5.13: Nuclear Overhauser Effect (NOE) and 2-D NMR

The use of pulses of different shapes, frequencies and durations in specifically-designed patterns or pulse sequences allows the spectroscopist to extract many different types of information about the molecule. Multi-dimensional nuclear magnetic resonance spectroscopy is a kind of FT NMR in which there are at least two pulses and, as the experiment is repeated, the pulse sequence is varied. In multidimensional nuclear magnetic resonance there will be a sequence **of pulses** and, at least, one variable time period. In three dimensions, two time sequences will be varied. In four dimensions, three will be varied.

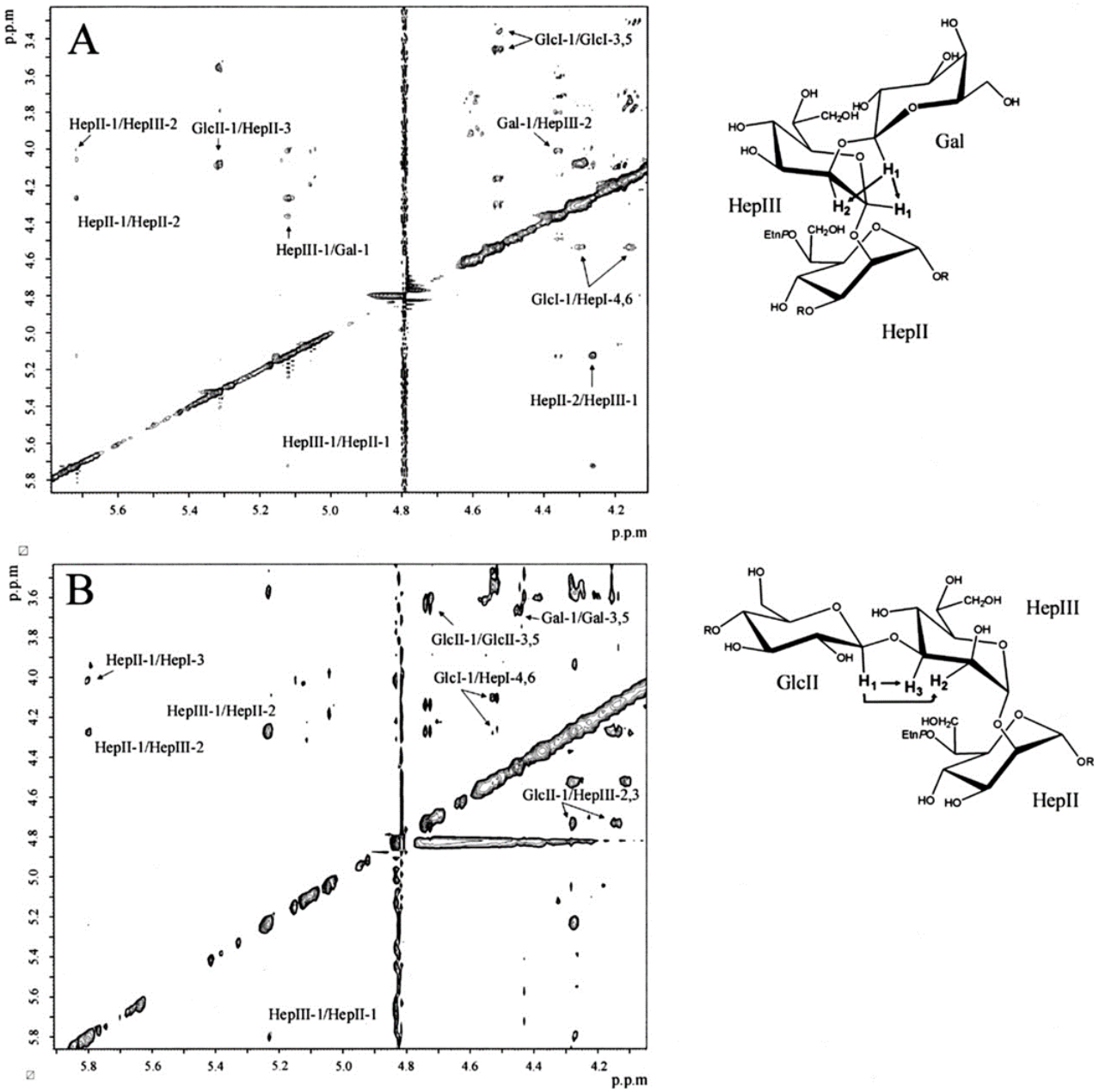
There are many such experiments. In one, these time intervals allow (amongst other things) magnetization transfer between nuclei and, therefore, the detection of the kinds of nuclear-nuclear interactions that allowed for the magnetization transfer. Interactions that can be detected are usually classified into two kinds.

1. There are through-bond interactions and
2. through-space interactions, the latter usually being a consequence of the nuclear Overhauser effect.

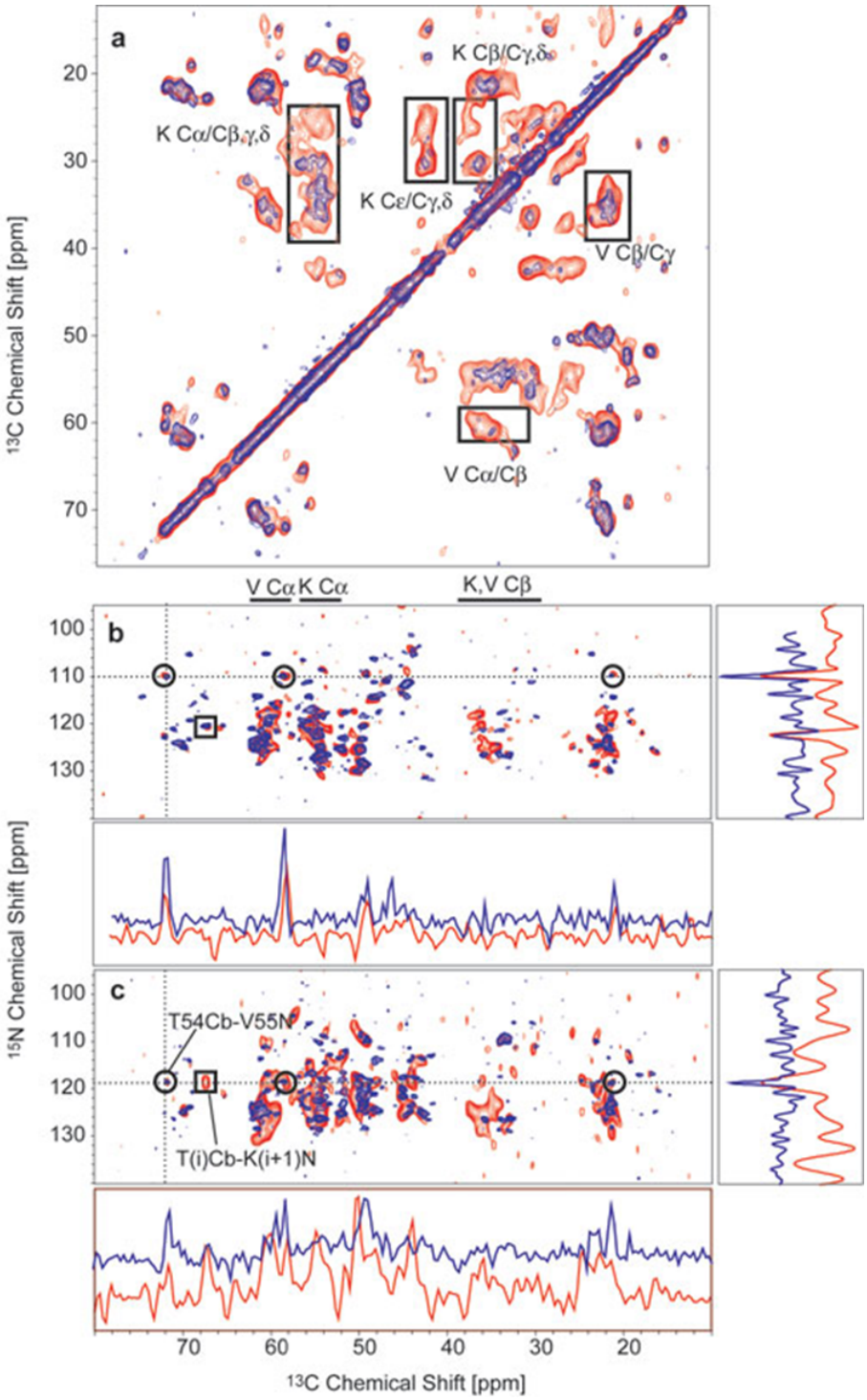
Such experiments may be employed to establish distances between atoms, as for example by 2D-FT NMR of molecules in solution. NOE differs from **spin coupling in the respect that NOE is observed through space, not through bonds**. Thus, all atoms that are in close proximity to each other give a NOE, whereas spin coupling is observed only when the atoms are bonded to same or neighboring atoms. Furthermore, the distance can be derived from the observed NOEs, so that the precise, three-dimensional structure of the molecule can be reconstructed.

Other experimental techniques exploiting the NOE include and are not limited to:

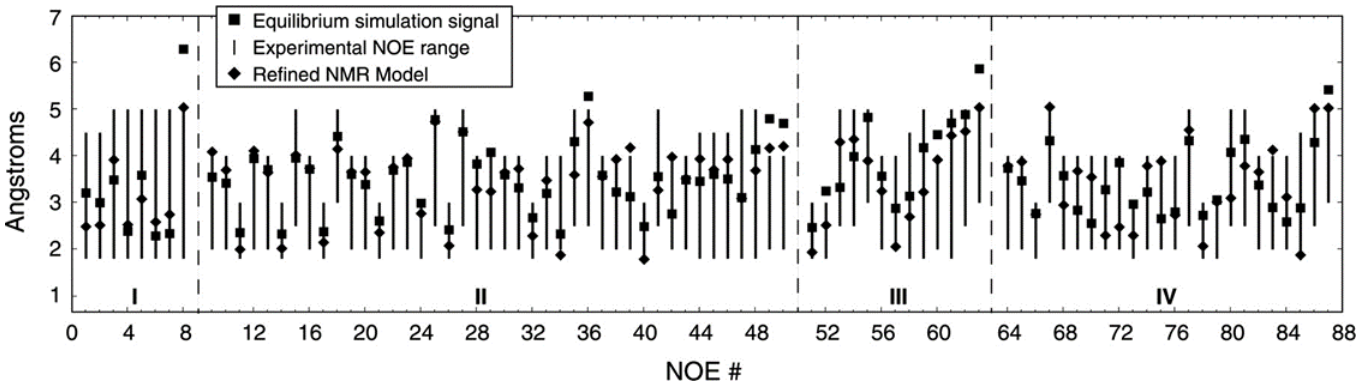
- HOESY, Heteronuclear Overhauser Effect Spectroscopy
- ROESY, Rotational Frame Nuclear Overhauser Effect Spectroscopy
- TRNOE, Transferred Nuclear Overhauser Effect
- DPFGSE-NOE, Double Pulsed Field Gradient Spin Echo NOE experiment



Selected regions from the 500-MHz two-dimensional NOE spectroscopy spectra (mixing time 200 ms) of OS derived from the LPS of 486lpsAEa (*A*) and RdlpsA486 (*B*). Cross-peaks that are characteristic are labeled. The NOE connectivities important for the Hex-HepIII-HepII region are illustrated in the structural elements.



The fastest upshot, is that the **off-diagonal peaks** indicate coupling between the two nuclei (through space of through bond), which gives greater information regarding the structure of a sample (this is the underlying approach to solution-phase NMR structure determination of proteins). See: http://pubs.acs.org/doi/abs/10.1021/ja00353a071



Simulation data are compared to the distance constraints determined from NOE cross peaks