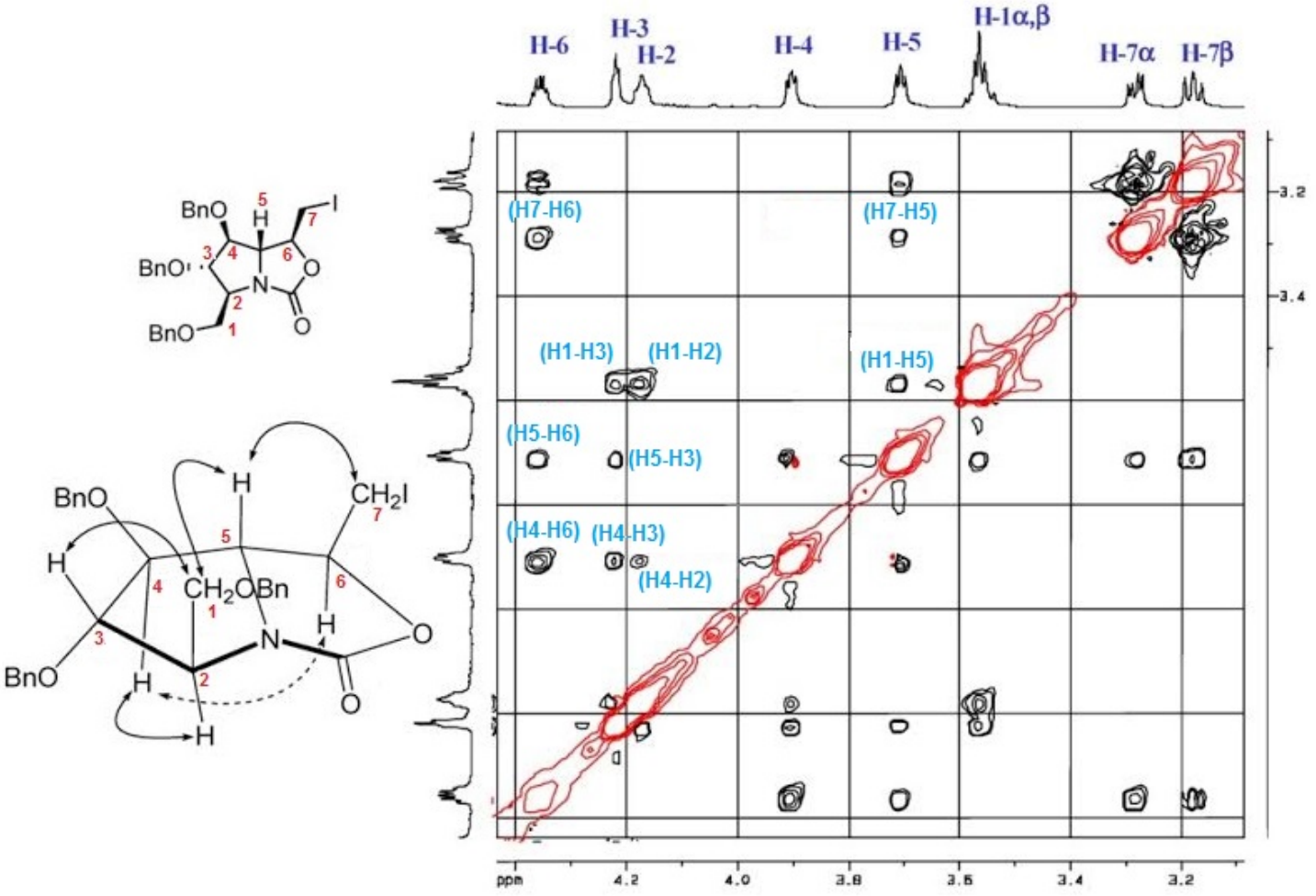
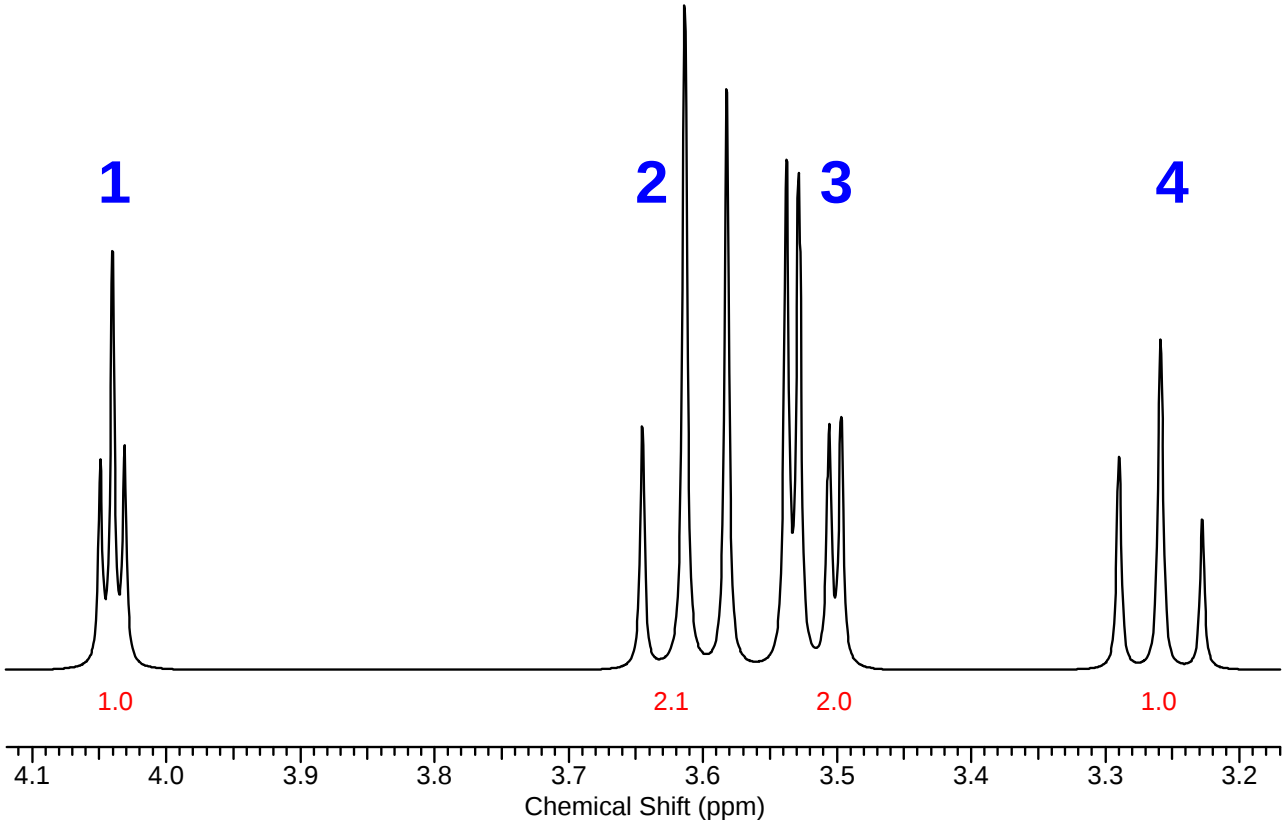


Interpretation of *J*-coupling

Unknown compound $C_6H_{12}O_6$ measured in D_2O
Detected J_{HH}-couplings: (2x9.6), (2.8, 9.6), (2x9.6), (2x2.8)



1D ^{13}C NMR spectrum contains **4 signals in the range 71-75 ppm**.

Interpretation of *J*-coupling

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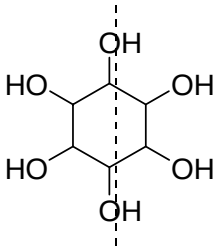
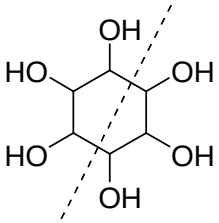
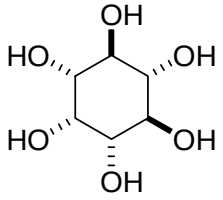
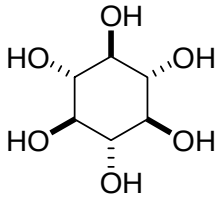






myo



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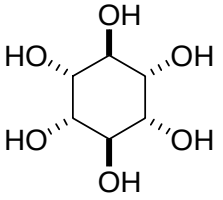
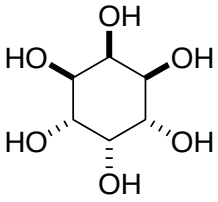
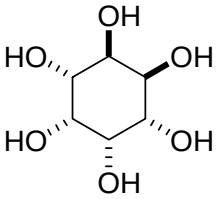


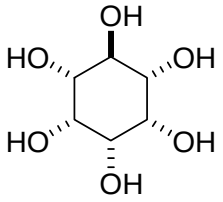
muco



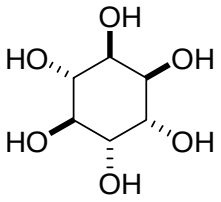
neo



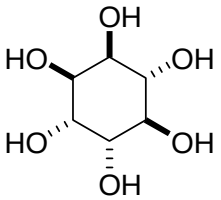
allo



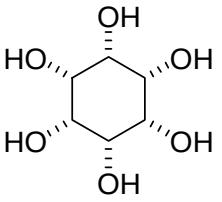
epi



D-chira



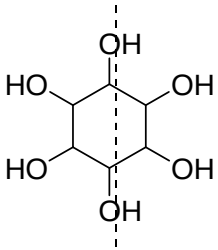
L-chira

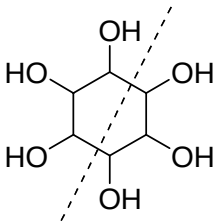


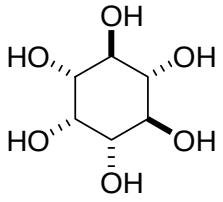
cis

Interpretation of *J*-coupling

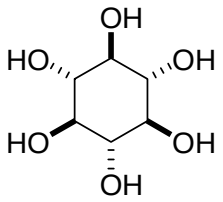
J_{HH}-couplings: (2x9.6), (2.8, 9.6), (2x9.6), (2x2.8)



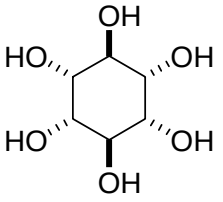




myo



scyllo



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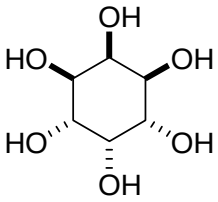

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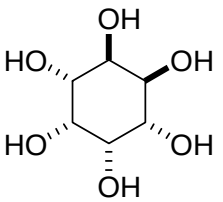
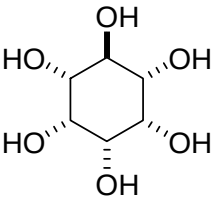
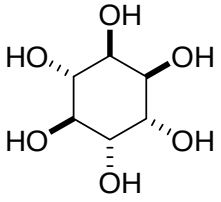


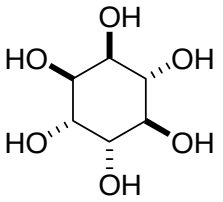
allo



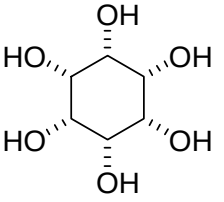
epi



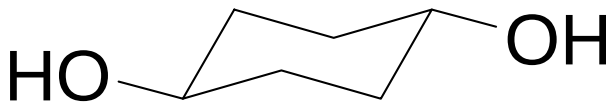
D-chira



L-chira

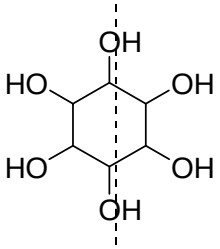


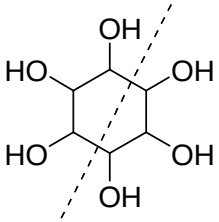
cis

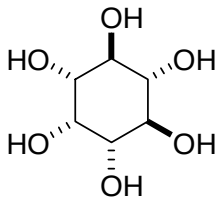


Interpretation of *J*-coupling

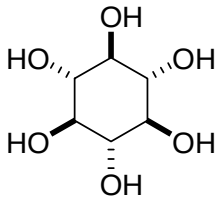
J_{HH}-couplings: (2x9.6), (2.8, 9.6), (2x9.6), (2x2.8)



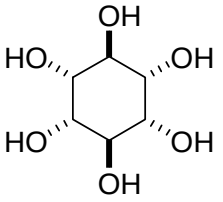




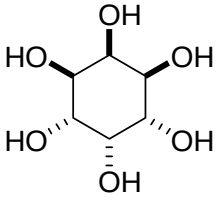
myo



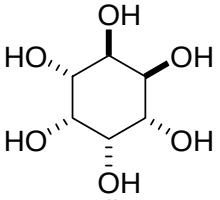
scyllo



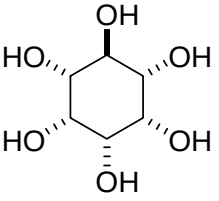
muco



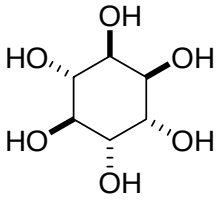
neo



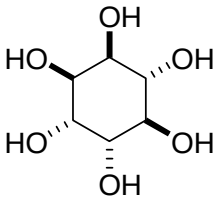
allo



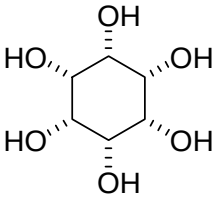
epi



D-chira

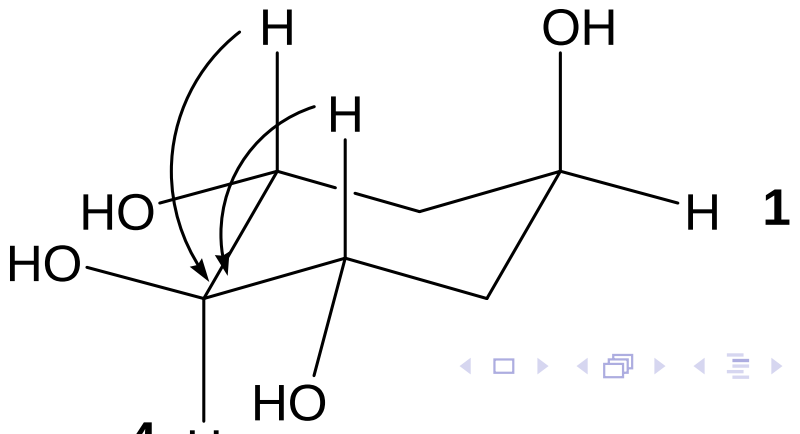


L-chira



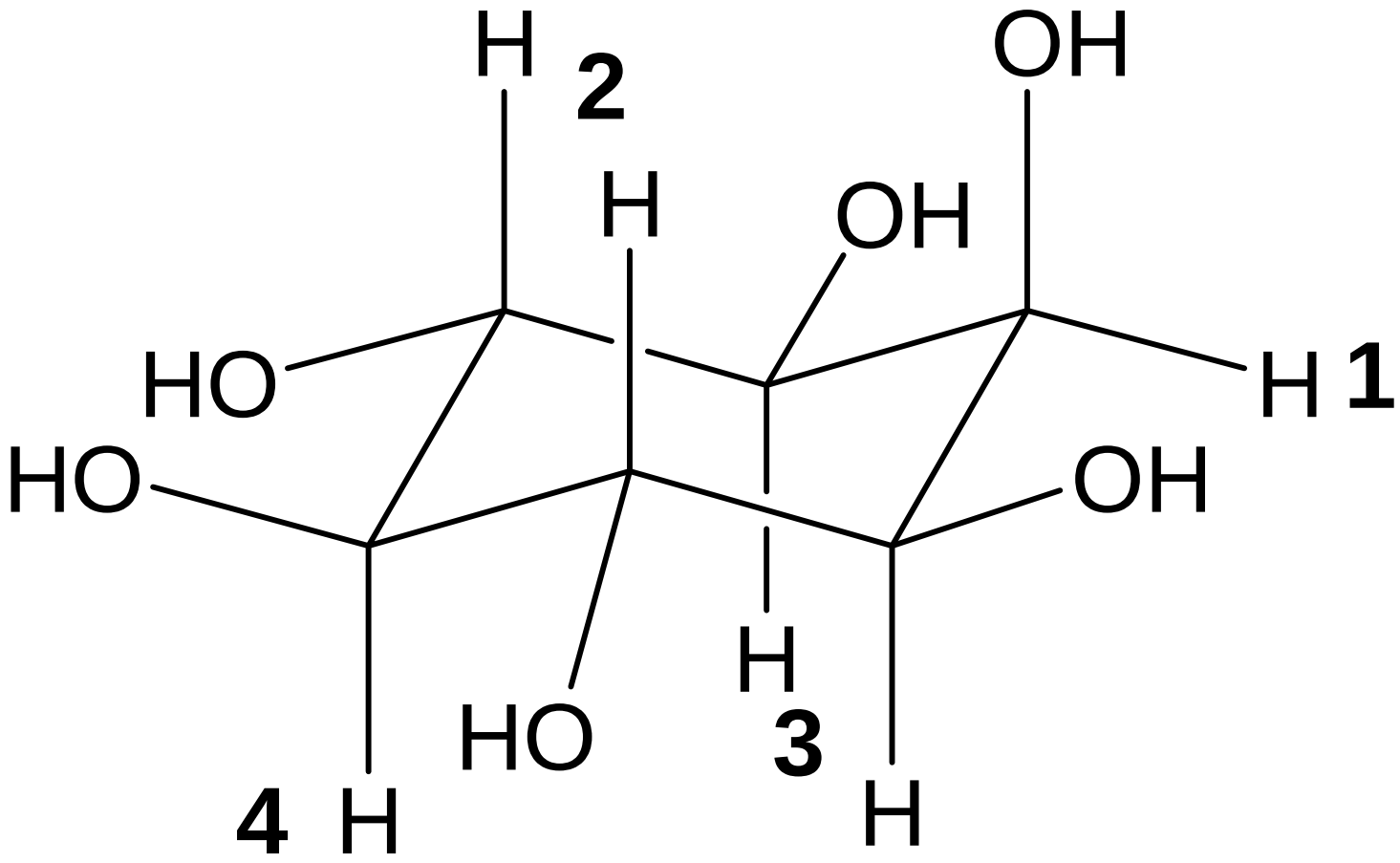
cis

trans couplings are large: about 10 Hz



Interpretation of *J*-coupling: **MYO isomer**

J_{HH}-couplings: (2x9.6), (2.8, 9.6), (2x9.6), (2x2.8)



Next time: Final test

- Basic theory from the lecture (interactions of nuclear spin, vector model, FT, dynamic NMR, relaxation, 2D techniques)
- Assignment of a known compound, determination of the configuration
- Identification of a simple unknown compound