

C8953  
NMR strukturní analýza  
seminář

Elucidating structure using NMR

Jan Novotný

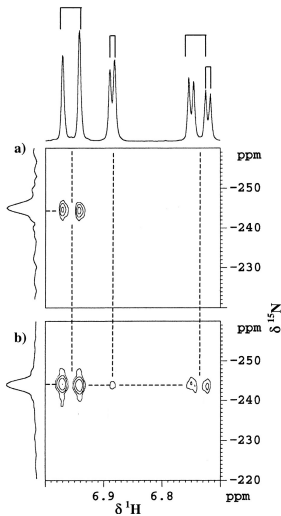
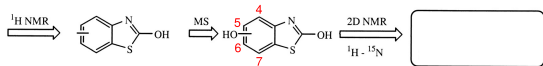
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April 8, 2020

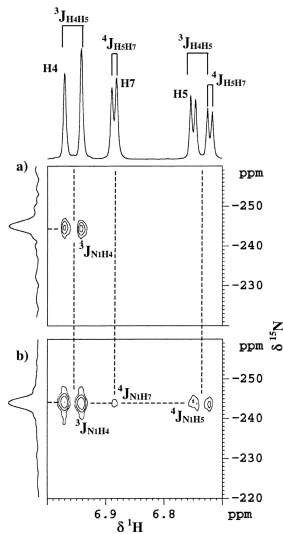
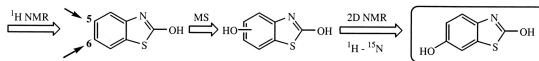
# Isomerisms and NMR

- ▶ 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)

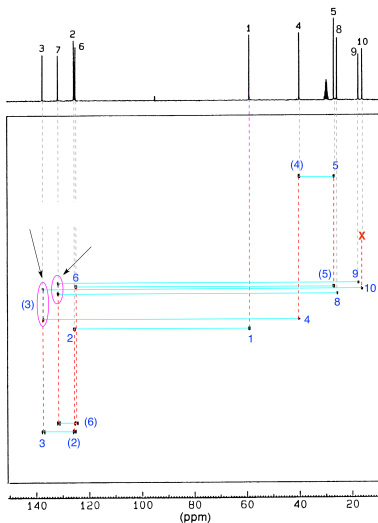
Benzothiazole Biodegradation:  $^1\text{H}$ - $^{15}\text{N}$  HMBC (*Appl. Environ. Microbiol.*, 2001, **67**)



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

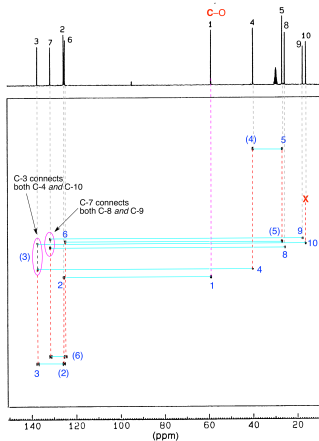
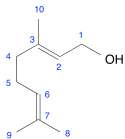


Determine the structure of  $C_{10}H_{18}O$  using INADEQUATE exp.

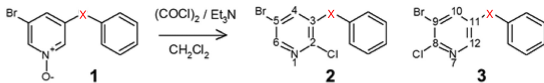


# Determine the structure of $C_{10}H_{18}O$ using INADEQUATE exp.

## 2D INADEQUATE spectrum of geraniol

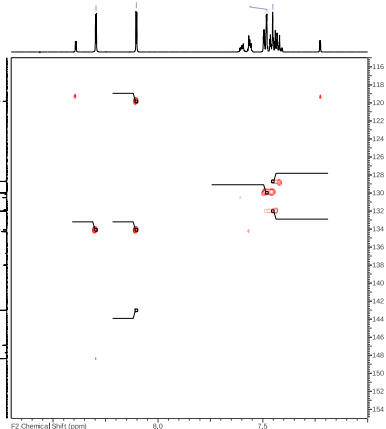
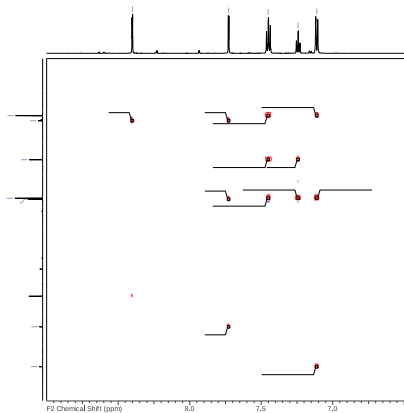


## Regioselectivity in the Halogenation: 1,1-ADEQUATE (*Org. Lett.*, 2016, 18, 19561959)

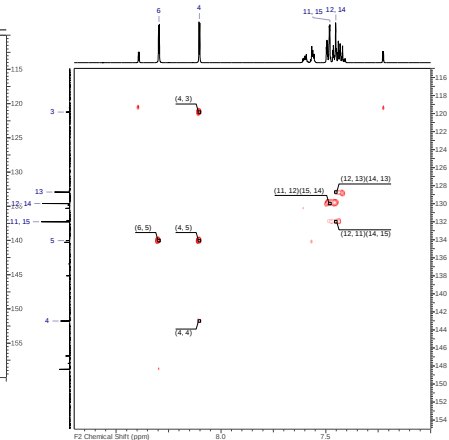
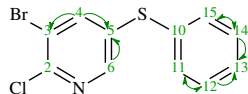
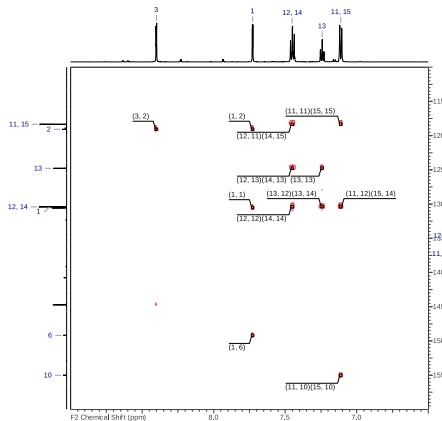
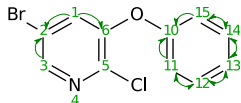


X = O

X = S



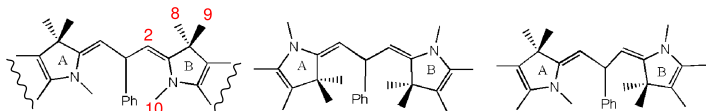
## Regioselectivity in the Halogenation: 1,1-ADEQUATE (*Org. Lett.*, 2016, 18, 19561959)





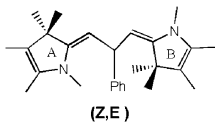
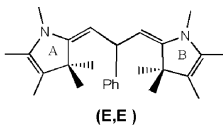
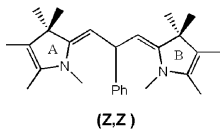
Configuration on double bonds (*Magn. Reson. Chem.*  
**2008**, 46, 872–877)

Describe the isomers of molecule shown bellow:

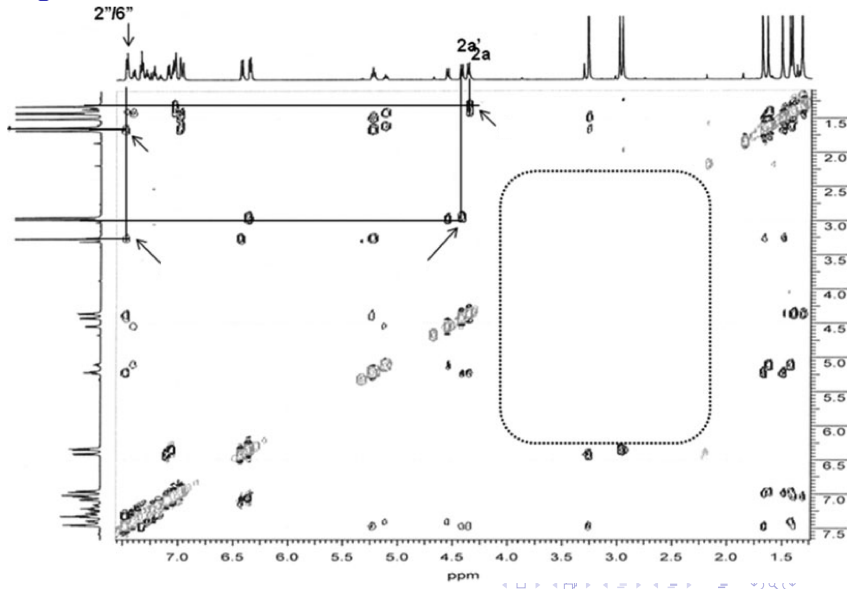


Configuration on double bonds (*Magn. Reson. Chem.*  
**2008**, 46, 872–877)

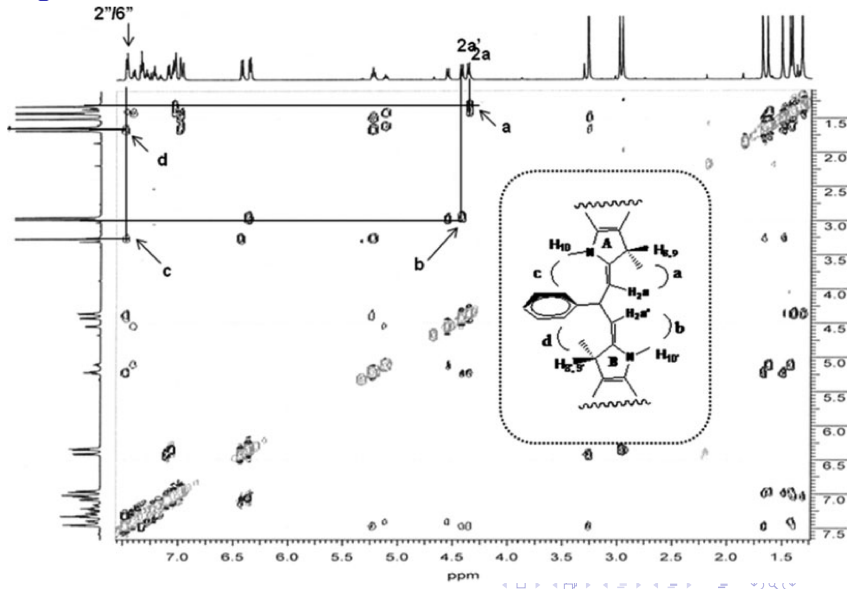
Describe the isomers of molecule shown bellow:



# Configuration on double bonds: NOESY

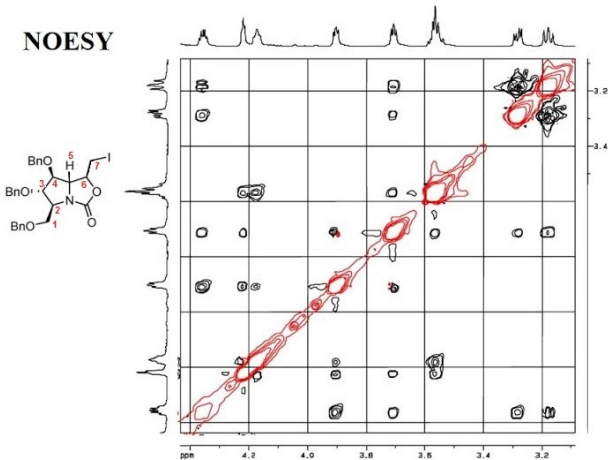


# Configuration on double bonds: NOESY

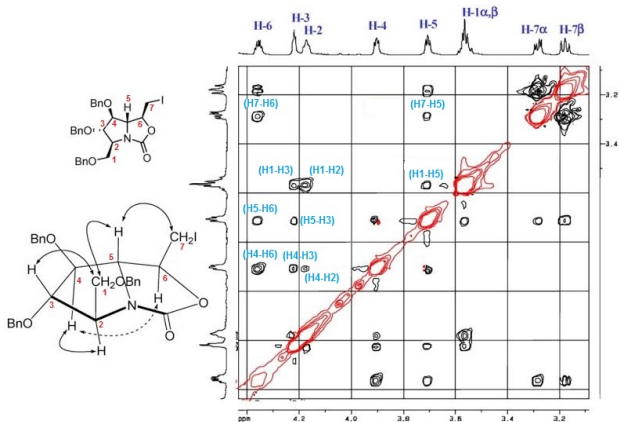


# Relative stereochemistry on a ring: NOESY

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

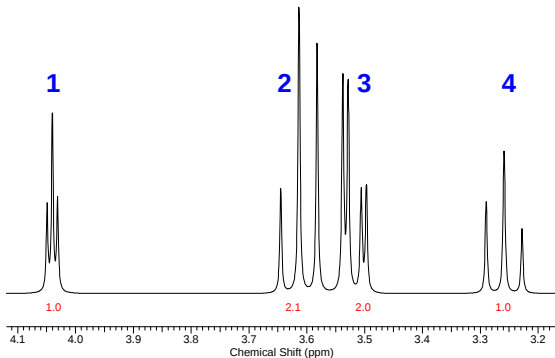


# Relative stereochemistry on a ring: NOESY



## Interpretation of $J$ -coupling

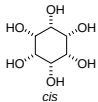
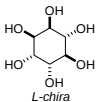
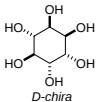
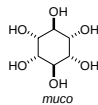
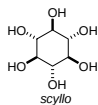
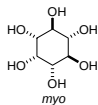
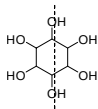
**Unknown compound  $C_6H_{12}O_6$  measured in  $D_2O$**   
Detected  $J_{HH}$ -couplings: (2x9.6), (2.8, 9.6), (2x9.6), (2x2.8)



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

# Interpretation of $J$ -coupling

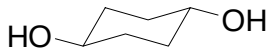
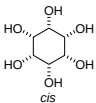
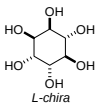
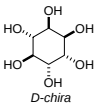
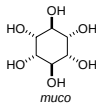
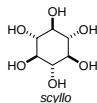
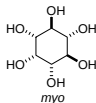
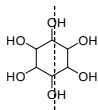
$J_{HH}$ -couplings: (2x9.6), (2.8, 9.6), (2x9.6), (2x2.8)





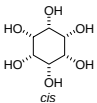
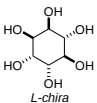
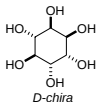
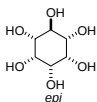
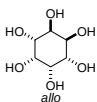
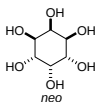
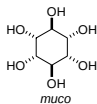
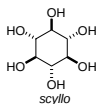
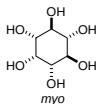
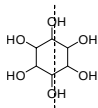
# Interpretation of $J$ -coupling

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

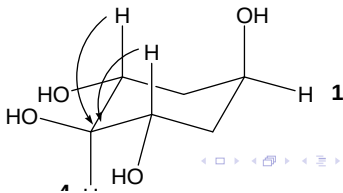


# Interpretation of $J$ -coupling

$J_{HH}$ -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)

