

# Applications of NMR spectroscopy in supramolecular chemistry

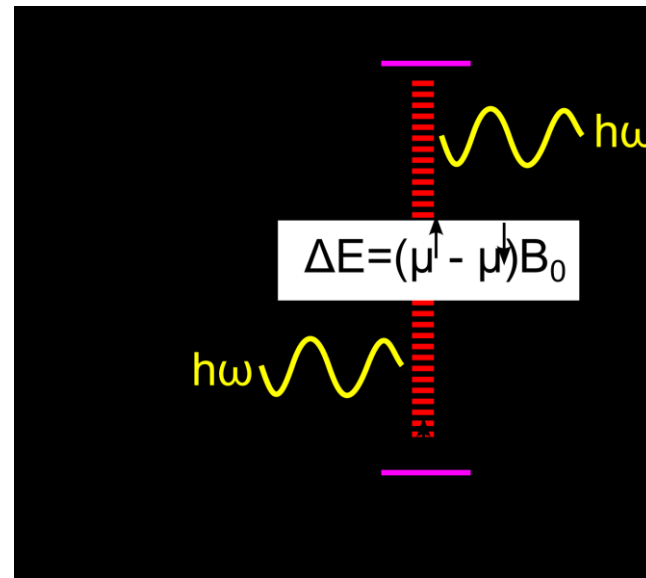
FSC2SB-M3 2023

**Jan Novotny**

RG of Radek Marek

# NMR spectroscopy

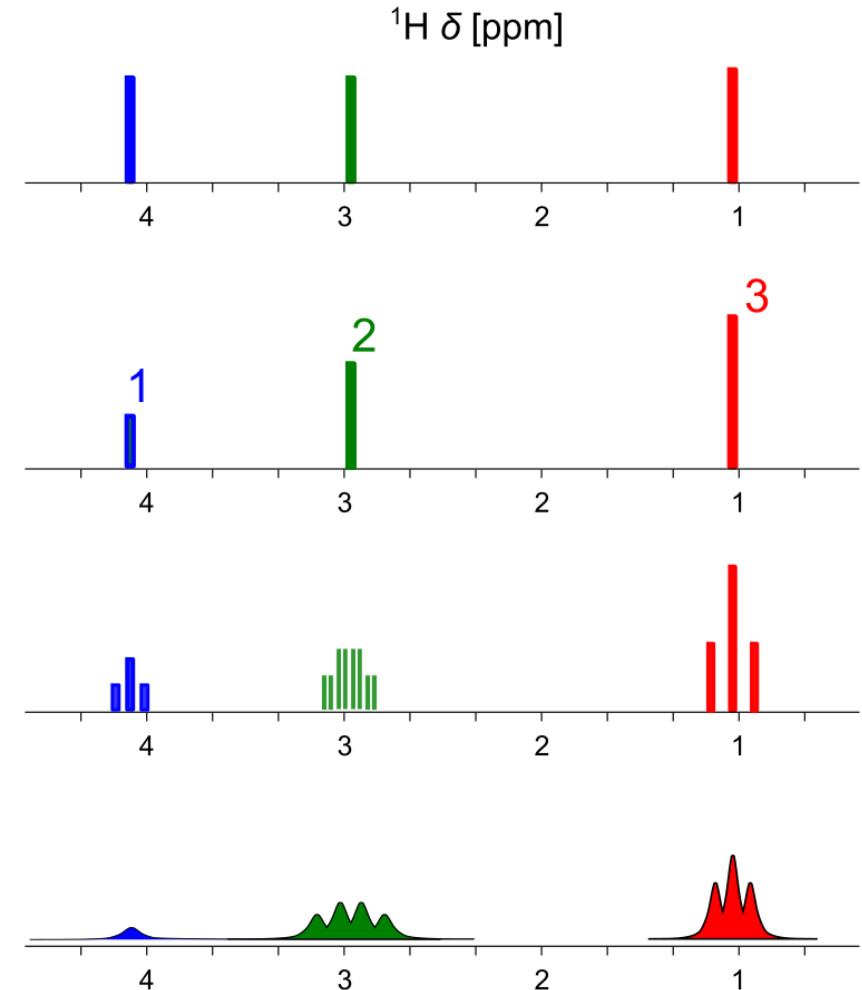
- Transitions of nuclear magnetic moment ( $\alpha \rightarrow \beta$ ) in external magnetic field ( $\sim 10$  T) through RF irradiations (MHz)



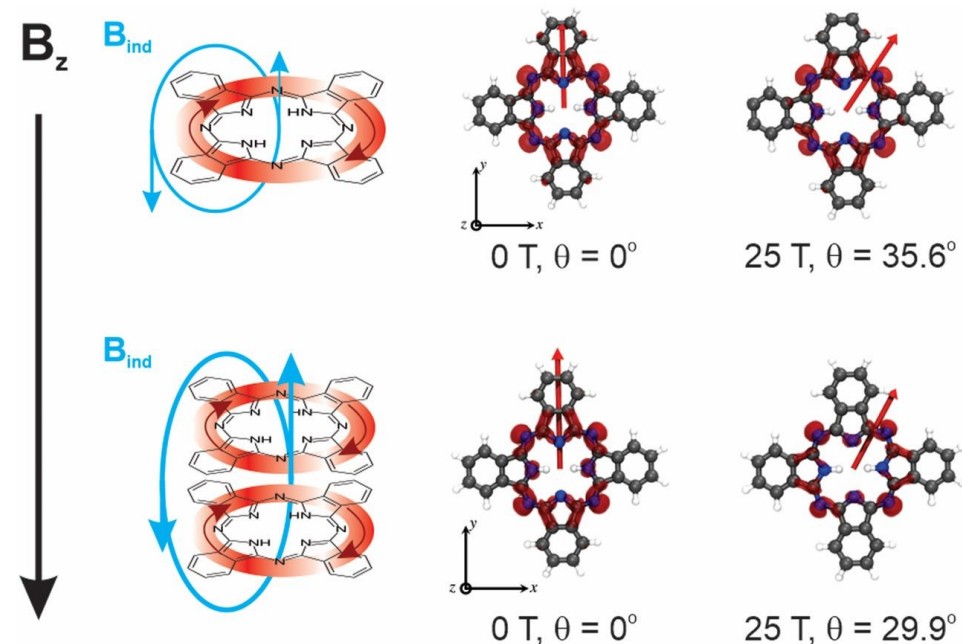
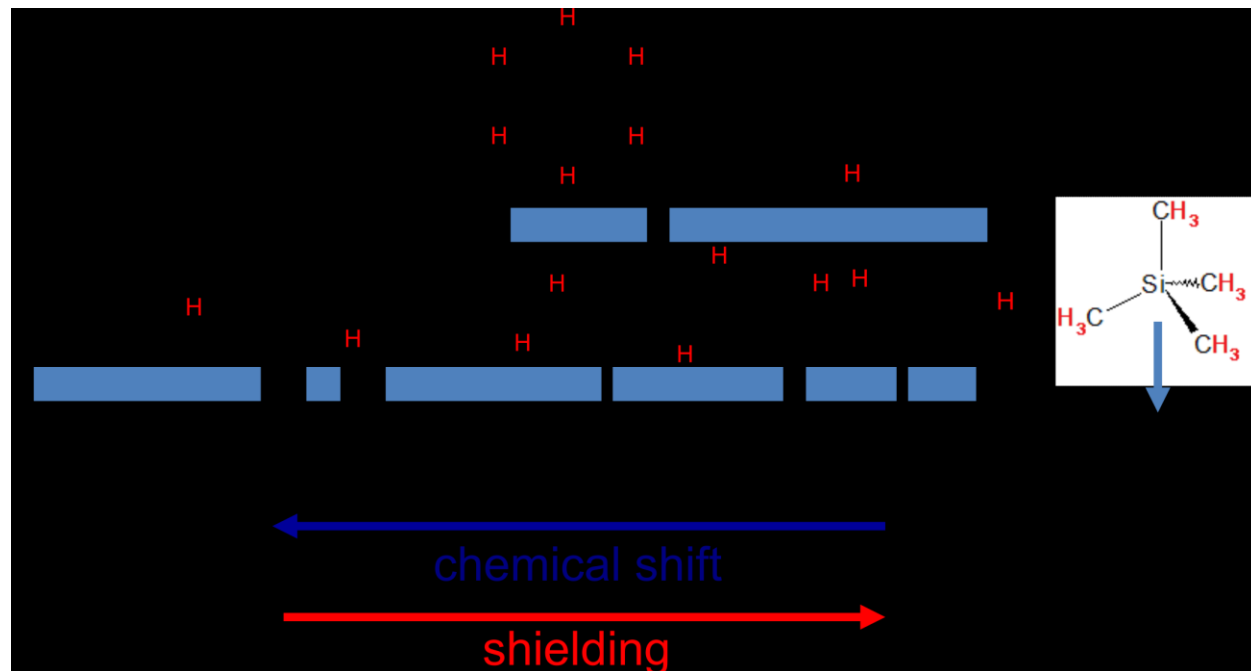
- Atomistic resolution, non-invasive, solution environment, structure & dynamics of molecules
- Low sensitivity, expensive equipment, spectra complicated by interatomic interactions and external inhomogeneities  $\rightarrow$  relaxation of signal

# 1D NMR spectrum – basic parameters

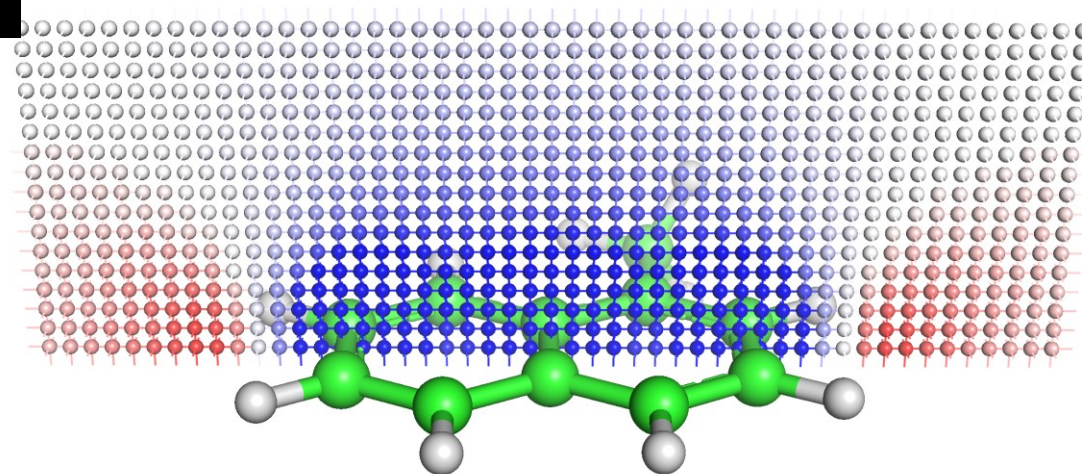
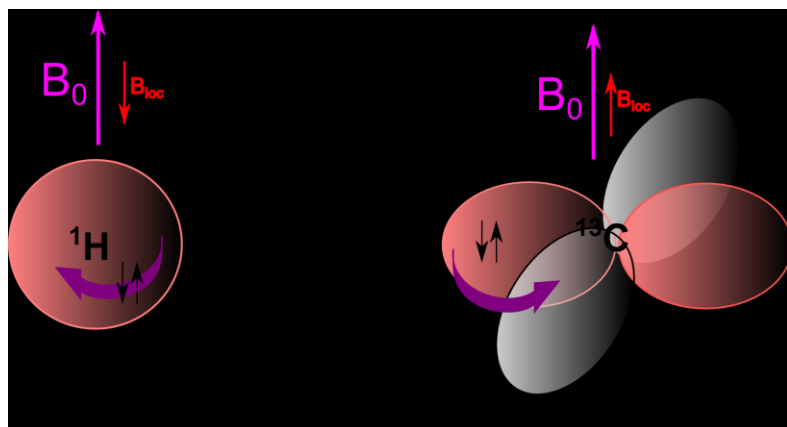
- Shielding by valence electrons – chemical shift ( $\delta$ )
- Chemically non-equivalent atoms – number of signals
- Quantity of species – signal integrals
- Spin-spin interactions ( $J$ -coupling) – peak multiplicity
- Relaxation of signal – peak linewidth



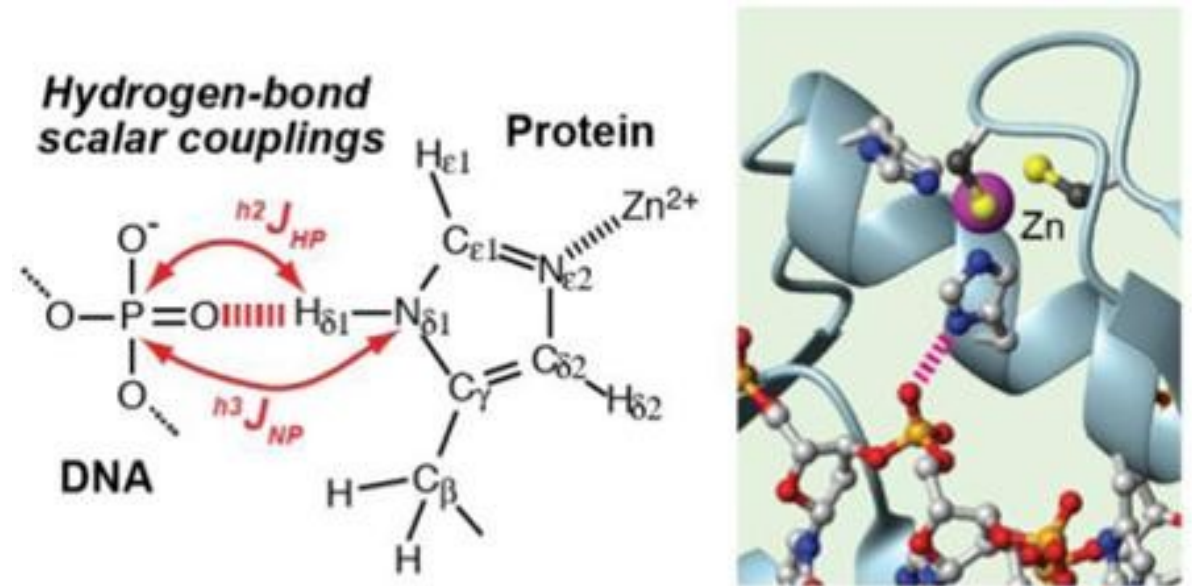
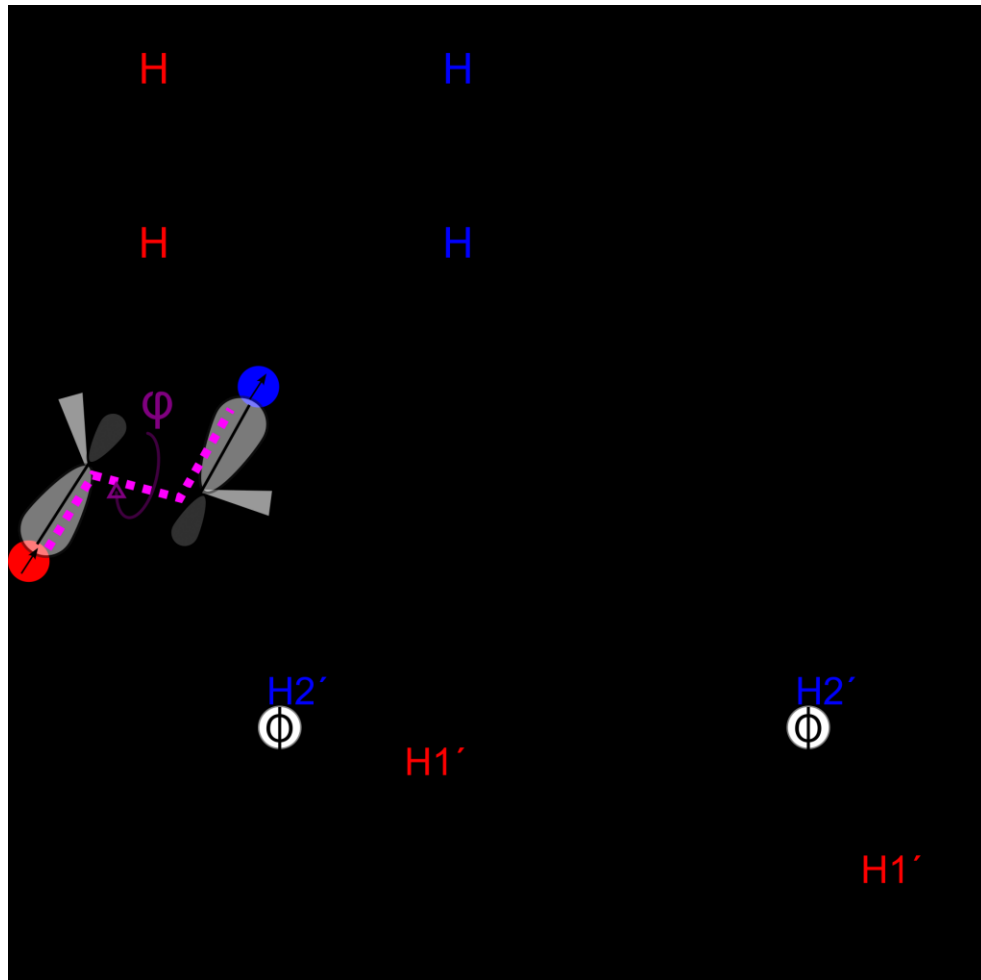
# Chemical shift and CSA



<https://doi.org/10.1073/pnas.1918148117>

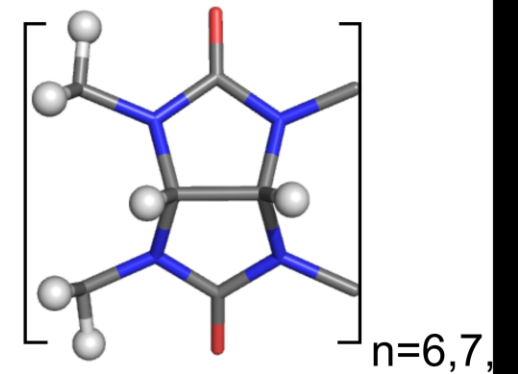
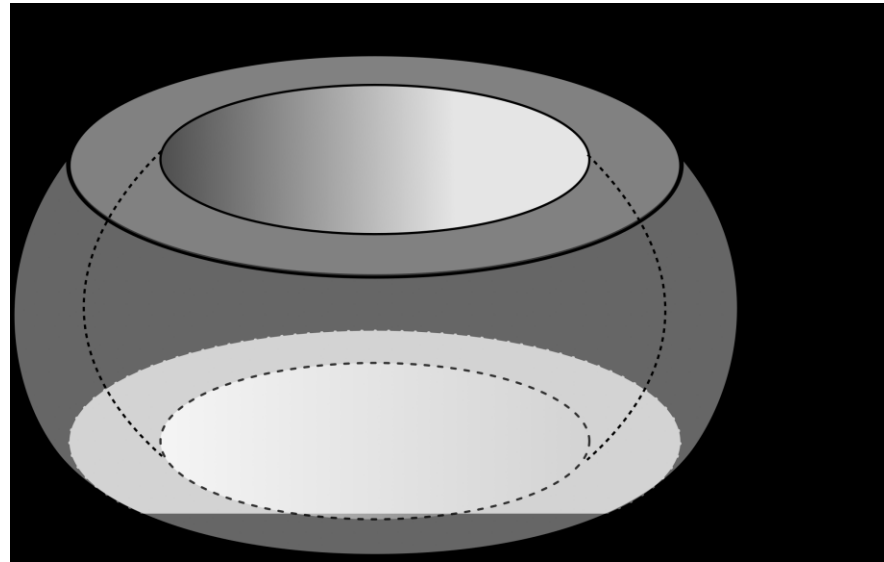
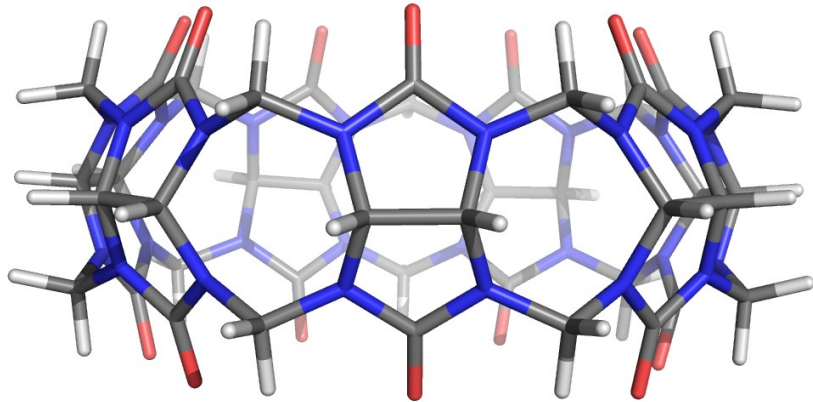


# Indirect dipol-dipol interaction ( $J$ -coupling) - spin polarisation of valence e

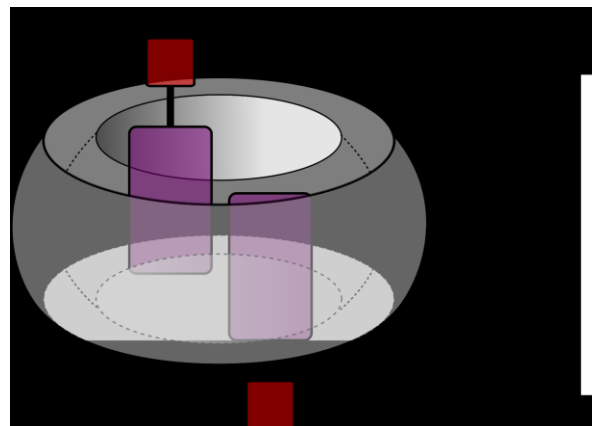
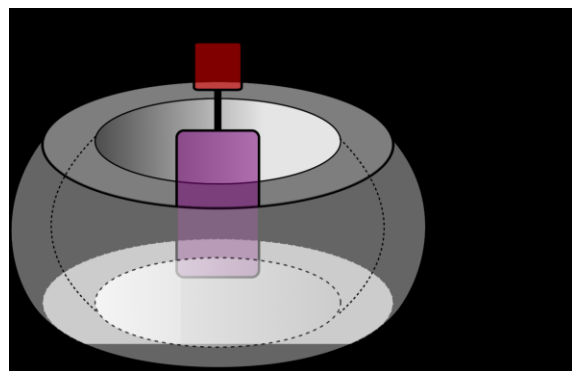
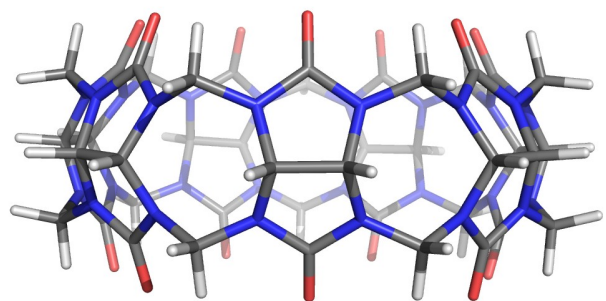
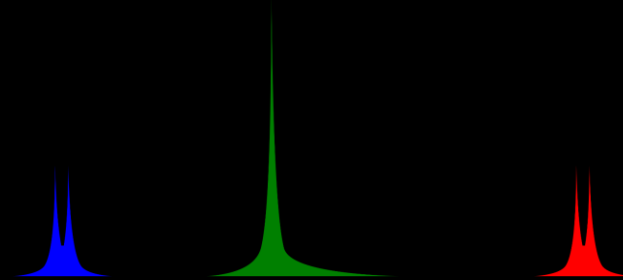
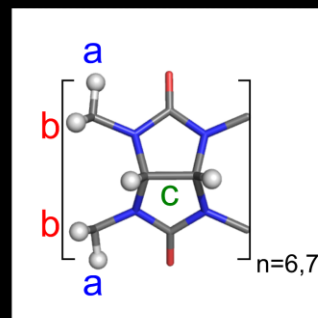
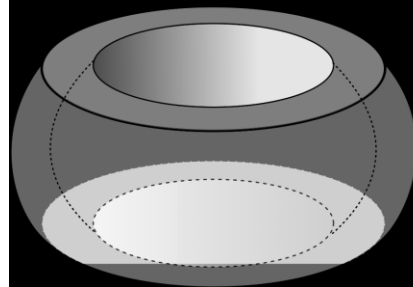


[J Phys Chem B. 2016 Oct 20; 120\(41\): 10679–10685.](#)

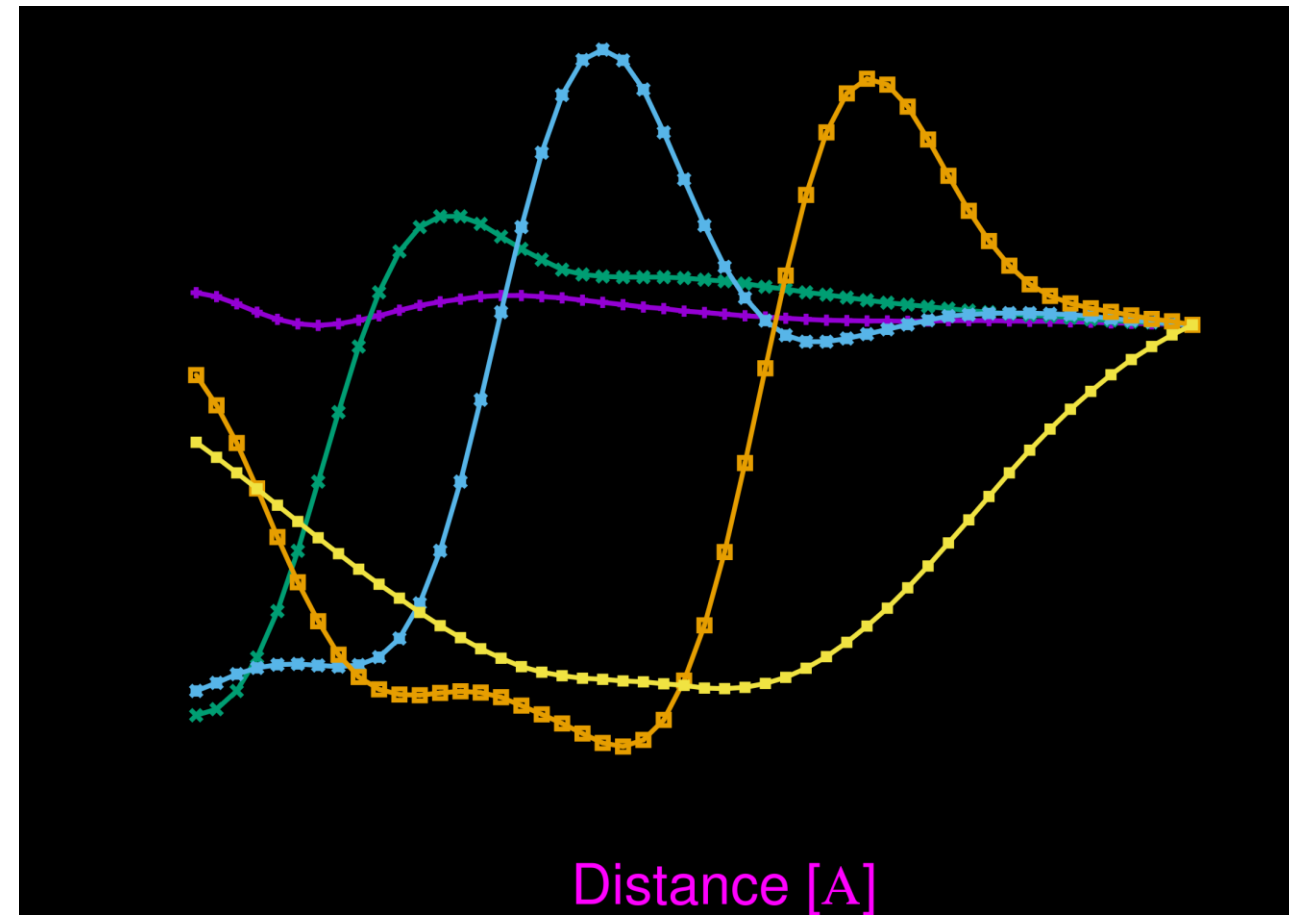
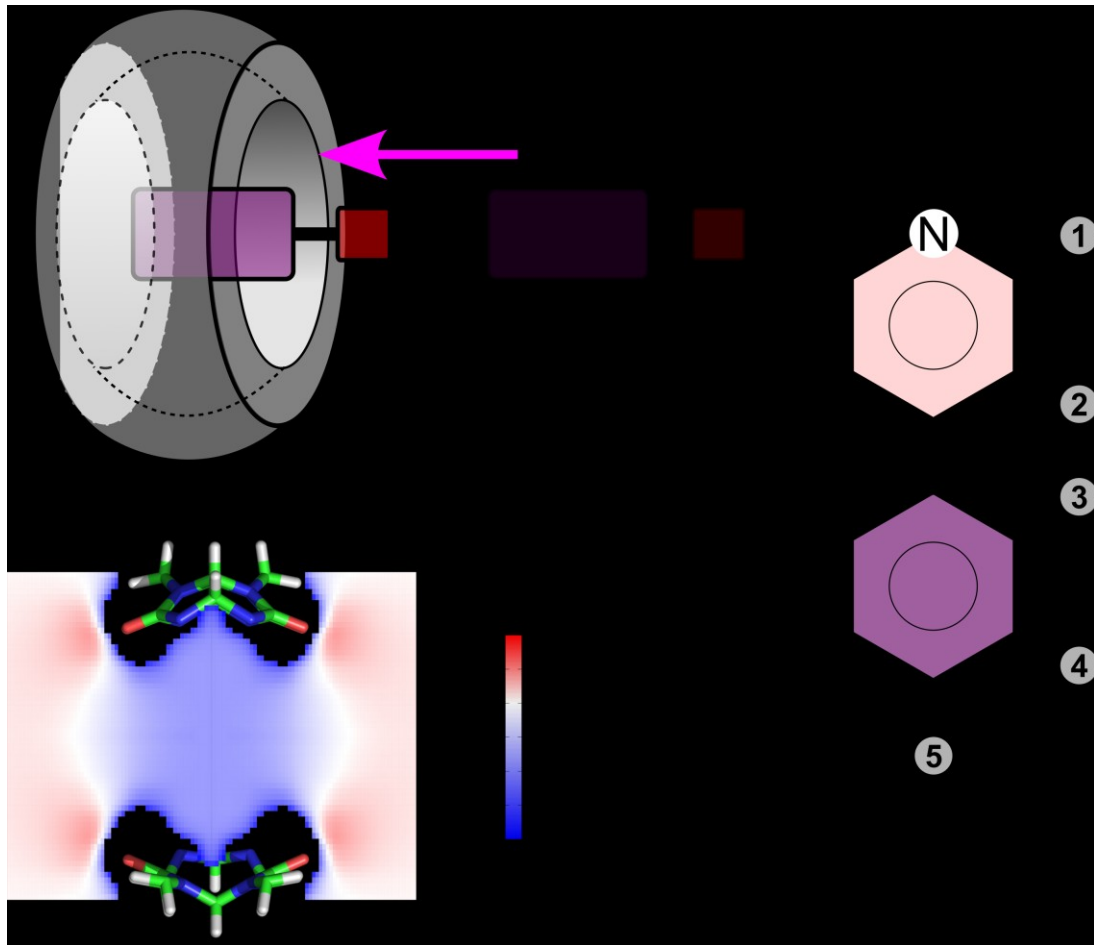
# Number of $^1\text{H}$ NMR signals – symmetry of the host system



# Number of $^1\text{H}$ NMR signals - symmetry of the UOCT system

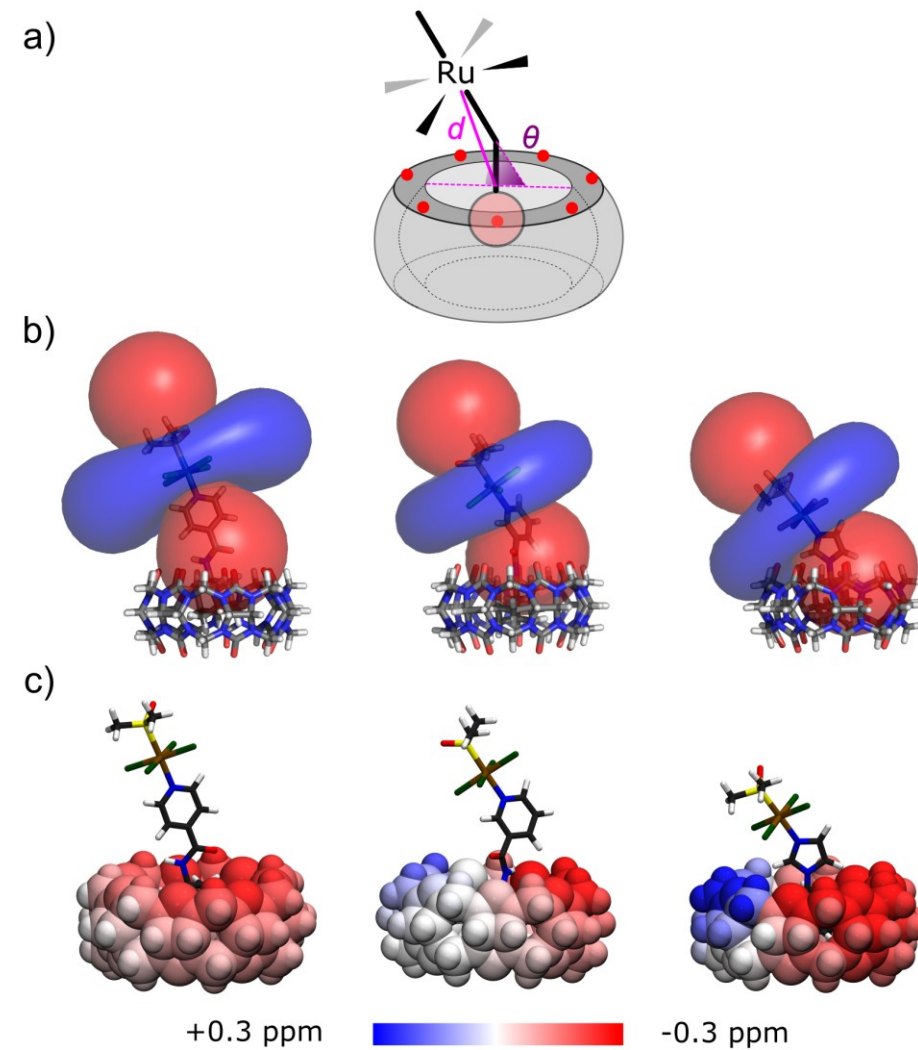
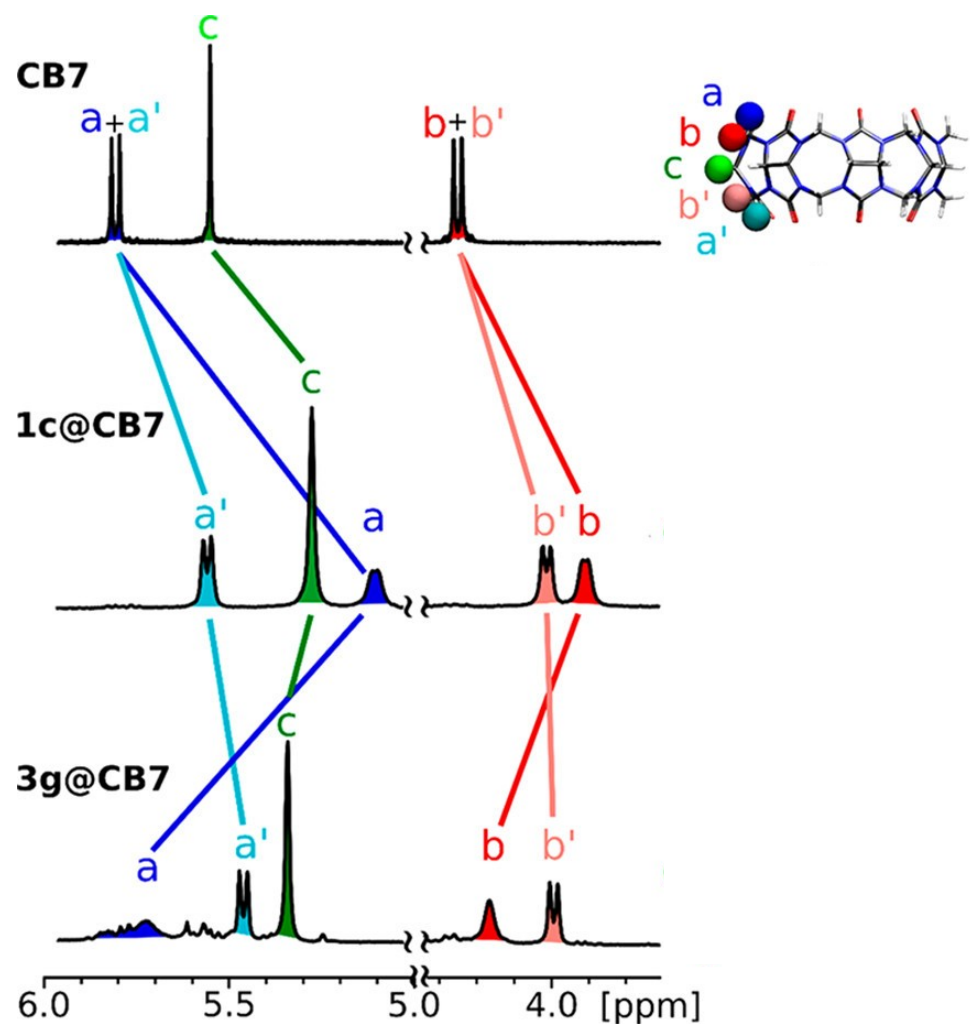


# Chemical shift induced by HOST on GUEST atoms

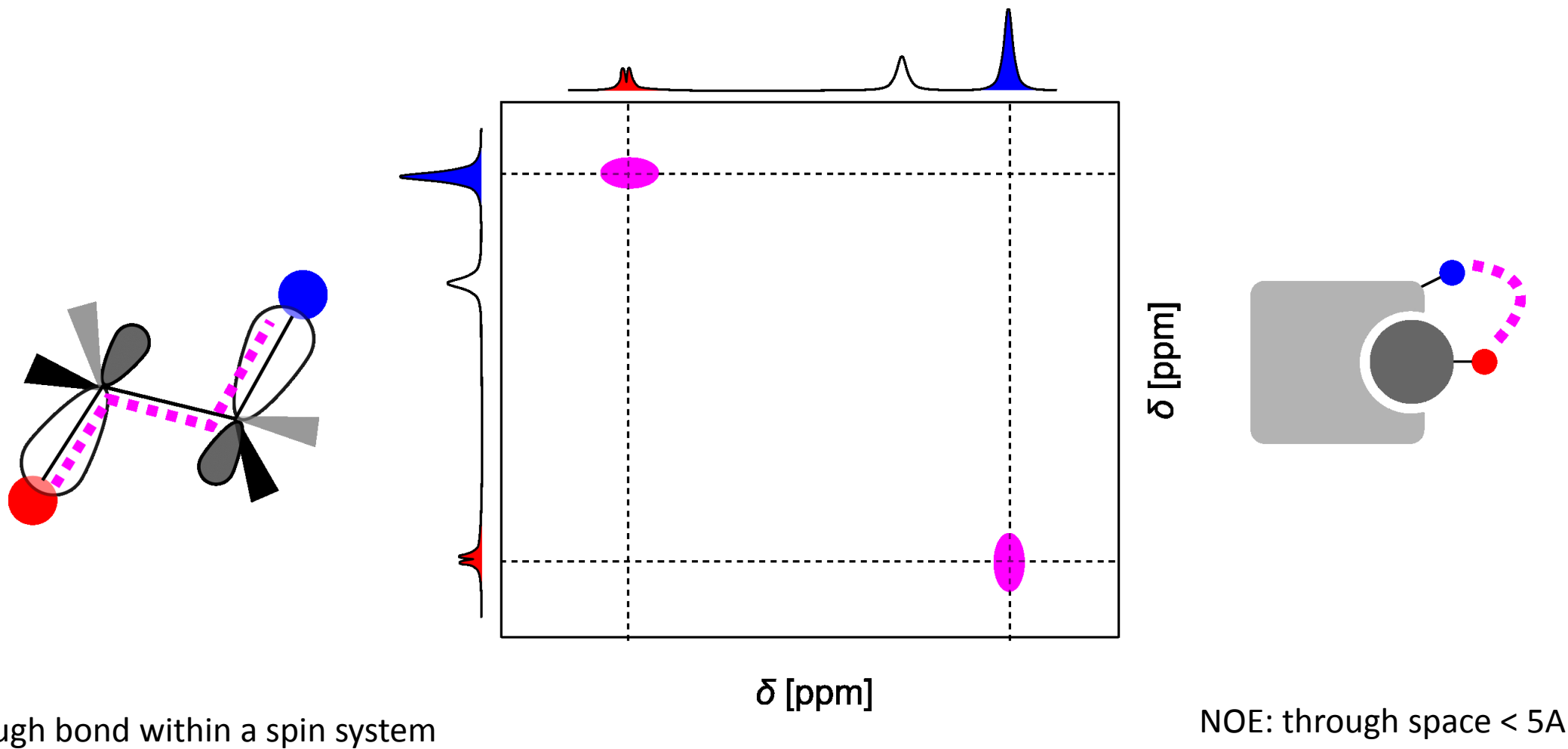




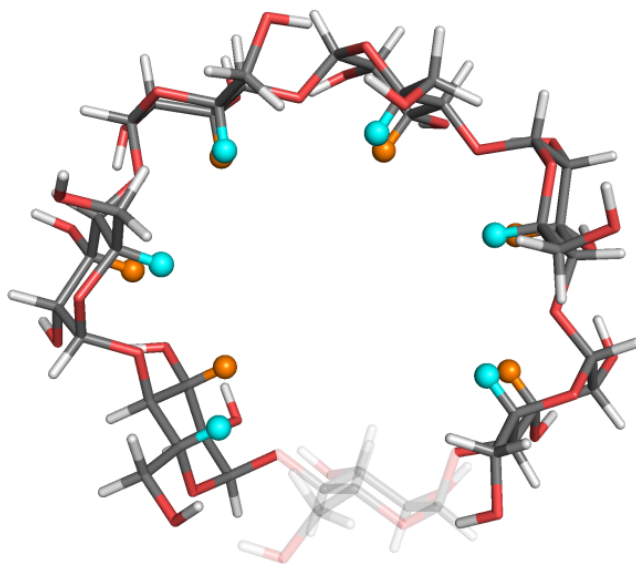
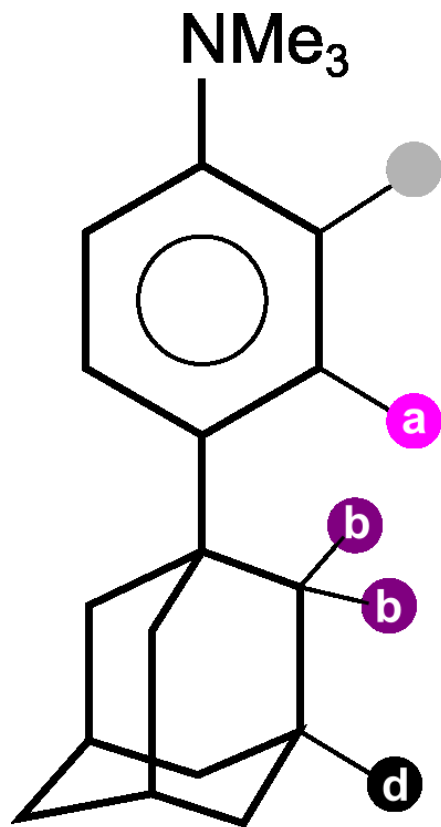
# Paramagnetic shift of Ru-guest perturbs $^1\text{H}$ signals of CB7 host



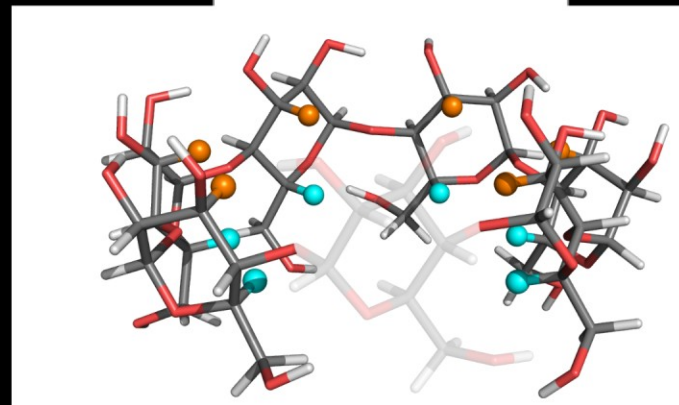
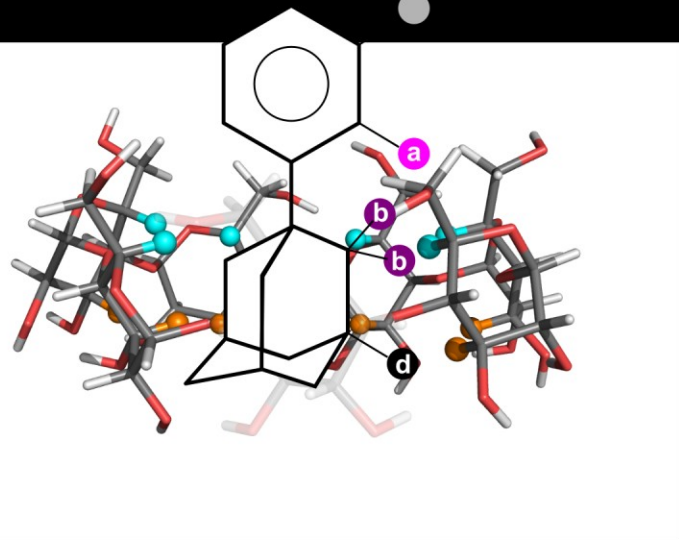
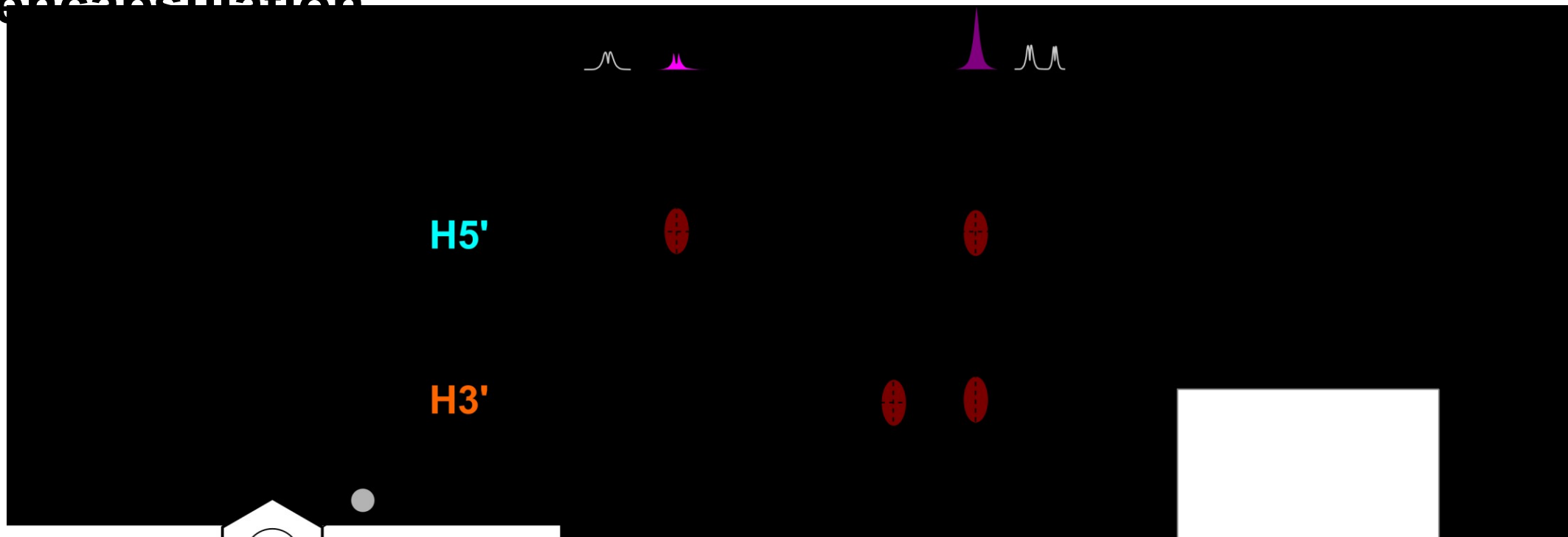
# Multidimensional NMR methods - correlations



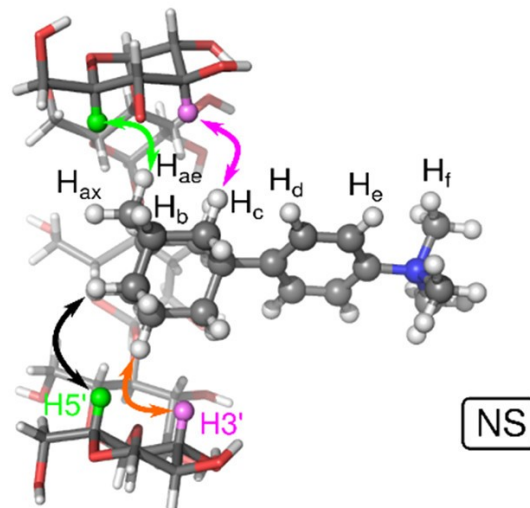
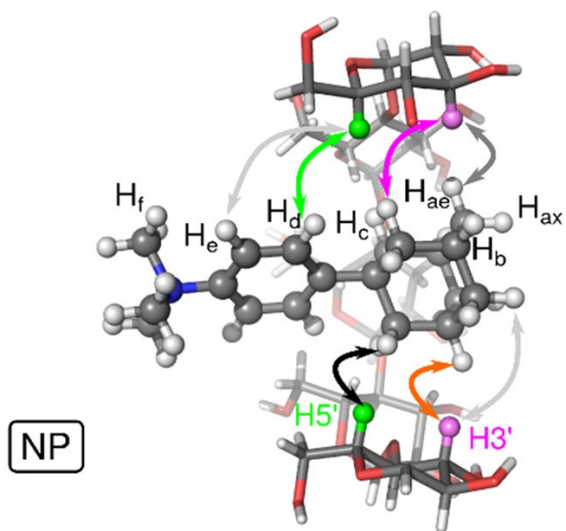
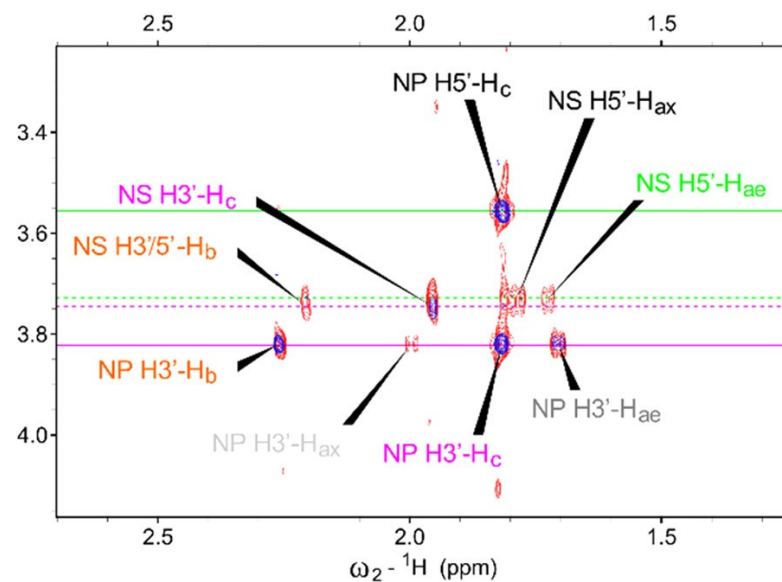
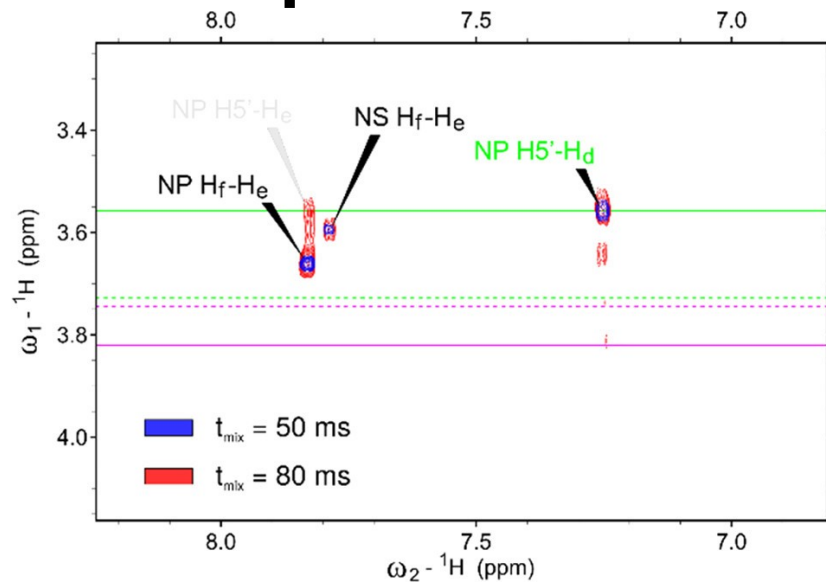
# Through space correlation – determination of the mode of encapsulation into asymmetric host



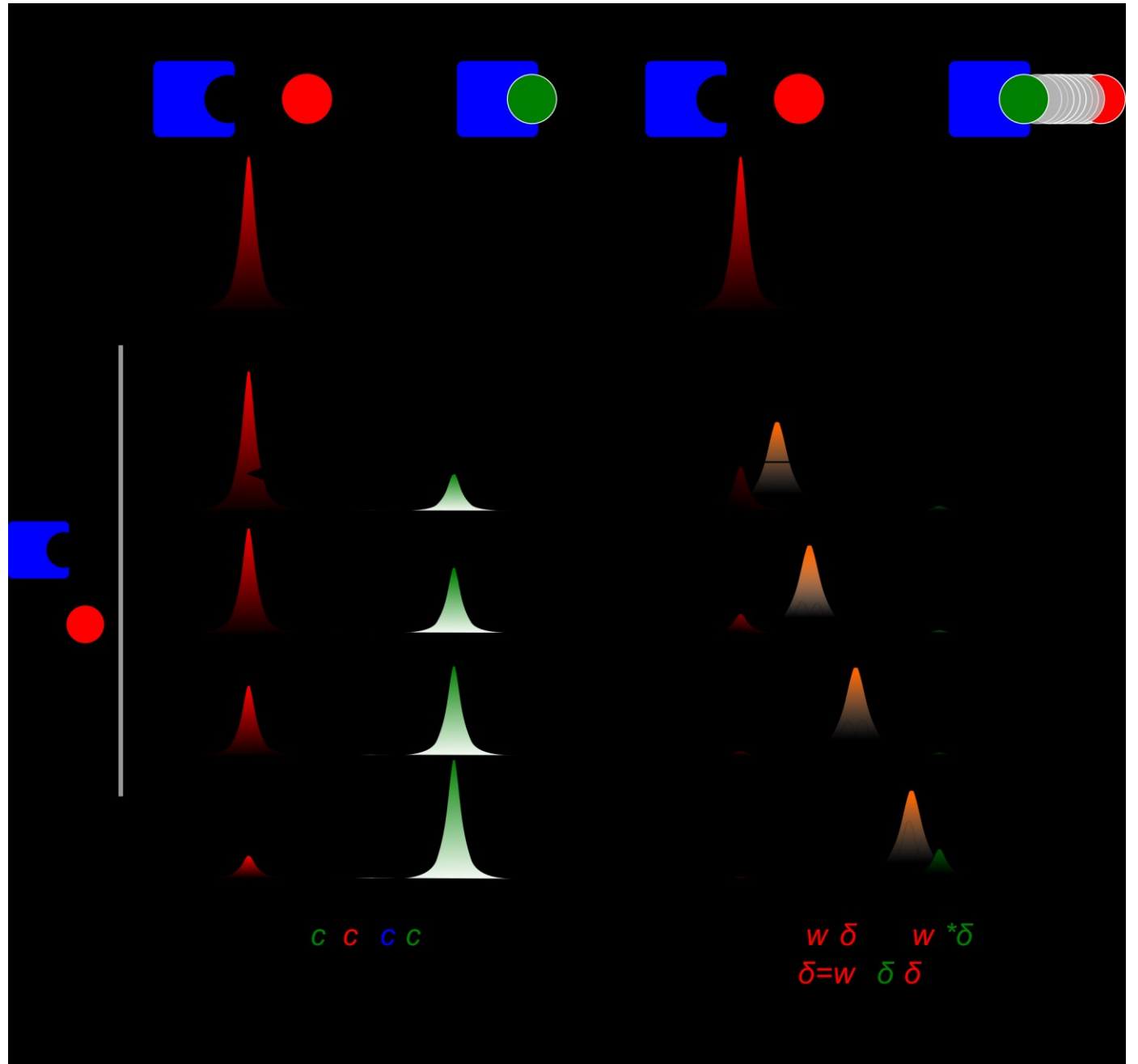
# Through space correlation – determination of the mode of encapsulation



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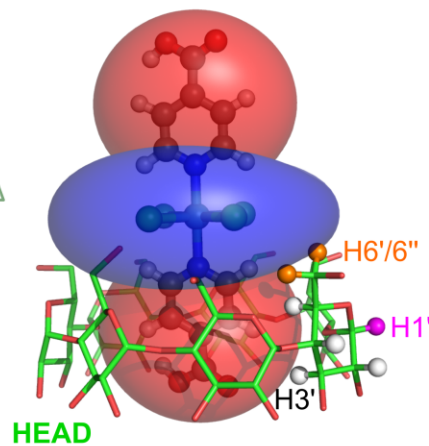
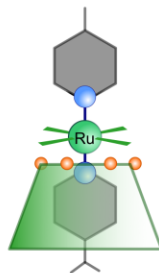
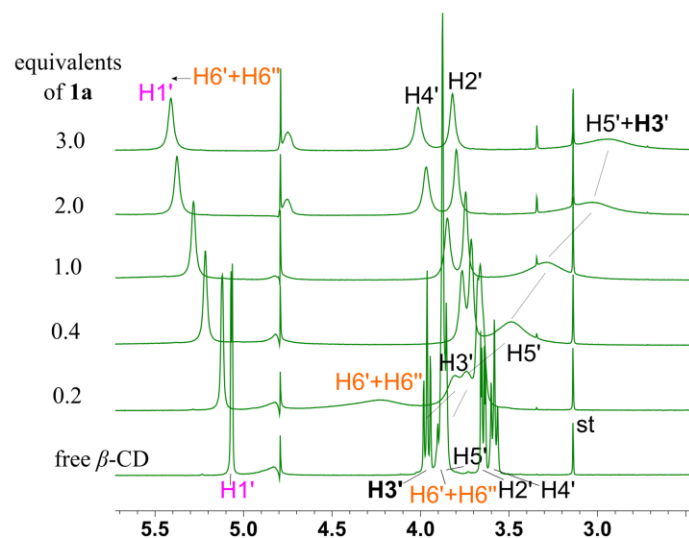


# HG binding – regimes of chemical exchange

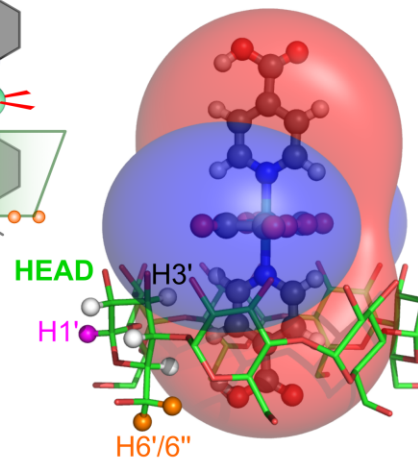
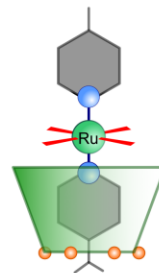
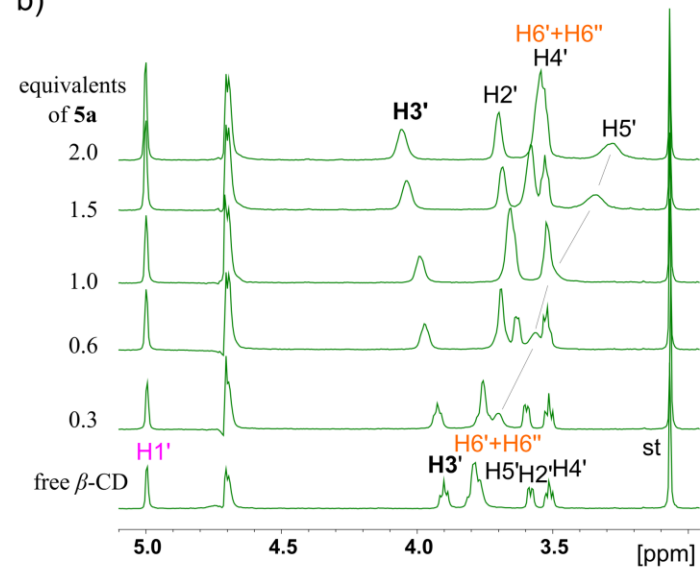


# NMR of inclusion complexes with open shell ligands

a)

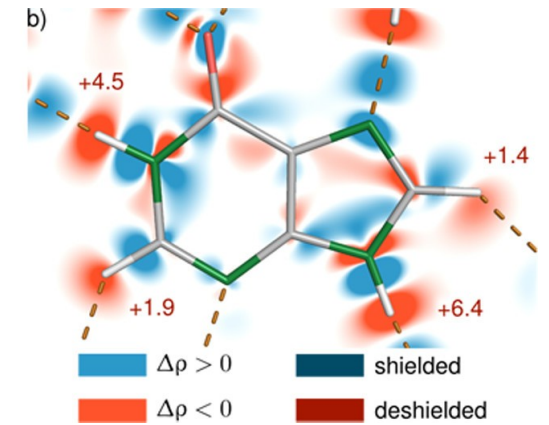
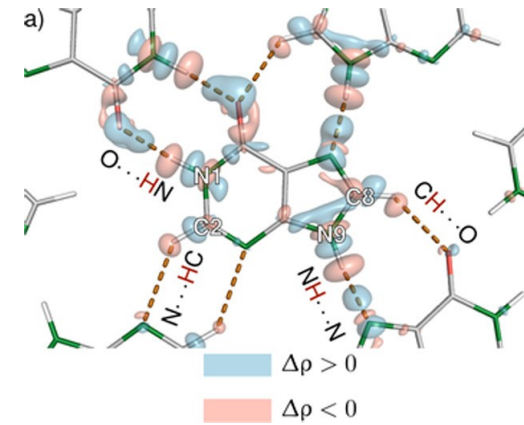
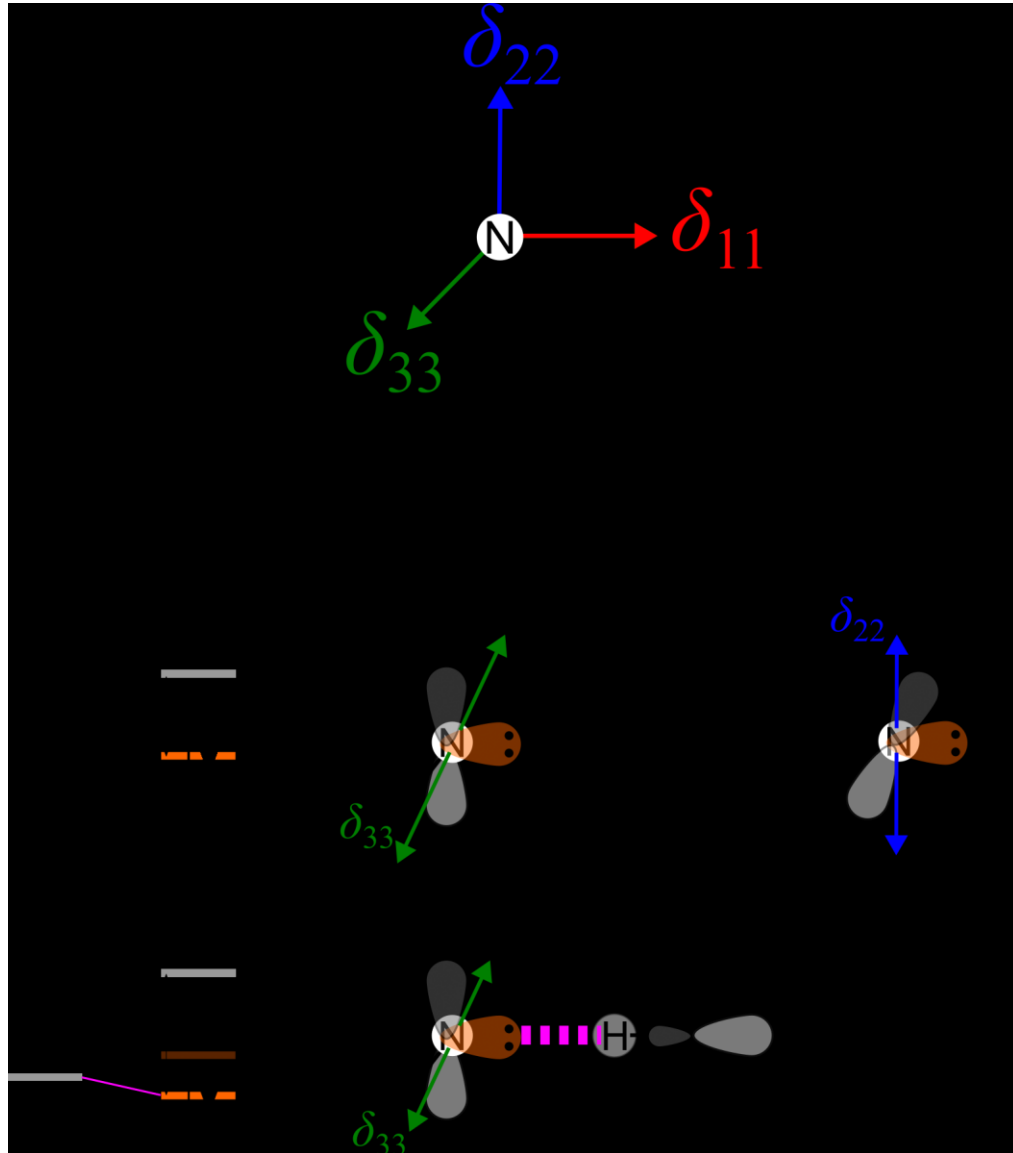


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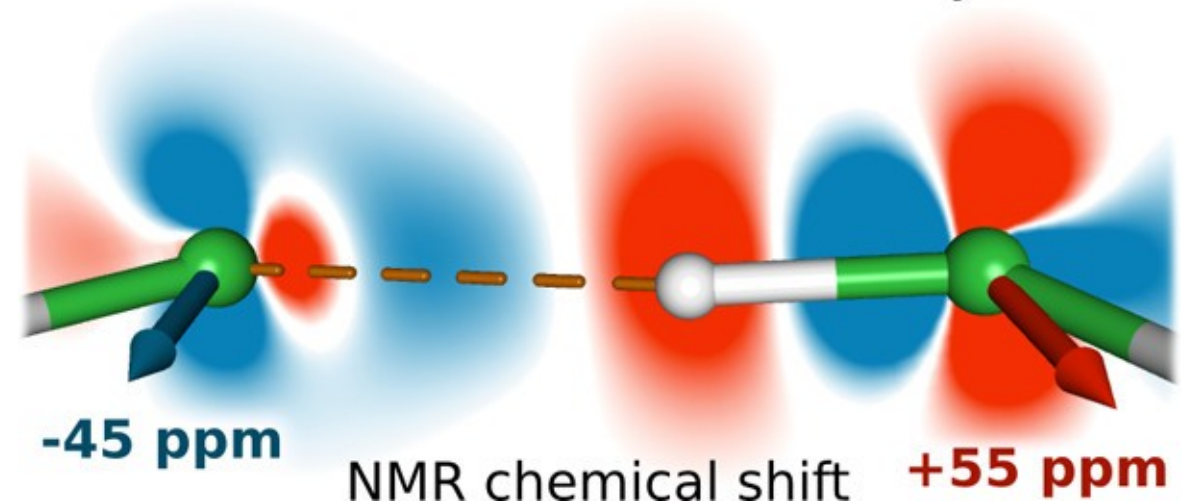




# Hydrogen bonding characterized by ssNMR



Electron deformation density





# Thanks for Your attention

<https://www.ceitec.eu/structure-of-biosystems-and-molecular-materials/rg108/publication>