# Slovenian NMR Centre

@ National Institute of Chemistry

vabi na **SEMINAR**:

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z naslovom:

## Instrumentation development for NMR of oriented systems and semisolid samples

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*Kratek povzetek:*
Extensive deuteration is often used to improve resolution in solid-state NMR of proteins. Substantially deuterating the sample dramatically reduces the homonuclear (1H-1H) and heteronuclear (1H-13C and 1H-15N) heteronuclear dipolar interactions. This improves resolution, reduces the magnitude of the RF decoupling needed, and enables 1H-detected experiments, even in rigid solids. However, this enhanced resolution is obtained at the cost of information loss due to the less abundant protons. Although it is not frequently applied in the context of protein structure determination, the deuterium quadrupole interaction can be used in both solid-state and solution-state NMR as a sensitive probe of local order and sample mobility. This has been productively applied to determine order and dynamics in a variety of liquid crystals, structurally interesting lipids, and biological membranes. In particular, 2H NMR has often been used to probe perturbations in deuterated membranes upon binding of peptides and proteins, or to determine the alignment of peptide bonds and planes relative to the bilayer normal in deuterated peptides. In membranes and other oriented systems, the 2H signal from bulk D2O can be used as a measure of overall orientation. Alternatively, in a deuterated protein this kind of measurement can potentially be done in the context of 2H-13C or 2H-15N correlations. In semi-solid samples with significant mobility, specific orientational and dynamic information obtained can be correlated with structure by assigning the 13C and 15N resonances from more conventional 2D and 3D experiments.
Given some attention to probe optimization, switched-angle spinning (SAS) NMR is also useful for structural studies of oriented media. SAS measures the isotropic spectrum that is needed to assign the resonances of different chemical sites, while providing valuable information that would be lost in a simple MAS experiment, all without changing the concentration of any components or the temperature. Here the dipolar couplings are scaled by simply changing the spinning axis. This approach takes advantage of the mobility of the sample, making it an asset instead of a liability. Finally, this method will enable RDC measurements and structure determination of membrane proteins, which do not assume their biologically relevant conformations in isotropic solution. In this presentation, I will describe the design, construction, and testing of SAS probes for oriented samples and a 1H/13C/2H/15N quadruple resonance MAS probe currently under development in my laboratory. Preliminary experimental results on protein and lipid samples will also be discussed.

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