

ICONS 2020

Intercontinental NMR Conference on Methods and Applications

ICONS₂₀₂₀

August 26-28, 2020

Time: 13:00-16:00 (Berlin/Paris, CEST)

Organised by:

Daniel Abergel, Gerd Buntkowsky, Konstantin Ivanov, P. K. Madhu



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Foreword

ICONS2020 is the first of a new on-line magnetic resonance conference series. It aims to cover recent topics in both NMR and EPR. The meeting is an off-shoot of the Intercontinental NMR Seminar Series that started on April 8, 2020. This seminar series enabled communication and dissemination of research ideas among the magnetic research community especially in the times of the COVID-19 pandemic. The seminar series also gave early-stage researchers an opportunity to give seminar talks and interact with colleagues from different countries. The success of the seminar series prompted us to introduce a new conference to highlight the recent research contributions from some of the leading groups in magnetic resonance. We target to reach a good cross-section of the community even despite the time-zone disparity. We sincerely hope that both the seminar series and the conference will prove to be beneficial to the magnetic resonance community and contribute to the growth of magnetic resonance science and help many of us to stay connected.

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Time/CEST/UTC	August 26 Wednesday	August 27 Thursday	August 28 Friday
13:05-13:45 11:05-11:45	Bodenhausen	Suter	Griesinger
13:45-14:25 11:45-12:25	Agarwal	Nishiyama	Felli
14:30-15:10 12:30-13:10	Polimeno	Brunner	Lesage
15:15-15:55 13:15-13:55	Polenova	Akke	Levitt

Strengths and weaknesses of dissolution DNP

Geoffrey Bodenhausen

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Although Dissolution DNP is best known for its ability to enhance the nuclear polarization by 4 to 5 orders of magnitude, it has actually spurred some unexpected discoveries. The best known is the ability to monitor the kinetics of the enzymatic conversion of pyruvate to lactate *in vivo* in healthy and cancerous cells. Many less well-known discoveries are due to the fact that the spin temperature can be made low enough (on the order of 10 mK) to violate the so-called "high temperature" approximation. (This somewhat misleading expression refers to the fact that the Taylor expansion of Boltzmann's exponential law can be truncated to keep only the linear term, while dropping quadratic and higher terms, which is legitimate for spin temperatures above, say, about 50 mK for many common nuclei in fields of a few Tesla.) When the high temperature approximation is violated in spin systems that feature some sort of spin permutation symmetry, very low spin temperatures readily allow one to obtain "imbalances" between populations belonging to different irreducible representations of the permutation group. These *population imbalances* (a term coined by Levitt) may be slow to return to thermal equilibrium, and therefore may have long lifetimes. An unexpected example is found in CD₃ groups where one would expect quadrupolar relaxation of the deuterium nuclei to be too rapid to allow any long-lived states to exist. There may be a host of similar effects that are yet to be discovered.

On the other hand, Dissolution DNP has taught us many lessons of humility and, at times, has been a source of frustration. Unlike many clever pulse sequences that can readily be taken up by any colleague or competitor, friend or foe, Dissolution DNP is too complex to be adopted by a broad community of fellow scientists. The method is very slow: in our hands, one can at best study one hyperpolarized solution every 100 minutes, since it takes time to cool down the sample to about 1.2 K after its insertion, and it takes time to allow the polarization to build up prior to dissolution. The method delivers deplorably dilute solutions: typically, a solid frozen pellet of 50 μ L is diluted in 5 mL of hot water, i.e., the material is diluted 100-fold, which in many cases implies that the advantage is reduced from four to two orders of magnitude. Furthermore, only a fraction of the resulting 5 mL solution, say 0.5 mL or 10%, can be detected in a conventional NMR spectrometer. It is not easy to select a fraction that contains a satisfactory concentration of hyperpolarized material, and the selected fraction may well contain a high concentration of radicals that are detrimental to the lifetime of the polarization. Attempts to separate these radicals by precipitation, separation into a non-miscible phase, or attachment to some porous material that can be retained in a filter have turned out to be challenging. As a result, Dissolution DNP remains limited to a handful of laboratories dispersed around the world, the insights that have been gained at considerable expense remain largely confidential, and our published work has been rarely cited, and hardly brought about any exciting intellectual debate, except for a handful of insiders. This may be due in part to the reluctance of instrument manufacturers to develop affordable commercial products for Dissolution DNP, a reluctance that can readily be explained by considerable R&D investments that are required, expensive magnets, fragile probes, tricky cryogenics, uncertainties about the lifetime of microwave equipment, high running costs for users, in particular liquid helium, unless it can be recycled, and challenging service requirements.

Fortunately, the advent of “Bullet DNP”, initiated by Benno Meier and his team [1] appears to opens up shiny new horizons: faster polarization, faster transfer, less dilution, less relaxation - it would seem that Bullet DNP promises relief to much of our misery!

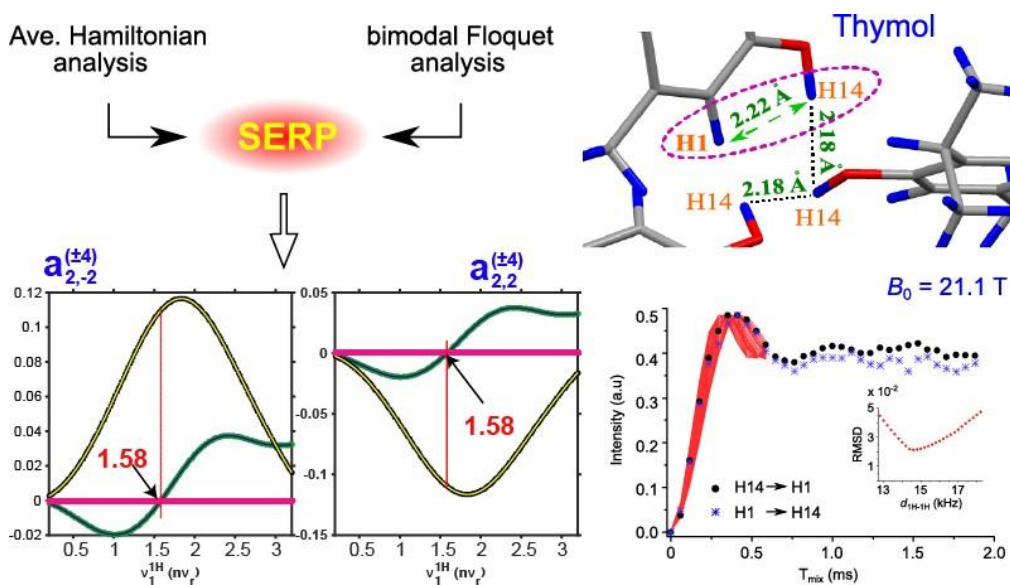
[1]. Kouril, K.; Kourilova, H.; Bartram, S.; Levitt, M. H.; Meier, B., Scalable dissolution dynamic nuclear polarization with rapid transfer of a polarized solid. Nat Commun. 2019, 10).

Accuracy of ^1H - ^1H distances measured using frequency selective recoupling and fast magic-angle spinning

Vipin Agarwal

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Solid-state NMR has become a routine technique to study a range of molecules in condensed matter. One of the challenging problems in solid-state NMR is the measurement of proton-proton distances due to a densely dipolar coupled network of proton spins. Recently, we proposed a novel method (called SERP) to measure ^1H - ^1H distances that combines phase-modulated pulses with fast magic angle spinning (*J. Phys. Chem. Lett.*, 9, 5948-5954). The estimation of proton-proton distances provides a major boost to structural characterization of small, pharmaceutical and biomolecules, given that proton positions are generally not well defined in x-ray structures.



In this presentation, we discuss a theoretical model based on bimodal Floquet theory to understand the intricacies of SERP recoupling. The modulation of the different terms of the recoupled Hamiltonian, as a function of experimental parameter can explain the origin of selective recoupling. The experimentally measured inter-proton distances were found to be approximately 10-15% shorter than those obtained from diffraction study. We elaborate on the factors that lead to short distances and conditions that will further improve the accuracy of the measured proton distances. We also show a modification to SERP that further improve the polarization transfer efficiency and selectivity. Also, some initial results on selective proton recoupling using the “C-symmetry” based sequence will be shown.

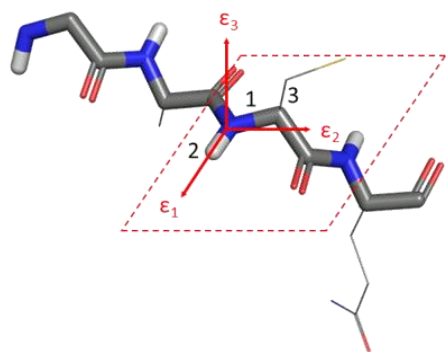
Modelling of flexible macromolecules in solution for relaxation magnetic resonance investigations

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Internal and overall dynamics of proteins are critically involved in the determination and the regulation of their physical and chemical properties, biological function and spectroscopic signatures. Examples of dynamic-controlled classes of processes are the allosteric effects in enzyme catalysis, the formation of non-specific transient encounter complexes in the protein-protein association and the regulation of molecular recognition [1]. Monitoring and describing proteins dynamics is therefore a fundamental area of investigation in modern physical chemistry. In particular the interpretation of magnetic resonance relaxation experiments require a precise description of such molecular motions, if possible within a sound theoretical framework. Here we discuss for the derivation of stochastic descriptions of flexible macromolecules in solution from atomistic models, based on the definition of a Liouville equation of



motion followed by projection operator techniques [2]. A generalized master equation is obtained, which can be recast into alternative modeling options, based on different choices of internal variables, of accuracy levels of the description of solvent effects, etc. A semi-flexible model [3] is recovered as the first, simplest approximated treatment of internal coordinates, i.e. harmonic motions, coupled to global tumbling. The computational protocol is based on *i*) energy minimization, *ii*) parametrization of the harmonic internal potential, *iii*) evaluation of the generalized diffusion tensor based on hydrodynamic modeling, *iv*) approximate solution of the stochastic equation. Case-study calculations are carried out on

a series of *n*-alanines with *n* ranging from 2 to 10. Auto/cross-correlation functions of 2nd rank Wigner matrices are compared with rigid body model calculations.

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August 26, 1515-1555 (CEST)

Fluorine MAS NMR and DNP of molecules small and large: Spin it fast!

Changmiao Guo, Manman Lu, Brent Runge, Roman Zadorozhnyi, Caitlin Quinn, Matt Fritz, Jodi Kraus, Sucharita Sarkar, Jochem Struppe, Ivan Sergeyev, Melanie Rosay, Angela M. Gronenborn, Tatyana Polenova

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Recent methodological advances in ^{19}F MAS NMR and DNP from our team will be discussed, with applications in small-molecule pharmaceuticals, microcrystalline proteins and large biological assemblies. It will be demonstrated that high MAS frequencies (40-60 kHz) are advantageous for the homo- and heteronuclear correlation experiments and the measurement of accurate interfluorine distances. With the remarkably high, up to 100-fold, signal enhancements in ^{19}F DNP MAS NMR spectra, observed in HIV-1 capsid protein assemblies, it was possible to record 2D ^{19}F - ^{13}C HETCOR spectra. These spectra contain long-range intra- and intermolecular correlations, which are not easily accessible in conventional experiments without DNP.

August 27, 1305-1345 (CEST)

Resonant excitation of nuclear spins by modulated hyperfine interaction

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Nuclear spins coupled to unpaired electrons often cover a wide frequency range; efficient excitation of all possible transitions is a challenging task, particularly if the available RF power is limited and short pulses are essential. Here, we demonstrate an excitation scheme that reduces the required RF power to zero: we only excite the electron spin, using a sequence of microwave pulses that modulate the hyperfine interaction in such a way that the nuclear spins are excited either selectively or nonselectively. We use a single nitrogen-vacancy (NV-) center in diamond to demonstrate the technique.

Probing local ^1H spin network through ^1H SQ, DQ and TQ coherences

Yusuke Nishiyama

RIKEN/JEOL RESONANCE Inc., Japan

^1H high resolution NMR spectra of rigid-solids are readily observed using very fast MAS > 70 kHz. Owing to its high natural abundance, a cluster of protons is ubiquitous and provides useful information for structural analysis through ^1H - ^1H homonuclear correlation experiments. Here we discuss various strategy and sequences to monitor local proximity of ^1H s.

2D ^1H single-quantum (SQ)/ ^1H SQ correlation is one of the straightforward approaches. In this experiment, a ^1H - ^1H mixing period is sandwiched by two ^1H SQ dimensions, giving cross peaks between two dimensions. However, ^1H - ^1H spin diffusion is largely suppressed at fast MAS conditions, making ^1H - ^1H mixing inefficient. In addition, the spin dynamics is complicated with the presence of chemical shift dispersions. To overcome these problems, zero-quantum (ZQ) mixing sequence can be used to re-introduce ^1H - ^1H dipolar coupling [1]. While ^1H SQ/ ^1H SQ correlation provide long range correlations through relayed magnetization transfer, the presence of uncorrelated peaks at the diagonal line often disturbs the spectral analysis. ^1H double-quantum (DQ)/ ^1H SQ correlation spectra, where all the peaks come from correlated ^1H pairs, are very useful building block [2, 3]. Because of high DQ filtering efficiency (20~30% of ^1H single pulse), ^1H DQ/ ^1H SQ correlation block is combinedly used with the other NMR dimensions [4, 5, 6] or used in constant time manner to improve resolution [7]. Similar to ^1H DQ/ ^1H SQ experiments, ^1H triple-quantum (TQ)/ ^1H SQ experiments are proposed to probe three-spin connectivity [8]. TQ coherence can be produced by applying DQ recoupling sequence to transversal magnetization [9, 10]. However, this procedure distributes coherence to the other odd order coherences. As a result, the TQ efficiency (~5%) is much lower than DQ, hampering its wide applications. This problem can be avoided by using three spin TQ Hamiltonian. The application of TQ Hamiltonian only produces $3N$ (N : integer) coherences, maximizing TQ efficiency. Although TQ Hamiltonian cannot be the first order, it is shown that second order average Hamiltonian can be used to design TQ recoupling sequence [11, 12] and demonstrated on ^1H adamantane and fully ^{13}C labelled samples at moderate MAS rate. We have designed ^1H TQ sequence based on this strategy and found that the TQ filtering efficiency (20~30%) can be comparable to DQ filtering efficiency [13]. The one of the practical problems of ^1H TQ/ ^1H SQ experiments is spectral complexity. The dense ^1H network results in numerous TQ/SQ correlations, making spectral analysis complicated or impossible in a worst case. In order to overcome this problem, we designed 3D ^1H TQ/ ^1H DQ/ ^1H SQ correlation experiments [14]. The TQ coherence is decomposed into DQ coherence, making the spectral interpretation easy.

We also introduce a new methodology to reveal hydrogen-bonding structure using microED and solid-state NMR [15].

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Solid-state and *in situ* NMR spectroscopy of flexible metal-organic frameworks

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NMR spectroscopy is a unique tool for studying structure, dynamics, and flexibility of metal-organic frameworks (MOFs). Adsorption processes as well as adsorption-induced structural changes, i.e., host-guest interactions, are also accessible for NMR spectroscopy. Within the present contribution, solid-state and *in situ* NMR studies of flexible MOF compounds are described [1-3].

An interesting example compound is DUT-8(Ni) (DUT: Dresden University of Technology) which undergoes a very pronounced adsorption-induced gate-pressure effect. A combination of ^{13}C cross polarization (CP) magic angle spinning (MAS) NMR spectroscopy and ^2H NMR spectroscopy is used to elucidate the structural reasons for the fact, that DUT-8(Ni) can be synthesized either as a flexible MOF with a huge change of the unit cell volume during gating transition or as a fully rigid material. Selective ^{13}C enrichment of the linker carboxylate groups [1] results in a huge sensitivity increase for these sites and enables two-dimensional NMR experiments. This provides a selective probe to study the paddle wheel unit, e.g., under the influence of different solvents. ^2H NMR is applied in order to study local motions in the framework.

In situ NMR studies of host-guest interactions are particularly powerful. Our homebuilt apparatus allows *in situ* high-pressure NMR spectroscopic studies by the application of variable gas pressures up to a relative pressure of 1 at variable temperatures down to 190 K inside the NMR spectrometer [2]. This apparatus is used in order to elucidate parameters affecting the gate opening process, e.g. by using NMR probes like ^{129}Xe or ^{13}C enriched CO_2 . This *in situ* NMR technique could for example be used [2] to characterize the xenon-induced phase-transitions in the recently discovered pressure-amplifying framework materials DUT-49 and DUT-50 with their unique negative gas adsorption transitions [4,5]. Xenon is capable of inducing mesopore contraction at 200 K.

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August 27, 1515-1555 (CEST)

Protein dynamics in ligand binding and allostery

Mikael Akke

Biophysical Chemistry, Lund University, Sweden

Protein conformational dynamics play important roles in ligand binding and allostery with regard to both kinetics and thermodynamics.

I will present recent results highlighting applications of NMR relaxation methods to study allosteric signaling in the glucocorticoid receptor ligand-binding domain (1).

In addition, I will describe a method for robust measurement of relaxation rate constants from non-uniformly sampled data, acquired using the accordion approach (2). This method shortens the total experiment time by an order of magnitude, making it possible to measure relaxation rate constants on samples of low-sensitivity or limited life-time. As an example, we have measured relaxation rates on samples resulting from isothermal titration calorimetry experiments, which typically involve much lower protein concentrations than those used in NMR.

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2. Carlström G, Elvander F, Swärd J, Jakobsson A, Akke M (2019) "Rapid NMR relaxation measurements using optimal nonuniform sampling of multidimensional accordion data analyzed by a sparse reconstruction method" *J. Phys. Chem. A* 123, 5718-5723.

Fast kinetics in proteins and anisotropic parameters from field induced alignment

S. Prathihar¹, N. Karschin¹, Nilamoni Nath¹, J.C. Fuentes¹, Sahithya Phani Babu Vemulapalli¹, K. Wolkenstein^{1,2}, D. Lee^{1,3}, S. Becker¹, and C. Griesinger^{1,4}

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Kinetics of protein dynamics will be discussed on examples of folded on unfolded proteins (1). We have developed high power relaxation methods in order to measure kinetics down to life times of a few 100 ns.

A second project is on the determination of configuration of small molecules. Here approaches for the analysis of minute quantities (some 10s of micrograms) of natural compounds using deuterated gels will be presented (2). A second approach using self alignment in strong magnetic fields will be presented as well (3).

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- (3) **Karschin, Niels; Wolkenstein, Klaus; Griesinger, Christian** "Magnetically Induced Alignment of Natural Products for Stereochemical Structure Determination via NMR" *Angew. Chem. Int. Ed.* DOI: 10.1002/anie.202004881 (2020)

August 28, 1345-1425 (CEST)

Intrinsically disordered proteins by NMR: What can ^{13}C direct detection can tell us?

Isabella C. Felli

CERM and Department of Chemistry “Ugo Schiff”, University of Florence, Italy

A variety of intrinsically disordered proteins (IDPs) or protein regions (IDRs) with important, yet unexplored, functional and regulatory roles is emerging. The contribution of exclusively heteronuclear NMR experiments based on ^{13}C direct detection to access atomic resolution structural and dynamic information on IDPs/IDRs will be discussed. Several examples of particular features or motives that often occur in IDPs/IDRs will be presented, revealing novel structural and dynamic modules not yet described in the PDB.

August 28, 1430-1510 (CEST)

DNP enhanced solid-state NMR spectroscopy: Recent developments at high magnetic field and fast MAS

Anne Lesage

Institut des Sciences Analytiques, University of Lyon, CNRS, ENS Lyon, France

Dynamic Nuclear Polarization (DNP) has recently emerged as an efficient, robust and versatile approach to circumvent the sensitivity limitation of Magic Angle Spinning solid-state NMR spectroscopy, allowing signal enhancements by several orders of magnitude. This technique, originally developed at low magnetic fields, has been shown to be applicable at high fields, opening new avenues to tackle materials and molecular substrates that were previously inaccessible to solid-state NMR studies. In this presentation we will present some new experimental developments and applications. In particular we will discuss recent results from high field (800 MHz) and fast MAS (~ 40 kHz) DNP NMR. The role of new, highly efficient polarization sources will be discussed.

August 28, 1515-1555 (CEST)

Scientific amusements with fullerenes and endofullerenes, using NMR and other spectroscopies

Malcolm H. Levitt

Department of Chemistry, University of Southampton, SO 17 1BJ, UK

Topics selected from:

- (1) Extra peaks in the ^{13}C NMR of fullerenes and endofullerenes due to $^{13}\text{C}_2$ isotopomers, and second-moment calculations of large spin systems.
- (2) The first observation of a J-coupling involving ^3He , and between species with no hint of a chemical bond.
- (3) Endohedral chemical shifts and their temperature and pressure dependence.
- (4) Ortho-para conversion of water-endofullerene observed by NMR and pulsed THz spectroscopy. A “free induction decay” with femtosecond time resolution.
- (5) Measuring the interaction of a noble gas atom with a carbon surface, using NMR, terahertz spectroscopy and neutron scattering.