

C8953
NMR structural analysis
seminar

Elucidating the structure using various NMR techniques

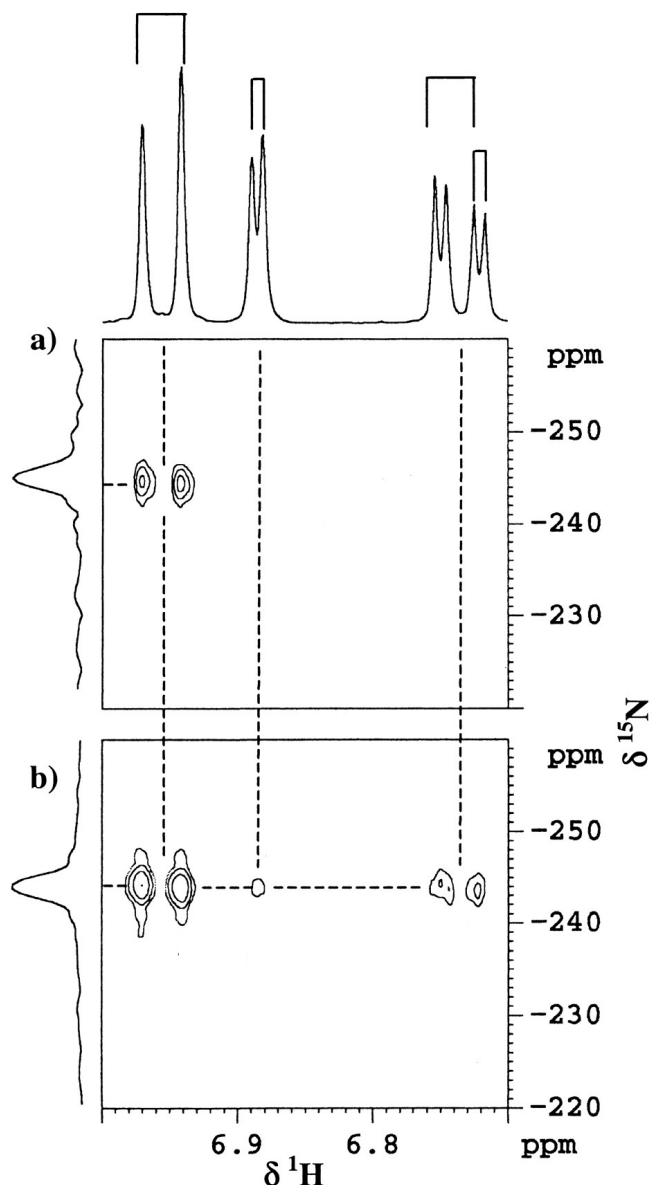
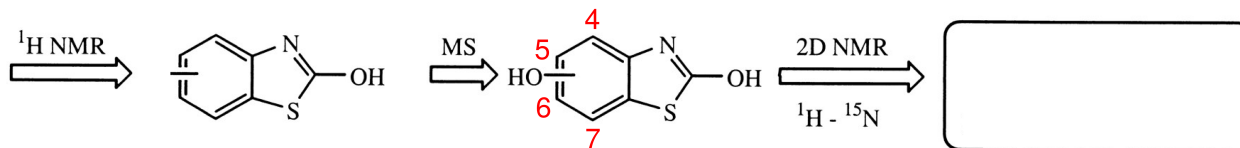
Jan Novotný
176003@mail.muni.cz

May 10, 2017

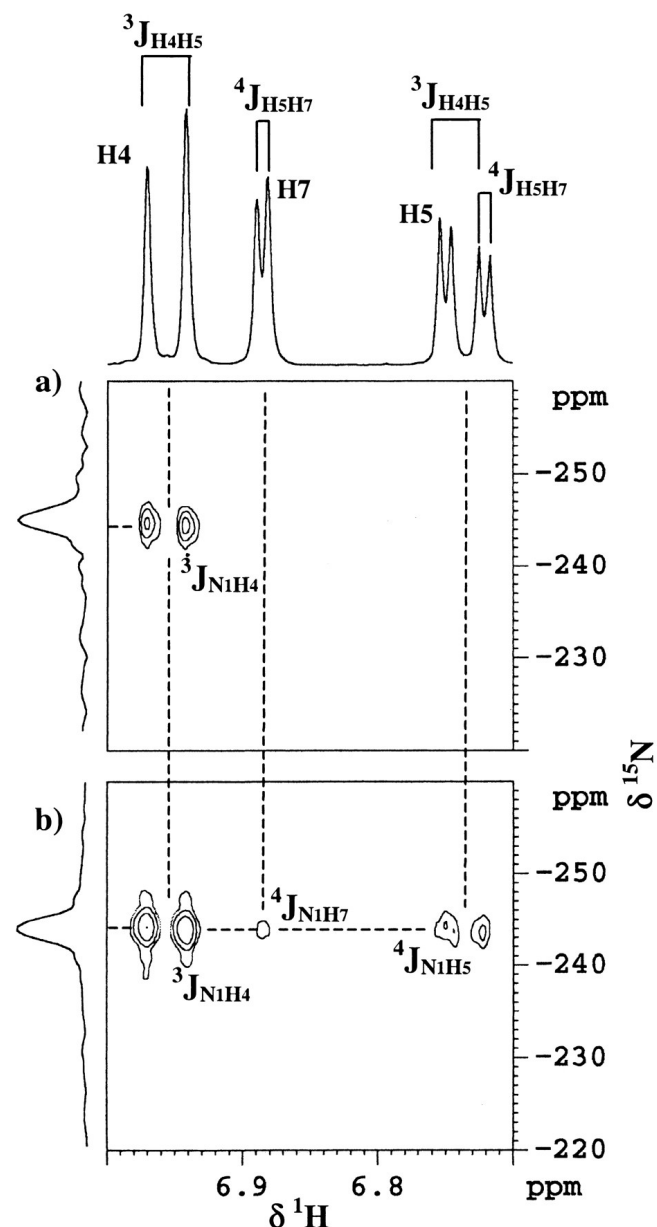
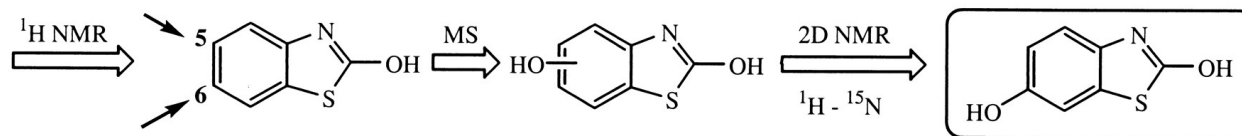
Isomerisms and NMR

- ▶ 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)

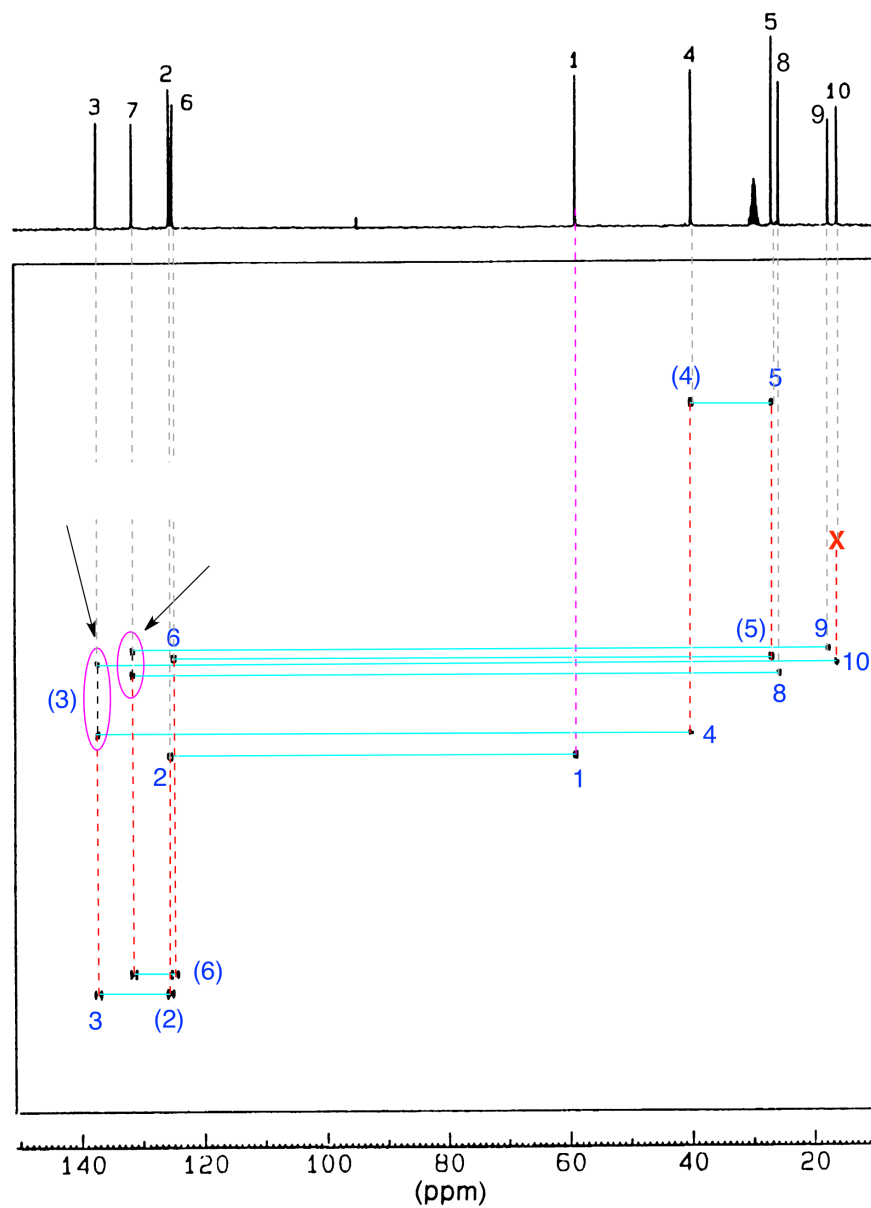
Benzothiazole Biodegradation: ^1H - ^{15}N HMBC (*Appl. Environ. Microbiol.*, 2001, 67)



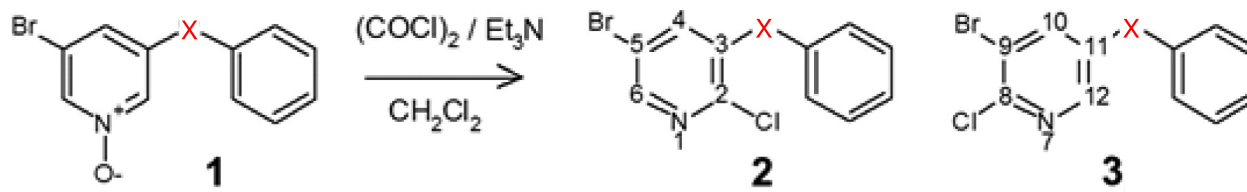
Benzothiazole Biodegradation: ^1H - ^{15}N HMBC (*Appl. Environ. Microbiol.*, 2001, 67)



Determine the structure of $C_{10}H_{18}O$ using INADEQUATE exp.

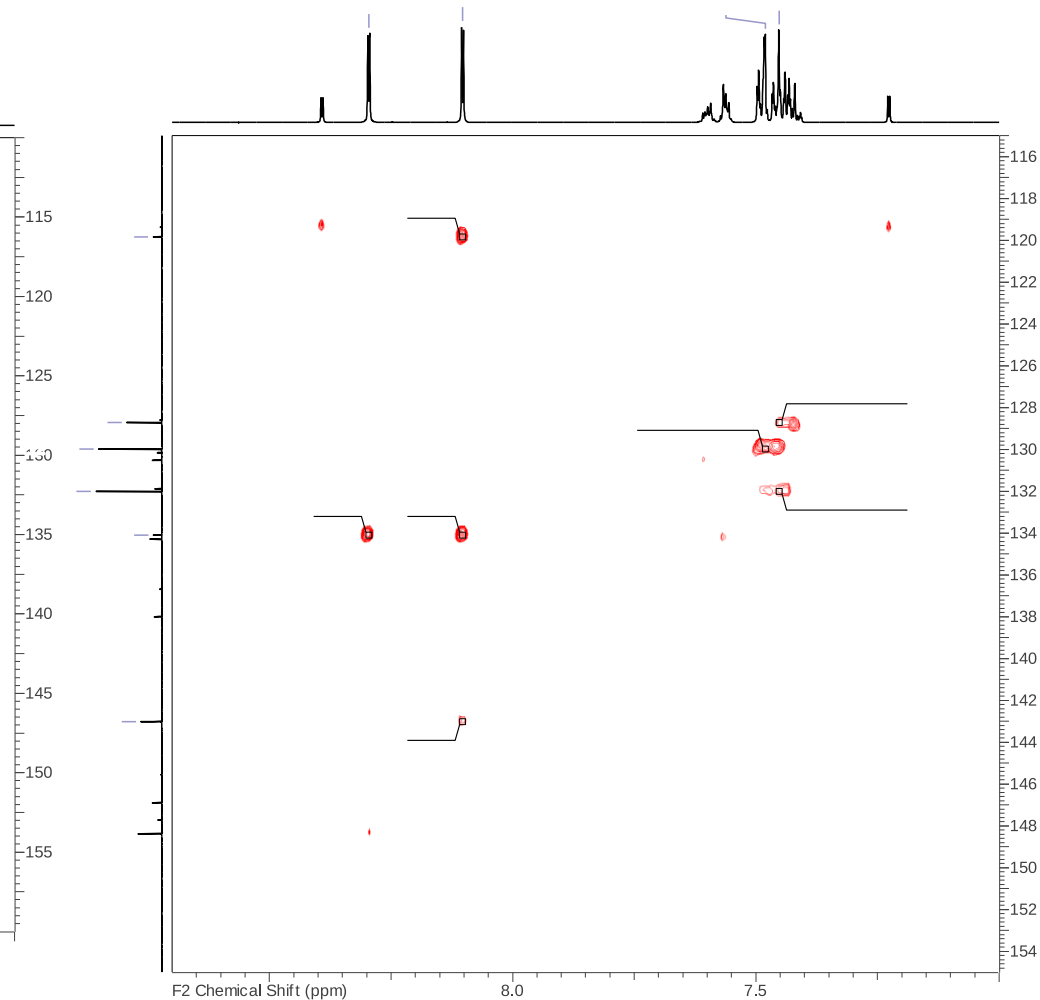
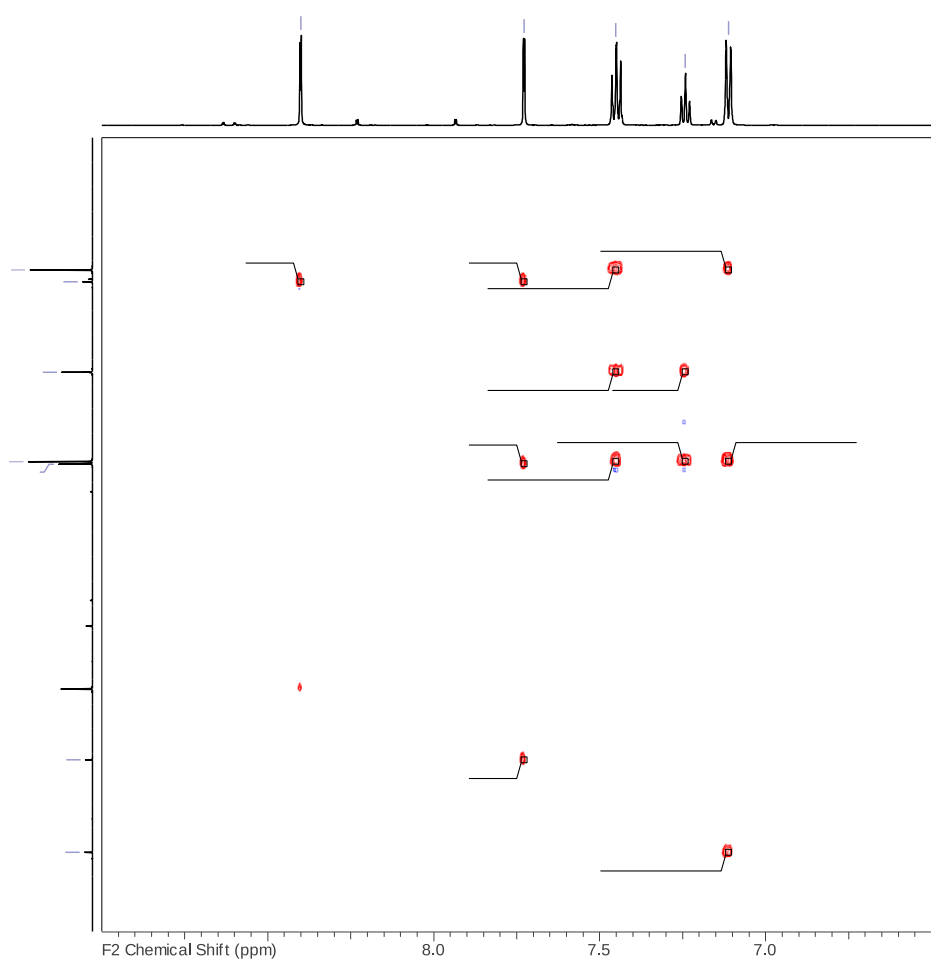


Regioselectivity in the Halogenation: 1,1-ADEQUATE (*Org. Lett.*, **2016**, 18, 1956-1959)

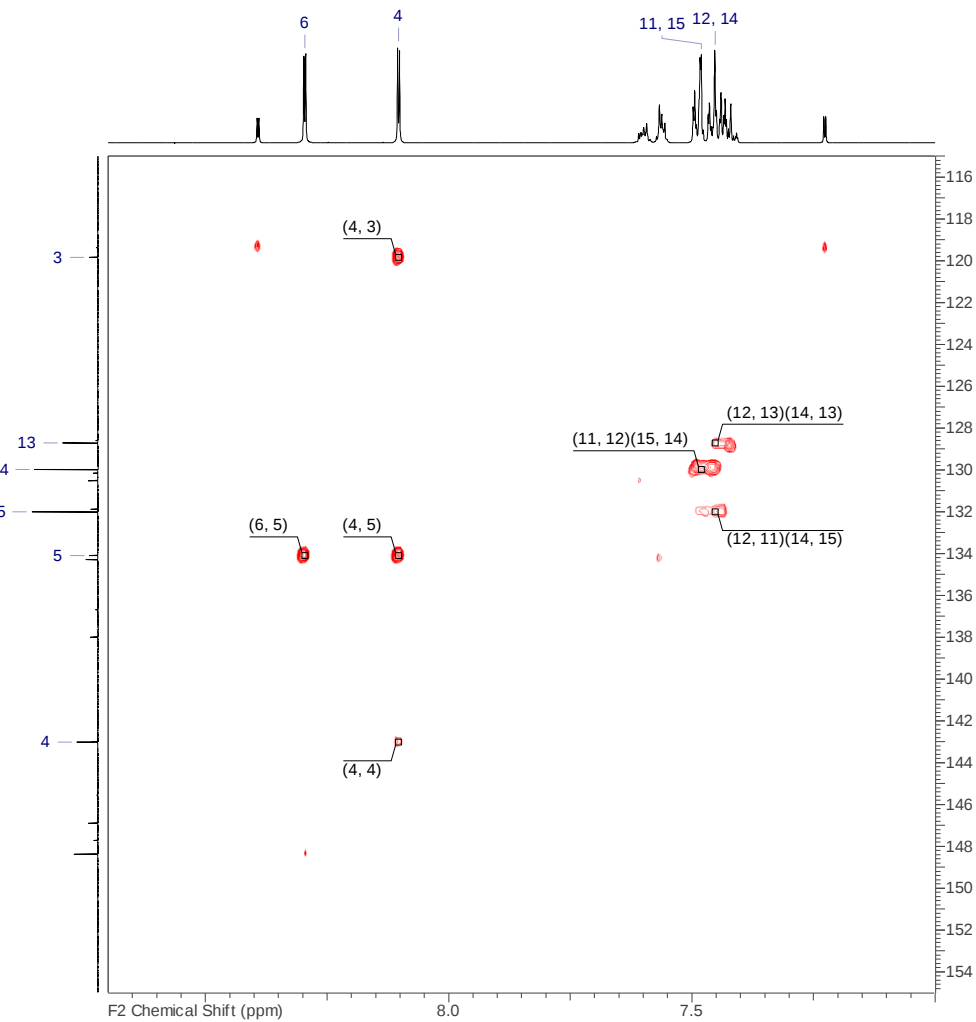
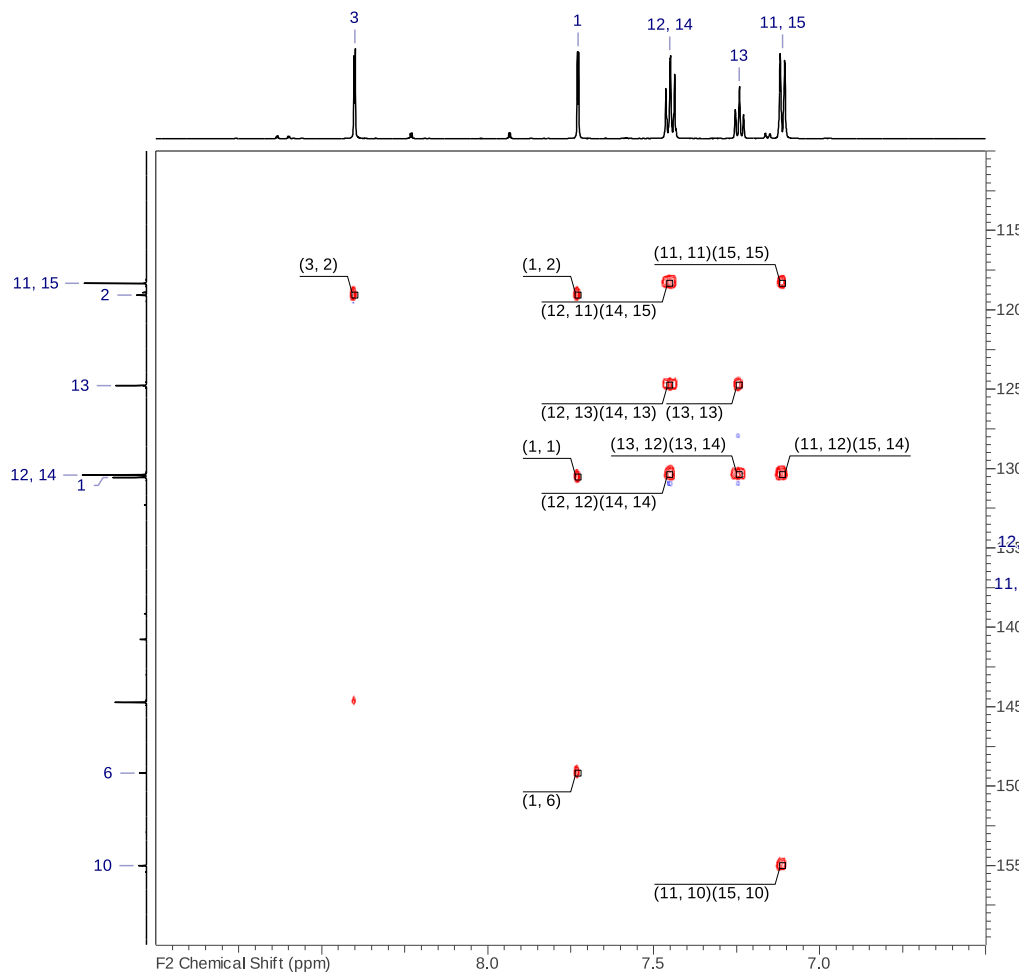
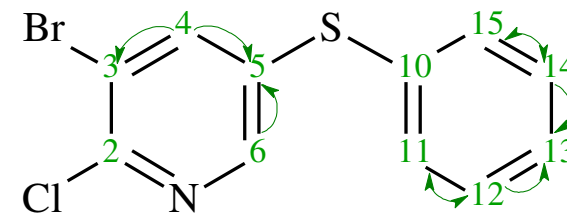
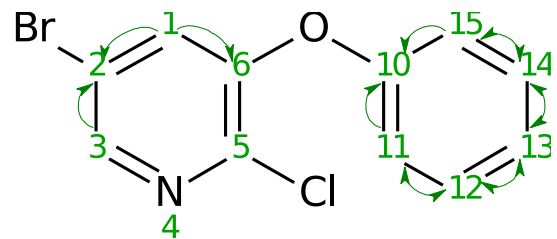


X = O

X = S

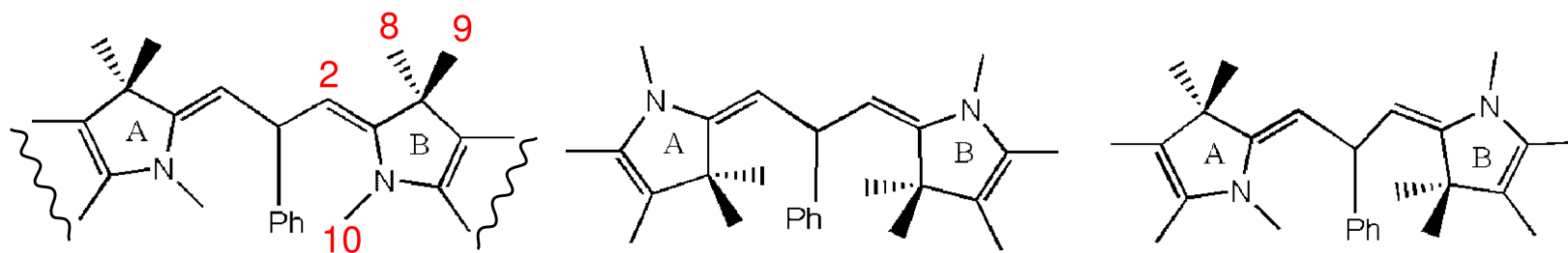


Regioselectivity in the Halogenation: 1,1-ADEQUATE (*Org. Lett.*, 2016, 18, 1956-1959)



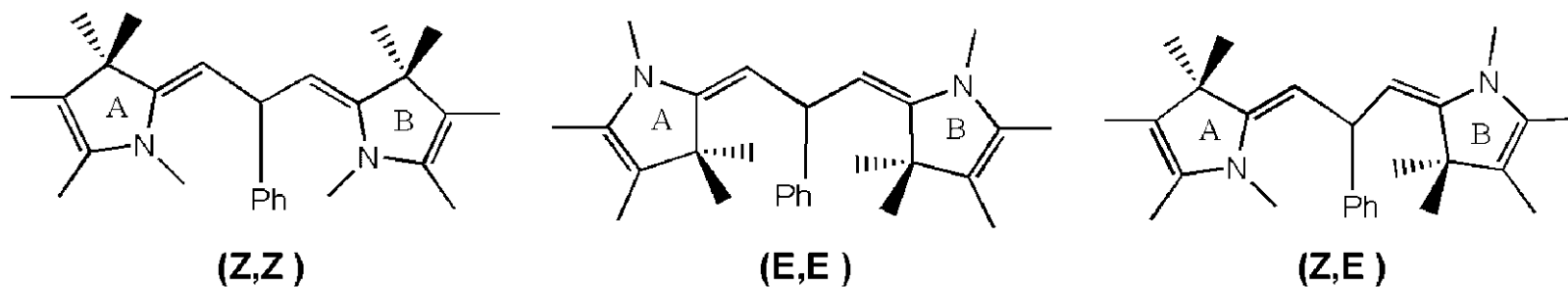
Configuration on double bonds (*Magn. Reson. Chem.* 2008, 46, 872–877)

Describe the isomers of molecule shown bellow:

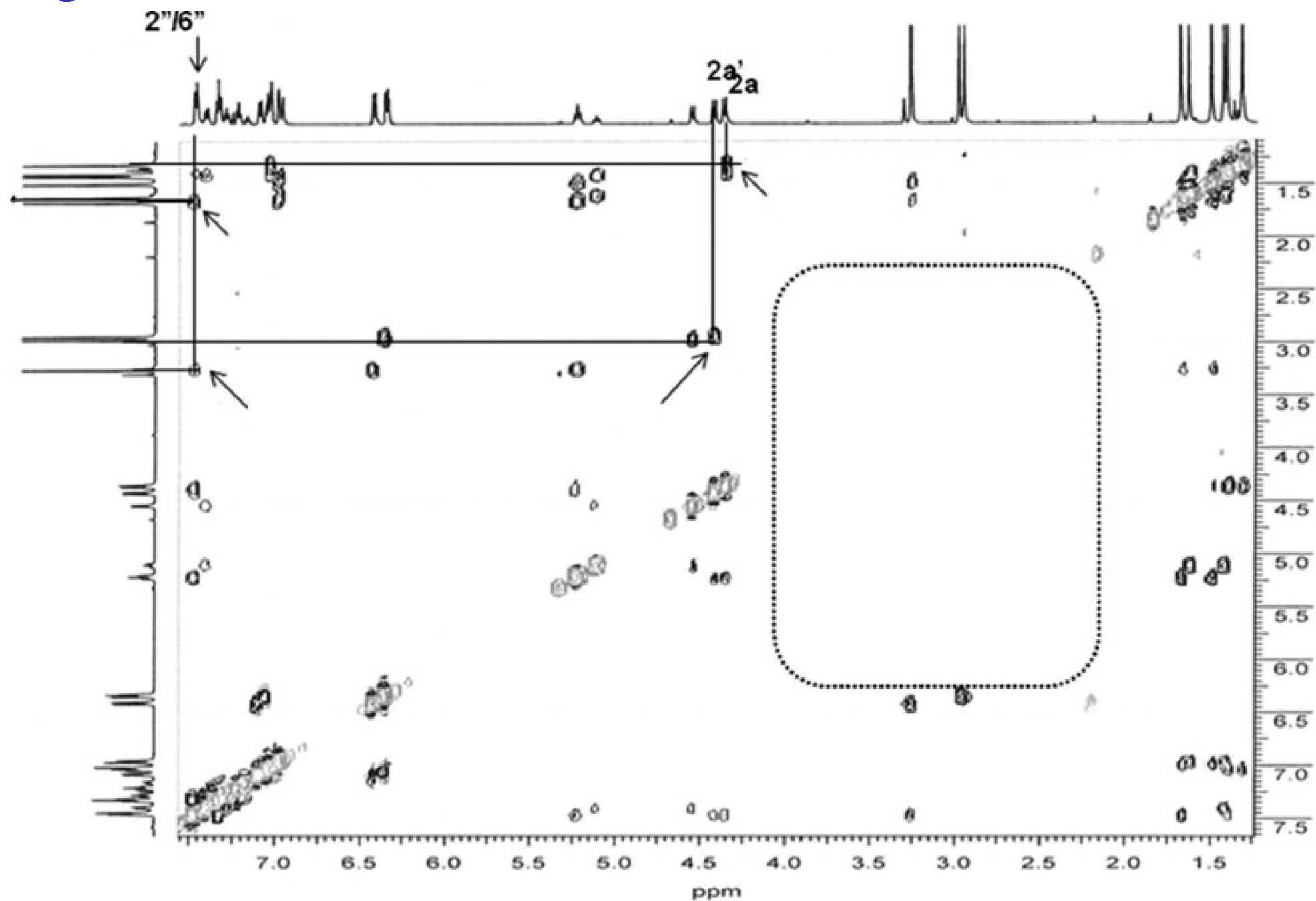


Configuration on double bonds (*Magn. Reson. Chem.* 2008, 46, 872–877)

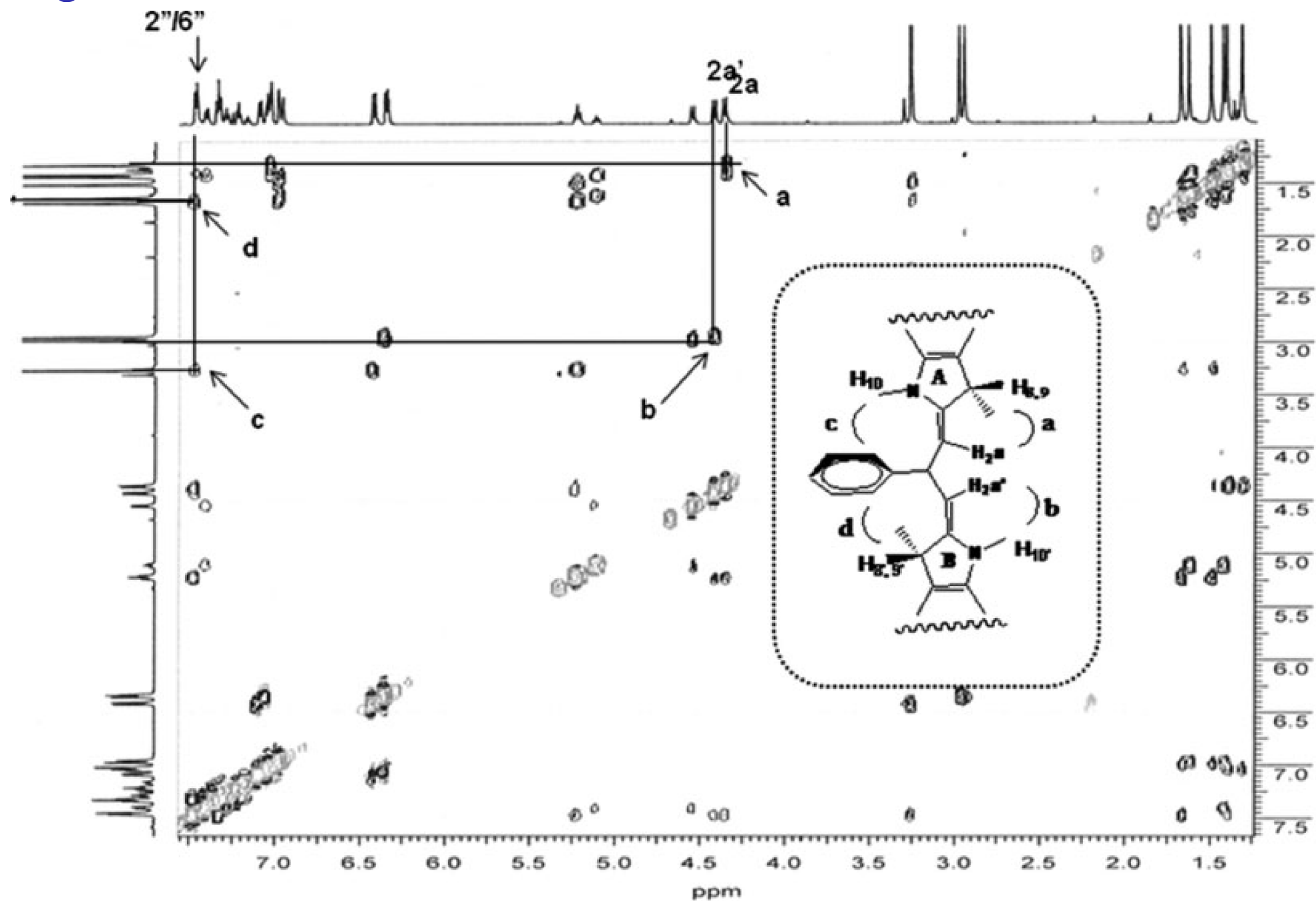
Describe the isomers of molecule shown bellow:



Configuration on double bonds: NOESY

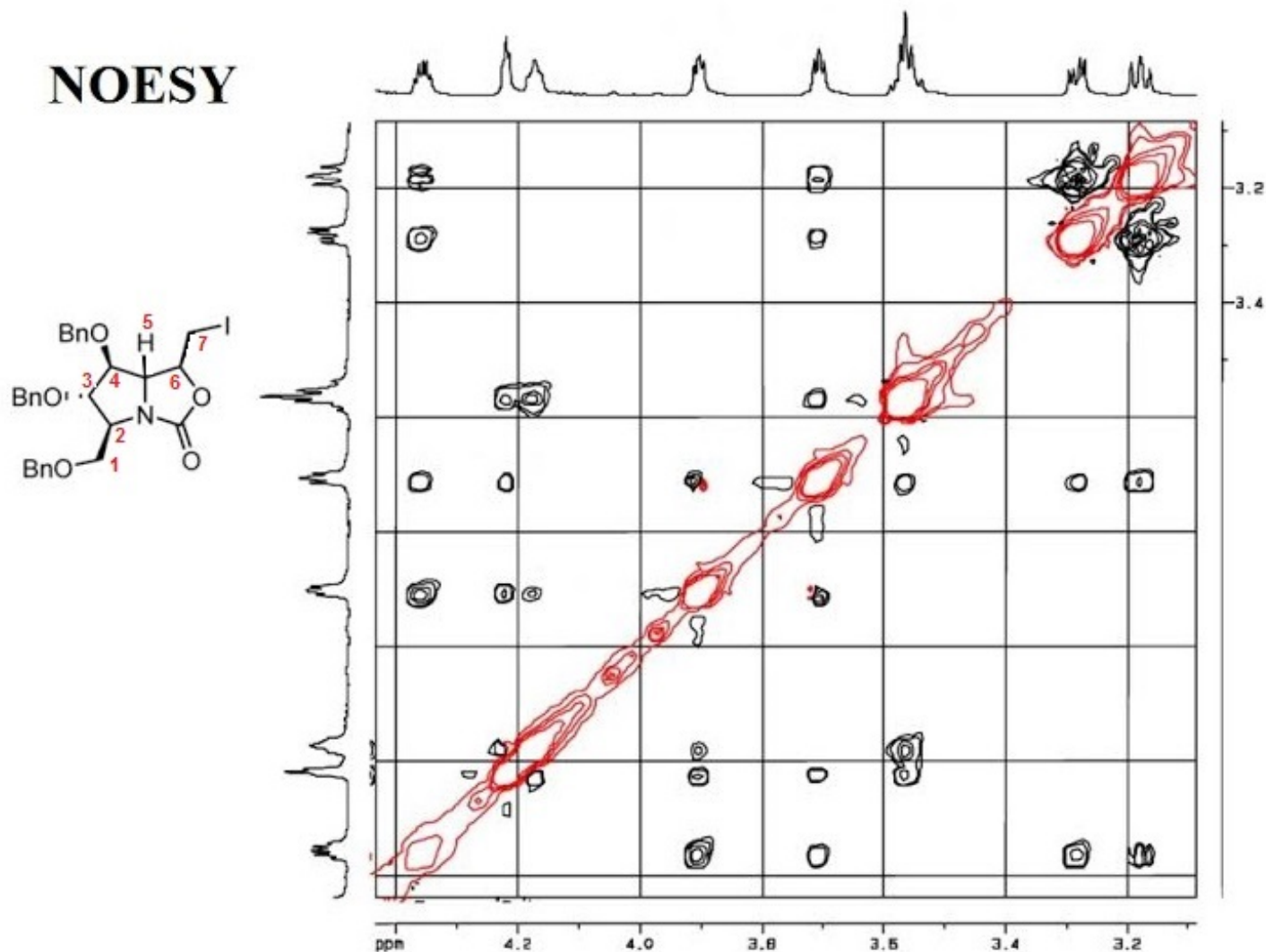


Configuration on double bonds: NOESY

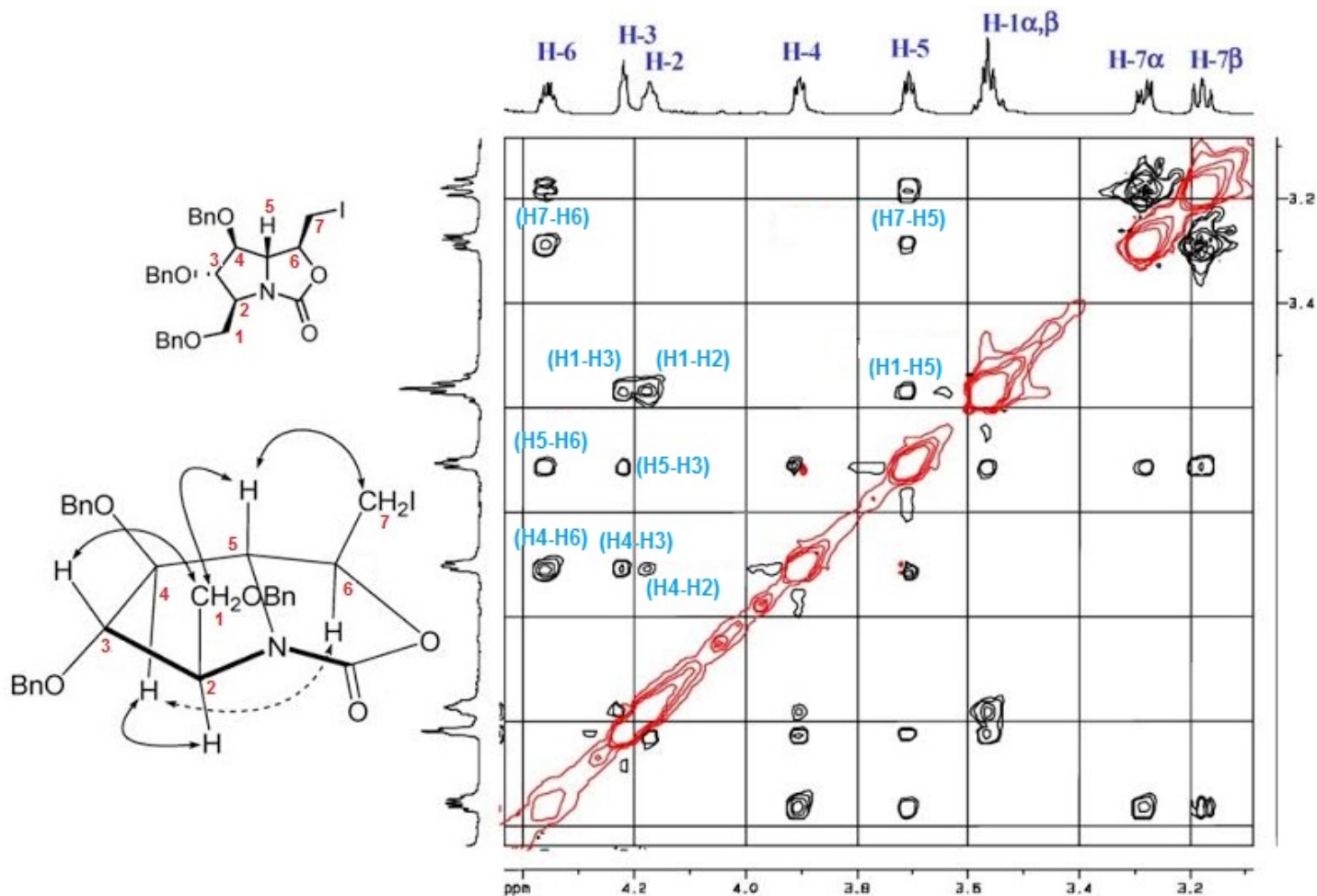


Relative stereochemistry on a ring: NOESY

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

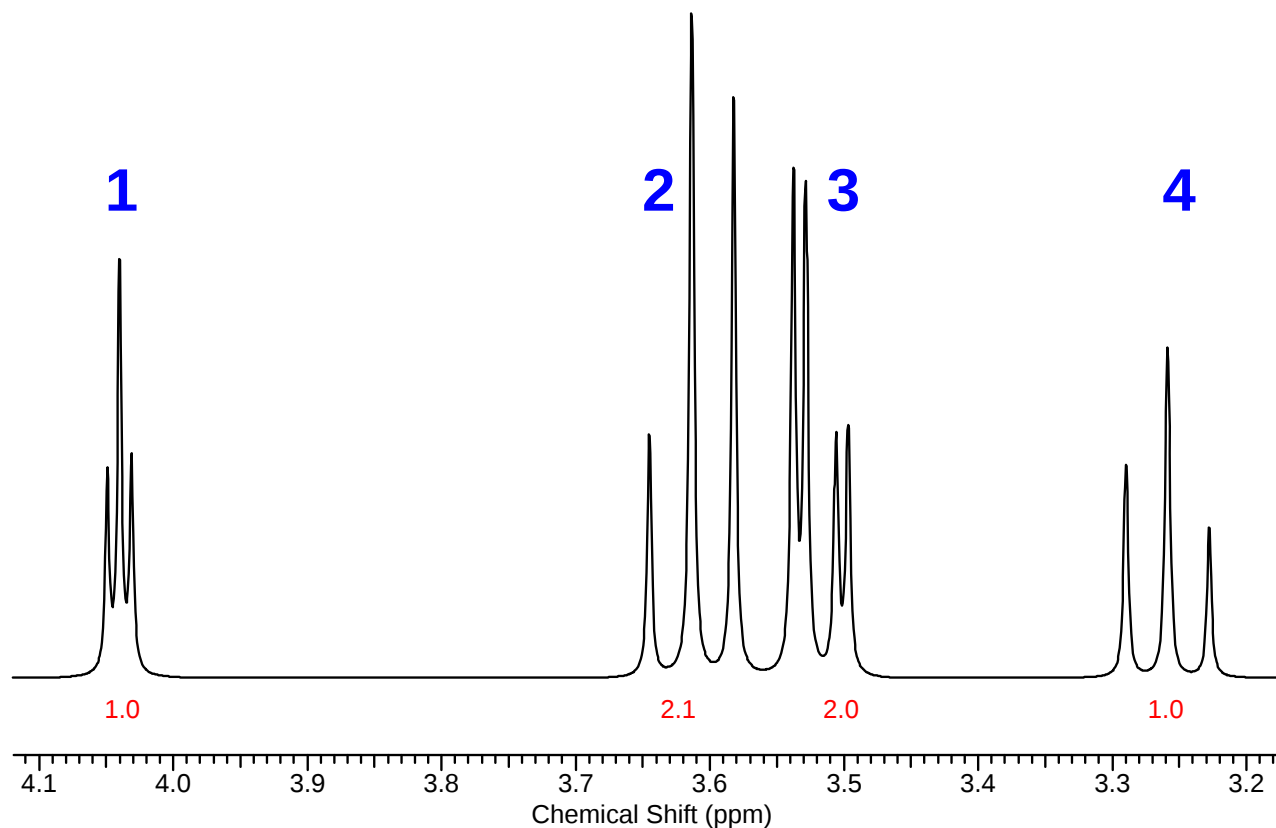


Relative stereochemistry on a ring: NOESY



Interpretation of J -coupling

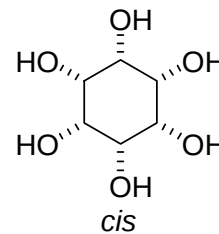
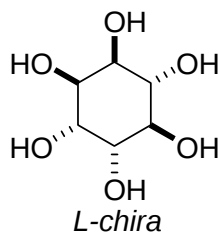
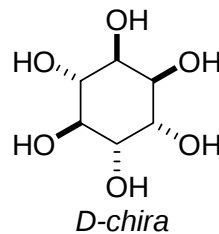
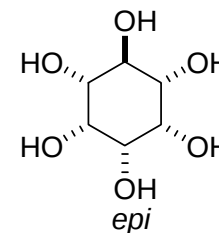
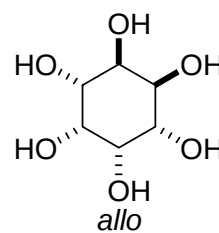
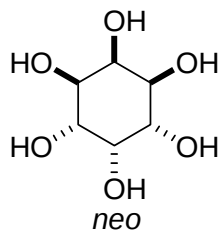
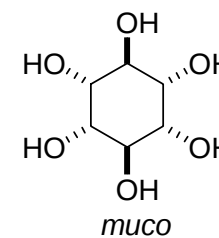
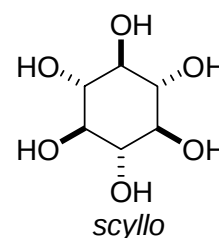
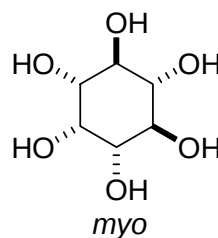
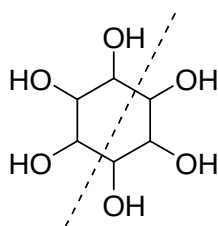
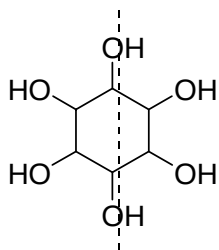
Unknown compound $C_6H_{12}O_6$ measured in D_2O
Detected J_{HH} -couplings: (2x9.6), (2.8, 9.6), (2x9.6), (2x2.8)



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

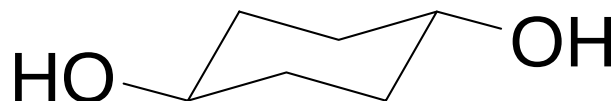
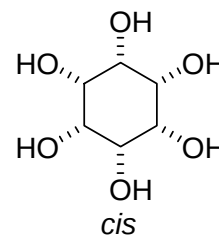
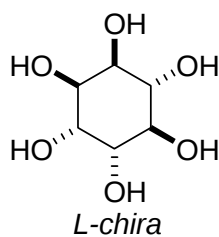
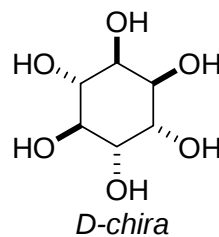
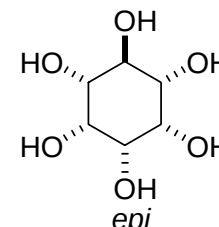
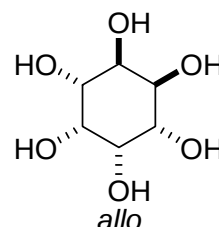
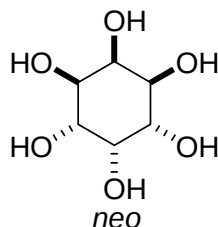
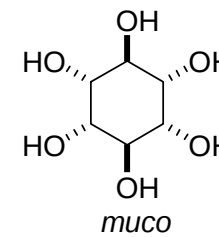
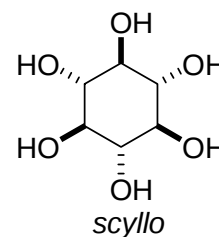
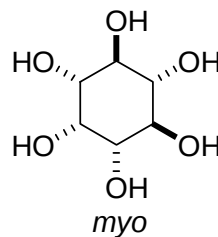
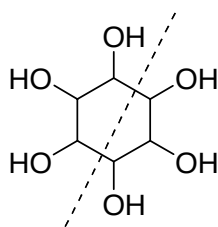
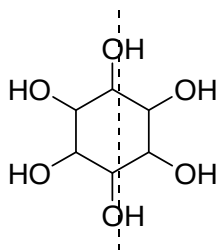
Interpretation of J -coupling

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



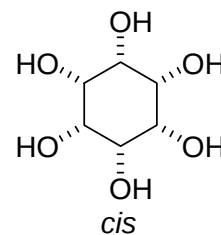
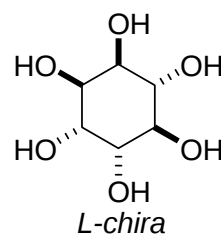
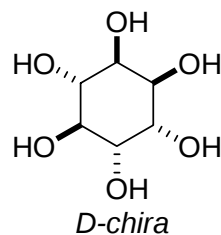
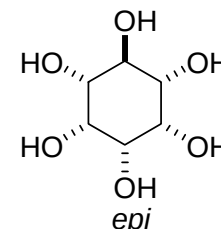
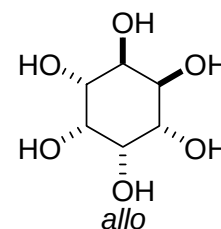
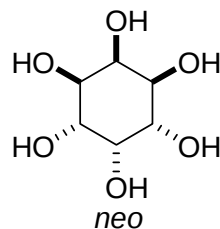
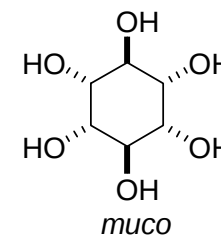
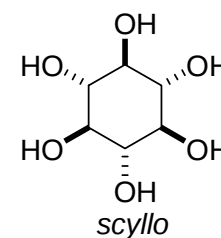
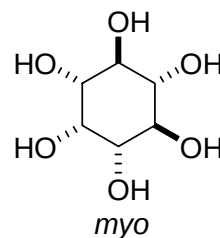
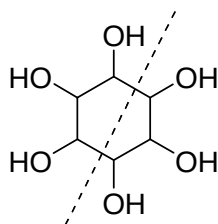
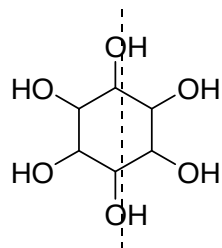
Interpretation of J -coupling

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

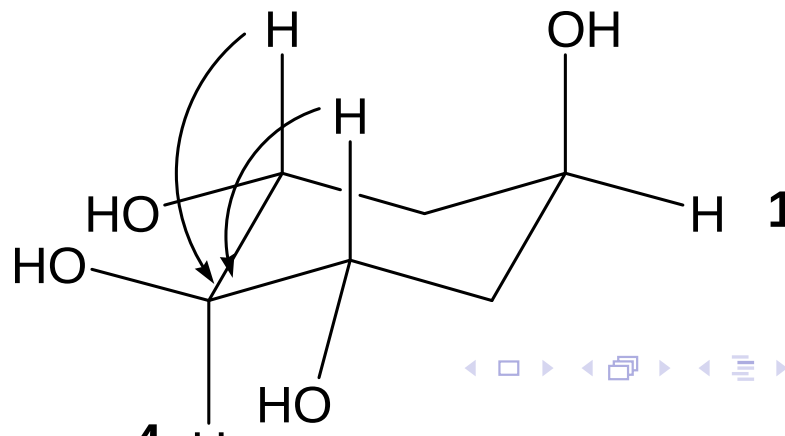


Interpretation of J -coupling

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



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)

