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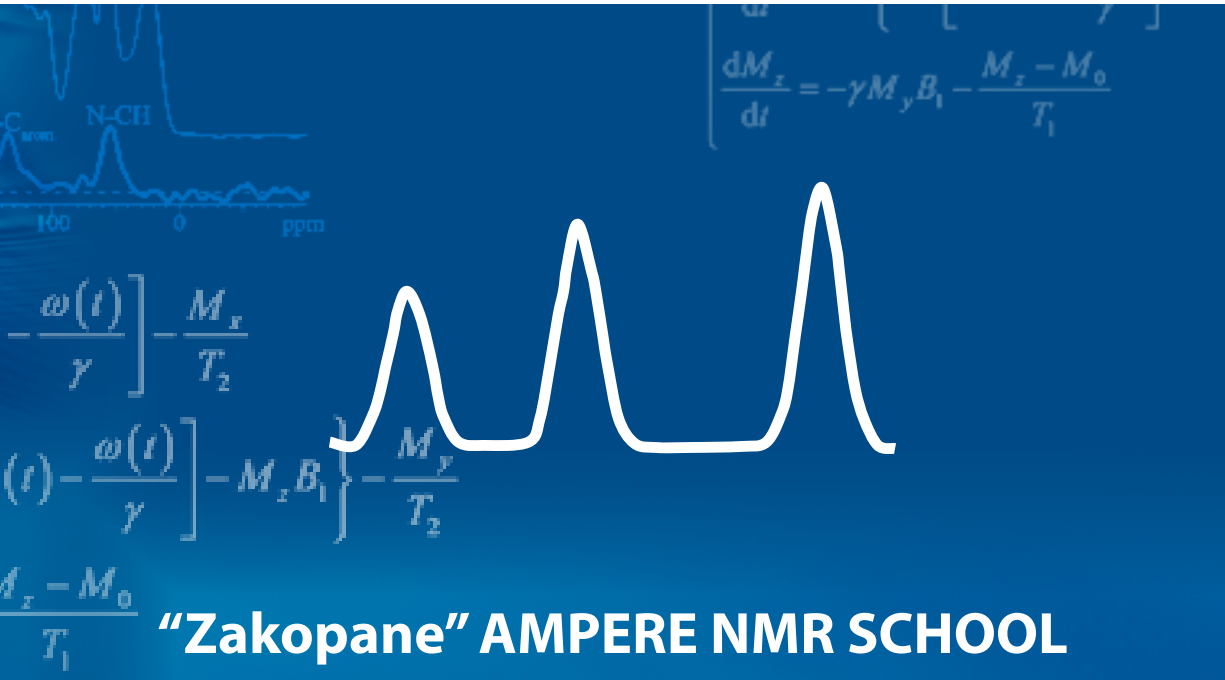

"Zakopane" AMPERE NMR SCHOOL

virtual event

BOOK OF ABSTRACTS

21st – 23rd June 2021
POZNAŃ, POLAND

Edited by S. Jurga
Adam Mickiewicz University in Poznań
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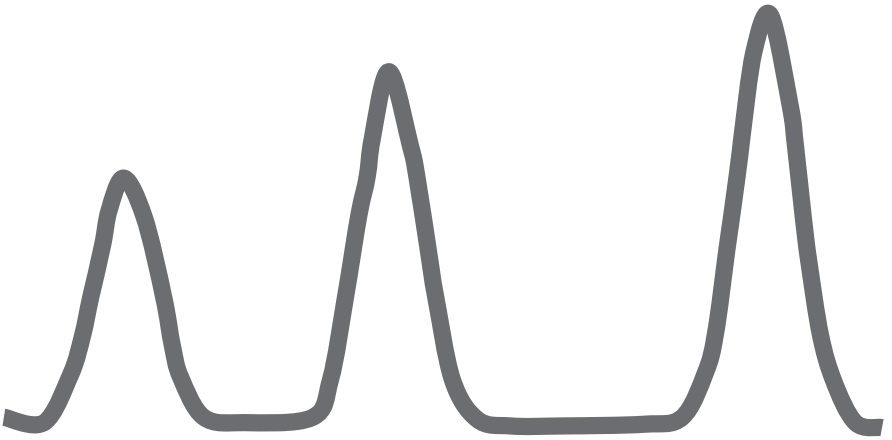

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"Zakopane" AMPERE NMR SCHOOL
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organized by

NanoBioMedical Centre
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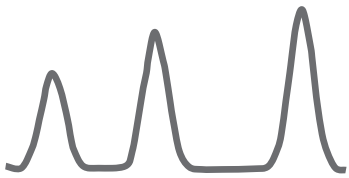

AMPERE **NMR** School

Molecular Dynamics by NMR for Materials Testing: Relaxation, Exchange and MRI

Bernhard Blümich

Formerly RWTH Aachen University, Germany

Relaxation and exchange are NMR phenomena, which along with MRI can serve to probe molecular dynamics on different time scales ranging from the inverse Larmor frequency to T_1. This lecture is an excursion into NMR studies of molecular motion for materials characterization covering different methodical concepts illustrated with examples of practical applications.

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BASICS OF MRI AND RESEARCH ON FAST FIELD-CYCLING MRI

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Mary J. MacLeod, P. James Ross and Robert Stormont

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MRI uses magnetic field gradients to encode spatial information into NMR signals. In frequency-encoding, the NMR signal is recorded while a field gradient is applied. Since the magnetic field varies with position along the gradient direction (e.g. X), Larmor frequency is a function of position, so the detected signal contains a range of frequencies; analysing the frequency content generates a one-dimensional projection of the water-distribution within the patient. Phase-encoding is employed in the second in-plane dimension (e.g. Y); here, the gradient is pulsed on and off prior to measurement of the signal, altering the phase of the NMR signal as a function of position. The image slice is defined using selective-excitation, in which the excitation 90° radiofrequency pulse is shaped (typically a sinc function) and is applied in the presence of a field gradient perpendicular to the slice plane (e.g. along Z for a transaxial X-Y slice). An excellent primer textbook on MRI has been published by McRobbie et al. [1].

During the last decade, our laboratory has focused on the development of Fast Field-Cycling Magnetic Resonance Imaging (FFC-MRI). By switching field strength during an experiment, this technique exploits the variation of T_1 with magnetic field (T_1-dispersion), with the aim of increasing the diagnostic potential of MRI [2,3]. FFC-MRI aims to obtain spatially-resolved T_1-dispersion data, by collecting images at a wide range of evolution field strengths. In our lab we have built a range of FFC-MRI equipment, including two whole-body human sized scanners, operating at detection fields of 0.06 T [4] and 0.2 T [5]. The recently-completed 0.2 T FFC-MRI system uses a single resistive magnet, composed of three coaxial coils.

We have shown that FFC methods can detect changes in human cartilage induced by osteoarthritis [6]. Experiments on resected tissues from breast cancer patients have shown significant differences in the dispersion curves between normal and diseased tissues [7]. We have performed *in vivo* studies on patients with acute ischaemic stroke; FFC-MRI images exhibited increased intensity in stroke-affected regions, with maximum contrast typically at the lowest field used (0.2 mT) [8]. We have also begun studies on patients with brain cancer and patients with breast cancer. All human studies were conducted following approval of the relevant Research Ethics Committees and with informed consent. Work to improve the hardware and software is ongoing, including the implementation of improved RF coils [9].

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 668119 (project "IDentIFY"). It also benefitted from COST Action CA15209, "European Network on NMR Relaxometry".

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The higher, the better?! The promises and perks of ultra-high field MRI at 22.3 T

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Nuclear magnetic resonance imaging (MRI) is well-known for non-destructively visualizing biological specimens, ranging from clinical, whole-body applications to microscopic imaging. Besides anatomical information, localized magnetic resonance spectroscopy (MRS) on biological specimens yields valuable information on the distribution of metabolites or storage compounds. As with all NMR-based techniques, MRI and localized MRS suffer from low sensitivities, leading to low spatial resolutions, long experiment times and high detection limits for chemical compounds and metabolites. Approaches to overcome this limitation are to increase the main magnetic fields strengths B_0 and to optimize the detector sensitivity.

In this talk, we will discuss examples of the highlights and advantages of ultra-high field MRI. Using a 22.3 T spectrometer (f(^{1}H) 950 MHz, unmr-nl spectrometer) and a custom-built detection coil, we show nominal spatial resolutions of (5.5 μm)^3 [1] on polymer microbeads in water and (7 μm)^3 on a plant root specimen [2]. Furthermore, we find that a reduction in experiment time of factor 24 could be achieved at identical image resolutions when increasing the main magnetic field strength from 14.1 T to 22.3 T [1].

Additionally, we will look into the challenges of ultra-high field MRI and MRS, such as increasing susceptibility mismatches and a need for modified sample preparation. Iron content [3] and air spaces [2] can cause susceptibility mismatches, leading to image artefacts. At last, an outlook and future perspectives for ultra-high field MRI and MRS will be discussed.

Acknowledgements

We thank the unmr-nl consortium and the technical support at the NMR-centers of Utrecht University, Leiden University and MAGNEFY at Wageningen University & Research for their support.

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DIFFUSION-RELAXATION CORRELATION MRI

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Brain tissues are heterogeneous anisotropic materials where each millimeter-scale voxel contains water in multiple environments with different cellular structures and chemical compositions, which are imprinted on the diffusion tensors **D** and relaxation rates R_1 and R_2 observable with MRI. In order to characterize the intra-voxel heterogeneity of tissue environments, we use multidimensional **D**-R_1-R_2 distributions [1]. Images are acquired with tensor-valued diffusion encoding [2] combined with variable echo [3] and repetition times [4], which upon Monte Carlo data inversion gives nonparametric distributions with dimensions corresponding to relevant microstructural properties, see Figure 1. The rich details that are inaccessible with conventional MRI may prove essential to correctly delineate tumors in voxels containing multiple tissue types, as well as to classify brain tumors and inform clinical decisions about targeted therapies preserving healthy tissues.

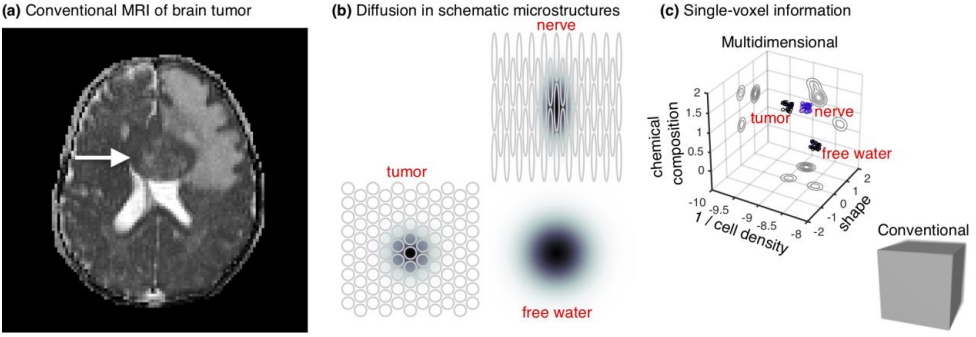

FIGURE 1. MRI of brain tumor and the concept of microscopic tissue heterogeneity. (a) Conventional apparent diffusion coefficient map where the tumor (indicated with arrow) appears slightly darker than the surrounding tissues. **(b)** Schematic tissue microstructures with water diffusion patterns shown as "ink stains" – corresponding to diffusion tensors **D** – with sizes, shapes, and orientations determined by the underlying cell structure. The shown structures correspond to dense tumor tissue (bottom left), free water in necrotic tissue (bottom right), and healthy nerve fiber (top right). **(c)** Zoom-in on a millimeter-scale MRI voxel. Whereas conventional MRI gives a single grayscale value for the entire voxel, our multidimensional methods enable resolution of sub-voxel tissue regions based on the local cell density and shape, imprinted on **D**, as well as the chemical composition of the water phase determining the relaxation rates R_1 and R_2. (Figure adapted from Refs. [5] and [6].)

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NMR STUDIES OF COMPLEX SAMPLES USING COMPACT INSTRUMENTS

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The usage of compact NMR instruments has steadily increased in the last decade. This comes to no surprise given that capital- and operational expenditures are about one magnitude lower compared to conventional high-field devices and no specialized personell is required for operation. In addition, the compact spectrometers themselves are continuously being improved towards stronger magnetic field strengths and better spectral resolution. However, when compared to the conventional high-field counterpart, benchtop NMR spectrometers are still inferior in terms of signal-to-noise-ratio and resolution. This circumstance manifests itself in the spectrum as broad and mostly overlapping peaks, which is an impediment for the analysis of complex molecules and complex mixtures.

In this presentation, three scenarios are demonstrated which analyse complex samples utilizing benchtop NMR spectrometers. Firstly, the identification and quantification of large plasticizer molecules found in polyvinyl chloride is shown. The proposed method abolishes long measurement times and deuterated solvents for a rapid and simple analysis, while keeping the concentratinonal limit of quantification low. Secondly, fuels are being examined to learn more about the formation of deposit. These deposits occur in mixtures of petrol- and bio-based oils, which are complex mixtures of natural products themselves. Lastly, a simple but versatile setup is presented, which allows to study highly pressurized samples. This setup was used to study gas-mixtures, gas-solid-, and gas-liquid interactions.

These three scenarios show different means of encountering the utilization of compact NMR spectrometers. Whether it be simplifying an analytical procedure so that any scientist could perform it, concentrating on peak clusters rather than fine structure or employing benchtop spectrometers in an environment not suitable for high-field magnets – there are many niches and potential applications for compact devices.

INSIGHT INTO THE DETAILS OF MOLECULAR TRANSLATION DYNAMICS IN LIQUIDS BY NMR GRADIENT SPIN ECHO METHOD

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Molecular dynamics in simple liquids[1] and binary mixtures of water and glycerol[2] was studied by measuring the spectrum of the velocity auto-correlation in the frequency range from $0.05-10$ kHz by using the NMR method of modulated gradient spin echo. The results of the measurements highlight the diversity of the diffusion signature at short spin trajectories, which proves the heterogeneity of molecular motion due to the motion in the micro-vortexes of hydrodynamic fluctuation, which is especially pronounced for free water and mixtures with low glycerol content. While at longer time intervals, and thus with longer trajectories, heterogeneity is averaged out, giving rise to a spectrum which is explained as a combination of molecular self-diffusion and eddy diffusion within the vortexes of hydrodynamic fluctuations. As concentration of glycerol increases above 10vol%, a new feature of spectrum appears due to interaction of water molecules with the clusters formed around hydrophilic glycerol molecules. New spectrum exposes a rate thickening of molecular friction, according to Einstein-Smoluchowski-Kubo formula, which inhibits rapid molecular motions and creates the conditions for a slow process of spontaneously folding of disordered poly-peptides into biologically active protein molecules when immersed in such a mixture. Measurement results on simple liquids can be explained reasonably well by a $t^{-3/2}$ long time tail decay only for non-polar liquid toluene, while the spectra of polar liquids, such as ethanol, water and pure glycerol, are more congruent with the model of diffusion of particles temporarily trapped in potential wells created by their neighbors.

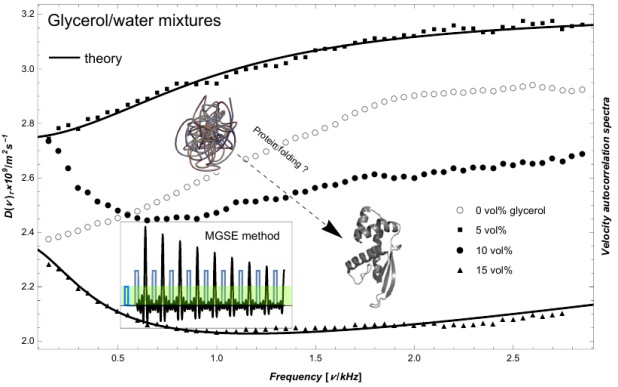

FIGURE. The dots show the velocity autocorrelation spectrum of glycerol/water mixtures obtained by the modulated gradient spin echo method, where the fitting curves for 5vol% and for 15 vol% of glycerol content correspond well to the equations for harmonic coupling of diffusing particles as derived in the reference [2].

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ULTRAFAST MULTIDIMENSIONAL RELAXATION AND DIFFUSION MEASUREMENTS

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NMR is one of the very few methods for measuring molecular self-diffusion coefficient without an invasive tag, even inside opaque samples. Relaxation experiments, in turn, reveal the rates of recovery of the initially perturbed magnetization to the thermal equilibrium, mainly due to random rotational motion of molecules. Diffusion and relaxation experiments, also called Laplace NMR experiments, provide versatile information about the dynamics of substances. Furthermore, they offer chemical resolution not available in the traditional NMR spectra. [1]

The resolution and information content of the Laplace NMR experiments can be improved significantly by a multidimensional approach. The approach allows one to correlate relaxation and diffusion parameters. Furthermore, it enables one to investigate the chemical or physical exchange even in the case when the exchanging sites are not resolved in the spectrum, via the relaxation or diffusion contrast. [1]

The multidimensional approach is, however, slow, as the experiment has to be repeated many times with incremented evolution time. In the ultrafast Laplace NMR method, the various evolution times are encoded into layers of the sample. [2-8] The spatial encoding was first introduced by Frydman in the context of ultrafast NMR spectroscopy. [9] The method enables one to measure multidimensional Laplace NMR data in a single scan, reducing the experiment time by one to three orders of magnitude. In addition, the single-scan approach facilitates significantly the use of modern hyperpolarization methods to increase the sensitivity of the experiment by several orders of magnitude. [3-5]

This lecture describes the principles of conventional and ultrafast multidimensional Laplace NMR and highlights its applications in various disciplines, including those using low-field, mobile, single-sided NMR instruments.

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MEASURING NMR RELAXATION TIMES – WHAT CAN POSSIBLY GO WRONG!?

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Measuring relaxation times is probably one of the first experiments that one learns in a laboratory course. For many research topics in physics, they are among the most relevant information to be acquired; but also in chemistry or medicine, the knowledge of relaxation times is required for optimal experimental design or for choosing the proper imaging contrast.

In this tutorial, some common mistakes shall be listed which are experienced by the beginner, but also by seasoned experimenters. While some may be rather obvious, there are also more subtle influences to relaxation times T_1, T_2 and $T_{1\rho}$.
In the first category, it will be discussed what can go wrong in the experiment itself – this addresses hardware and software issues, but also fundamental problems with the application of pulses.
Secondly, the wide complex of data fitting is addressed, easily the most difficult field with a range of methods and strategies used for fitting anything in between single exponentials and broad distributions of relaxation times (or, in fact, any parameter).
On the other hand, there are a number of physical and chemical reasons why the measured relaxation times are not the ones one actually wants to determine – exchange of any kind, or simply the presence of air in a liquid sample, can shorten a relaxation time by several orders of magnitude, just as the influence of the sample's magnetization itself in a process called radiation damping.
Finally, it is worth remembering that "the relaxation time" of a sample does not exist, any measured quantity should always be accompanied by the experimental parameters, most importantly the magnetic field strength since most "interesting" relaxation processes strongly depend on the Larmor frequency.

Why are some nuclei non-spherical and what does that have to do with spin?

Ilya Kuprov

University of Southampton, United Kingdom

Nuclear quadrupole moments are an electrical phenomenon – a non-uniform charge distribution inside the nucleus creates a quadrupole moment which interacts with electric field gradients. A question that is rarely well explained is about how exactly this (electrical) interaction ends up creating a term in the (magnetic) spin Hamiltonian. Chemists are rarely taught nuclear structure to begin with.

This tutorial lecture will give a brief overview of the theory of nuclear structure, of the processes that lead to the emergence of nuclear "spin" and magnetic moment, and of the mechanism that connects the direction of nuclear magnetic moment to the principal axis frame of the nuclear electric quadrupole. These processes form the foundation of nuclear quadrupole resonance which is a major sub-field of NMR.

Residual Line Width in MAS Solid-State NMR

Matthias Ernst[a]

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Magic-angle spinning is routinely used to average anisotropic interactions in solid-state NMR. Due to the fact, that the Hamiltonian of a strongly-coupled spin system does not commute with itself at different time points during the rotation, second-order and higher-order terms lead to a residual line broadening in the observed resonances. Additional truncation of the residual broadening due to isotropic chemical-shift differences can be observed. We analyze the residual line broadening in coupled proton spin systems based on theoretical calculations of effective Hamiltonians up to third order using Floquet theory and compare these results to numerically obtained effective Hamiltonians in small spin systems. We show that at spinning frequencies beyond 50 kHz, second-order terms dominate the residual line width leading to a $1/\omega_r$ dependence of the second moment which we use to characterize the line width. However, chemical-shift truncation leads to a partial ω_r^{-2} dependence of the line width which looks as if third-order effective Hamiltonian terms are contributing significantly. We show that second-order contributions not only broaden the line but also lead to a shift of the center of gravity of the line. Experimental data reveals such spinning-frequency dependent line shifts in proton spectra in model substances that can be explained by line shifts induced by the second-order dipolar Hamiltonian.

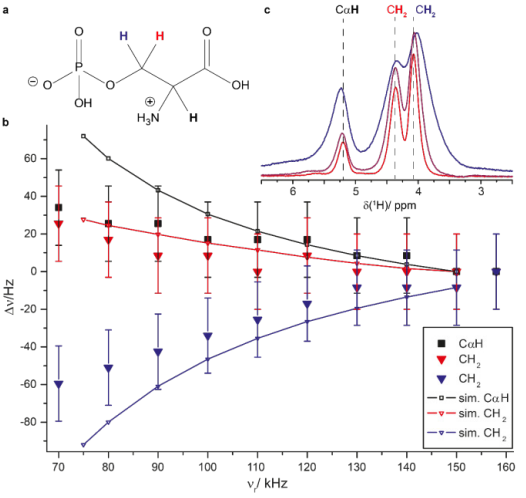

Fig. 1: Experimentally measured spinning-frequency dependent line position of the protons in ortho-phospho-L-serine for H_α and the CH_2 protons between 70 and 160 kHz MAS (solid lines). The filled symbols show the simulated line shifts as a function of the MAS frequency for a simple model including only the couplings and the isotropic chemical shifts of the three spins. This very simple model agrees quite well with the measured shifts.

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SOLID-STATE NMR OF QUADRUPOLAR NUCLEI AND THEIR NEIGHBORS

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Quadrupolar nuclei with spin $I \geq 1$, such as ^{17}O and ^{27}Al with $I = 5/2$ or ^{11}B and ^{23}Na with $I = 3/2$, represent about two thirds of stable NMR-active nuclei and are present in a broad range of materials. Nevertheless, the solid-state NMR of these nuclei in solids remains often challenging because of their large the density matrix and the large anisotropic quadrupolar interaction, which broadens the NMR spectra and complicates the spin dynamics. As a result, many techniques developped for spin-1/2 isotopes, such as cross-polarization under magic-angle spinning (CPMAS), lack of robustness and efficiency for quadrupolar nuclei.

This lecture will present a brief overview of the main techniques used to improve the resolution and the sensitivity of NMR spectra of quadrupolar nuclei. In particular, multiple-quantum magic-angle spinning (MQMAS) experiments can be employed to remove the broadening of the signals by quadrupolar interaction. The sensitivity for the detection of quadrupolar nuclei can be improved by the acquisition of a train of echoes using Carr-Purcell Meiboom-Gill (CPMG) sequence as well as population transfers between the different energy levels of the quadrupolar nucleus.

More recently dynamic nuclear polarization (DNP) under MAS has been applied to enhance the sensitivity for the detection of quadrupolar nuclei. This technique has notably benefited from the development of efficient pulse sequences to transfer the DNP-enhanced ^{1}H polarization to quadrupolar nuclei, which have allowed the NMR observation of insensitive quadrupolar nuclei, such as ^{17}O, ^{47,49}Ti or ^{67}Zn, near surfaces [1-3]. We have also developped efficient techniques to probe homo- and hetero-nuclear proximities involving quadrupolar nuclei [4-5]. These methods have provided novel insights into the structure of materials and heterogeneous catalysts. New prospects open by the advent of 1.2 and 1.5 GHz NMR spectrometers will also be discussed.

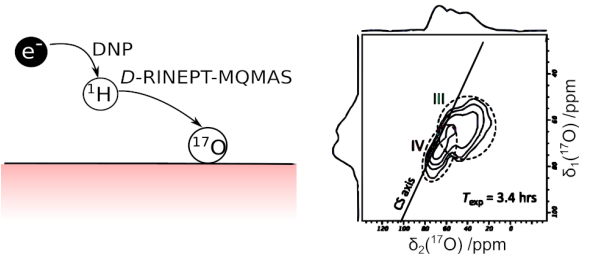

FIGURE 1. DNP-enhanced high-resolution MQMAS spectrum of ^{17}O nuclei near the surface of γ-alumina.

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HIGH-ENTROPY ALLOYS

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Within the past decade, a new approach to metallic alloys design with multiple principal elements in equi-atomic concentrations, termed high-entropy alloys (HEAs), has been proposed [1]. According to this concept, high entropy of mixing can stabilize disordered solid solution phases with simple structures like a body-centered cubic (bcc), a face-centered cubic (fcc) and a hexagonal close-packed (hcp) with small unit cells, in competition with crystalline intermetallic phases that often contain structurally complex giant unit cells, phase-segregated mixtures and amorphous (glassy) structures. In order to achieve high entropy of mixing, the alloys must be composed of five or more (up to thirteen) major elements in similar concentrations, ranging from 5 to 35 at. % for each element, but do not contain any principal element whose concentration exceeds 50 at. %. The HEA structure is characterized by a crystal lattice with an exceedingly high chemical (substitutional) disorder, so that a HEA can be conveniently termed as a "*metallic glass on a crystal lattice*", sharing simultaneously the properties of crystalline and amorphous materials. Examples of HEAs are alloys derived within the systems Al-Si-Co-Cr-Cu-Fe-Mn-Ni, W-Ta-Nb-Hf-Zr-Ti-Mo-V, and Gd-Tb-Dy-Ho-Er-Tm-Lu-Y.

Most existing studies of HEAs focus on the formation, stability and the relationship between the phase, microstructure and mechanical properties. It was demonstrated that HEAs exhibit enhanced mechanical properties like high hardness and solid-solution strengthening. Physical properties of the HEAs remain largely unexplored. It was demonstrated [2,3] that HEAs constitute a unique class of multi-elemental superconductors, the behavior of which departs from the conventional BCS superconductivity. Ferromagnetic HEAs were found to be practically ideally magnetically soft materials [4]. Such materials can be used in transformers, motors, generators, magnetocaloric refrigerators and other electromagnetic machinery, where energy losses in alternating magnetization-demagnetization cycling must be brought to minimum. HEAs with a hexagonal structure were discovered in the lanthanide series Y-Ce-Gd-Tb-Ho-Er-Tm-Lu, which show richness of magnetic phases in the temperature–magnetic field phase diagrams, comprising helical antiferromagnetic phases (both incommensurable and commensurable with the crystal lattice), ferromagnetic phases, metamagnetic phases and exotic magnetic phases with long-range ordered moments [5].

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THE KINETICS OF PROTON TRANSPORT AND THERMAL PROCESSES IN ANHYDROUS NANOCOMPOSITE PROTON CONDUCTOR BASED ON CELLULOSE

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Understanding, developing, and applying knowledge in different fields leads to overcoming existing problems, increasing the standard of living, dealing with pollution and intoxication of space in which we live is the essence of sustainable science. To make a change and efficiently in long perspective protect the Earth we need the energy – power source, which will be clean and sustainable itself. Searching for a new concept of creating sustainable materials for the power sources, we have turned to almost the most abundant and inexhaustible material on our planet – cellulose, using it as a base element of new nanocomposite materials designed for the solid proton conductor membranes in fuel cells. Cellulose, especially in its nanocrystal form, has received significant interest due to its mechanical, optical, chemical, and rheological properties. Moreover, its surface can be functionalized to meet various challenging requirements, such as the development of high-performance nanocomposite solid proton conductors. Using different heterocyclic molecules as functional groups to ensure the high proton conductivity of such material allows to work in anhydrous conditions. The high conductivity, which will be comparable or higher than commercially available Nafion®, is the prerequisite. However, the thermal properties and stability of the nanocomposite limit usability in many applications. Thus become one of the critical factors in designed these materials. Our study investigates the thermal properties and kinetics of thermal processes acting in the proposed nanocomposite proton conductor. The study of molecular dynamics with the SS MAS NMR technique reveals the mechanism of proton conduction. The combined experimental approach of thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) allowed defining the activation energies of the decomposition stages. The study of the evolution of the thermal processes with the conversion degree allowed determining the lifetime of the nanocomposite at various external thermic conditions. Based on the obtained results, some suggestions in the "bottom-up" process of synthesizing CNC-based nanocomposite proton conductors were made to increase the overall performance of the designed material.[1-3]

Acknowledgments
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NMR STUDIES OF POLYMER GEL ELECTROLYTES

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The need for green energy is stimulating the development of new materials for energy storage systems. This includes the search for better electrolytes for applications in batteries and supercapacitors. Polymer electrolytes (a combination of polymer and salt) have been introduced to overcome safety issues of liquid electrolytes, however, at the cost of low conductivity [1]. To overcome this disadvantage, a polymer and a liquid electrolyte solution can be combined to form a polymer gel electrolyte (or gel polymer electrolyte). These electrolyte materials exhibit higher conductivity than solid polymer electrolytes but suffer from insufficient mechanical stability. Therefore, inorganic fillers (often nano-sized) are added, which serve a twofold purpose: they enhance mechanical stability and increase conductivity (by suppressing polymer crystallization). In addition, they can be used to tune the electromagnetic properties, for example, to obtain shielding materials against electromagnetic interference [2]. More recently, ionic liquids have been used as solvent for the salt. Altogether, polymer gel electrolytes are complex multi-component systems, and a better understanding of these systems at the molecular level will provide guidelines for optimizing the formulations.

Multi-nuclear NMR can be employed in many ways to investigate polymer gel electrolytes. Pulsed-field gradient NMR can be used to selectively study the diffusion of ions. Most systems studied to date contain lithium cations, the counterions of the inorganic salt and possibly the ions of an ionic liquid, which may contain ^{1}H, ^{19}F, ^{11}B or other NMR nuclei. The local molecular mobility of all components (polymer, salt ions, solvent (ions), filler materials) is accessible via line shapes and relaxometry. In this contribution, examples of NMR investigations on polymer gel electrolytes will be reviewed, with a focus on our investigations of polymer gel electrolytes which are based on the random copolymer poly(vinylidene fluoride co-hexafluoropopylene) (PVdF-HFP) and swollen with a salt-containing ionic liquid. Different fillers are added to optimize conductivity [3] or electromagnetic shielding effectiveness [2].

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CONFORMATIONAL AND AGGREGATIONAL BEHAVIOR OF SOME SURFACTANTS IN AQUEOUS SOLUTIONS BY NMR OF 1H AND 13C NUCLEI

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Surfactants belong to actual chemical products, which are consumed in a large amount and find numerous applications in various areas. Amino-acid based surfactants attract an increasing interest, mainly because of their environmentally friendly properties. In comparison to conventional surfactants they are characterized in particular by a fast biodegradation and excellent antimicrobial and antifungal activities. Among them one of the most commonly used is sodium N-lauroyl sarcosinate (SLS).

To investigate the chemical and physical properties of surfactant solutions, a great number of experimental and theoretical methods have been used. Among them a special place can be assigned to nuclear magnetic resonance methods (used in the present study): NMR spectroscopy, NMR relaxometry, NMR diffusometry. Despite numerous studies of the properties of SLS in various systems, there are currently many questions that require clarification, in particular it concerns the value of the critical micelle concentration (CMC), conformational transitions in solutions and mixtures, local mobility of individual segments of amphiphilic molecules, etc. For example, as reported in the literature, the value of CMC for SLS lies in the range between 0.009 and 0.014 M, depending on the used method.

From the spectra it was possible to estimate the time scale of the internal reorientations of the COO-group at the room temperature: 3÷5 ms. There is a relatively fast exchange of the SLS molecules between the monomer and micellar states: the exchange time is much less than 1 ms. Modern NMR spectrometers allow the measurements of relaxation rates for resolved spectral lines belonging to different molecular groups and, therefore, the investigation the local molecular mobility. For example, it was possible to obtain for the set of CH_2 groups the correlation time of $\tau_c \sim 10^{-10}$ s. The bigger values of spin-relaxation times for the CH_3 groups indicate a higher mobility of these groups. Using the NMR method with a pulsed magnetic field gradient, the self-diffusion coefficients of surfactant molecules in the monomeric and micellar states were measured. The obtained data allowed us to refine the CMC values and develop a new method for estimating the decrease in the concentration of monomers with an increase in the total concentration of surfactants.

In practical applications the surfactant mixtures are often used because surfactants in mixtures exhibit new properties. In particular, mixtures of cationic and anionic surfactants are of interest for research. In the present study, in addition to SLS the dodecyltrimethylammonium bromide (DTAB) was chosen as the starting point. Data indicating the formation of mixed micelles in SLS+DTAB systems have been obtained. As a hypothesis, we suggest the electrostatic binding of molecules in the SLS+DTAB systems, which manifests itself in the broadening of the spectral lines for certain concentration regions. The spin-lattice relaxation times of ^1H and ^{13}C nuclei have been measured. Based on the relaxation of 13C, the reorientation times of the molecules are calculated. The bigger correlation times were observed for some molecular groups that correlate with the broadening of lines in the ^1H spectrum for these groups.

Carbon- and proton-detected solid-state NMR sequential assignments and applications to fibrils and membrane proteins

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Sequential assignments are the basis for structure determination. While carbon-detected spectrocopy is the classical approach, proton detection has been developed in the last years and show substantially increased sensitivity. We will show assignment experiments and strategies for the two different appraoches, and give examples for applications.

Fast MAS and Biomolecules

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Progress in NMR in general and in biomolecular applications in particular is driven by increasing magnetic-field strengths leading to improved resolution and sensitivity of the NMR spectra. Recently, persistent superconducting magnets at a magnetic field strength (magnetic induction) of 28.2 T corresponding to 1200 MHz proton resonance frequency became commercially available. We present a collection of high-field NMR spectra of a variety of proteins, including molecular machines, membrane proteins, viral capsids, fibrils and large molecular assemblies. Both carbon-13 and proton-detected experiments are discussed

Proton detection and fast MAS requires small rotor diameters and correspondingly small sample amounts in the order of 100 picoliters, limiting the signal-to-noise ratio. The limitations encountered depend on the linewidths and coherence lifetimes and will be discussed.

NMR STUDIES OF SOLID NANOCRYSTALLINE CELLULOSE-BASED PROTON CONDUCTORS

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In recent years, there has been particular interest in the development of biodegradable and biocompatible natural polymers that could act as solid polymer electrolytes. The advantages they offer are high abundance, biocompatibility, biodegradability, and cost-effectiveness. However, the conductivity of biopolymers is very low and technically they are not electric conductors. Therefore, different approaches have been proposed that have improved their ambient conductivity: for example the blending of two polymers, using a variety of salts or the nitrogen-containing heterocycles.

In our group, we focused on synthesizing a proton-conducting polymeric material in which nanocrystalline cellulose (CNC) acts as the host matrix and imidazole (Im) or 1*H*-1,2,3 triazole (Tri) act as a proton solvent.[1-4] Cellulose with heterocycles can be an alternative material for membranes in the intermediate temperature range of 100-200 °C because the amphoteric nitrogen-based heterocycles, attached to cellulose chains, do not evaporate from the fuel cell at high temperatures, unlike water in the classic Nafion. The physicochemical properties of the composites were determined by various experimental methods. It has been established that the proton conduction takes place under anhydrous conditions up to 170 ºC and the highest value of conductivity at 160 ºC is about 10^{-1}S/m and 10^{-4}S/m for CNC-Im and CNC-Tri, respectively. The behavior of conductivity is consistent with the Grotthuss mechanism. Only protons jump from the imidazole's or triazole's protonated nitrogen to the unprotonated nitrogen in the adjacent heterocycle molecule and proton transport is conditioned by the imidazole or triazole ring reorientation. The solid-state $^{1}H-^{15}N$ and $^{1}H-^{13}C$ CPMAS NMR spectra, measured as a function of temperature revealed the occurrence of this motion and showed the coexistence of two fractions of imidazole (triazole) molecules: slowly reorienting and exchanging protons and fast reorienting and fast exchanging protons. The two-phase model used for the spectral analysis permitted estimation of the activation energy of the triazole ring reorientation and imidazole tautomerization. The properties of the CNC-Tri and CNC-Im composites were compared and possible factors influencing the lower value of conductivity in CNC-Tri than in CNC-Im were discussed.

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HIGH DIMENSIONALITY AND HIGH RESOLUTION NMR EXPERIMENTS FOR BIOMOLECULES

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Studies of biomolecular structure and dynamics by NMR spectroscopy at atomic resolution require acquisition of multidimensional spectra. However, the recording time of sufficiently resolved multidimensional spectra is often very long due to the sampling limitations. A variety of different methods, mostly based on non-uniform sampling, were proposed to overcome this limitation in multidimensional NMR spectroscopy. They could be utilized in two different ways, either to shorten the experiment duration without loss of resolution, or to perform experiments that are not obtainable conventionally, i.e. with significantly improved resolution and/or of high dimensionality. Most often first of these two, so called "Fast NMR" approach, is shown as the example of the utility of these methods, as it saves expensive spectrometer time. However, in many cases spectra which are not possible to record conventionally, featuring extraordinary resolution and high number of dimensions may be more interesting from scientific point of view as they reveal effects that are hidden, when spectral lines are broad, or enable resolving spectral ambiguities when peaks are overlapped. This second approach we refer to as "Accurate NMR". Its full potential is manifested when the overall experiment time is less important than a new information available from spectra of high dimensionality (4-6D) or of high resolution approaching natural line-width. The new methods were applied for NMR studies of intrinsically disordered proteins, where the structural disorder in combination with highly repetitive amino-acid sequences causes severe peak overlap in the spectra. Several novel 4-7D pulse sequences are proposed. The new experiments employ non-uniform sampling that enables achieving high resolution in indirectly detected dimensions.

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EXPLORING SCALAR COUPLINGS AND CHEMICAL EXCHANGE FROM LOW TO ULTRA-HIGH FIELDS

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Nuclear Magnetic Resonance is used routinely in thousands of laboratories focusing on Chemistry, Material Science, Structural Biology, etc.. In most cases, NMR is performed at high magnetic fields, with proton resonance frequencies in the hundreds of MHz. Accordingly, most of NMR is taught and described in the context of high magnetic fields. Yet, the popularization of benchtop NMR spectrometers, operating at proton resonance frequencies in the tens of MHz makes it possible to apply the entire NMR toolbox over a range of magnetic fields covering almost two orders of magnitude. In this context, we will discuss the effect of magnetic fields variation on scalar couplings and chemical exchange over this broad range of magnetic fields and beyond. In particular, we will focus on the insight provided by methods that allow to couple high and lower magnetic fields, such as two-field NMR [1] and ZULF-NMR [2-3].

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FIELD-CYCLING MRI RELAXOMETRY

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Field-cycling MRI has been developing continuously during the last years [1]. Although still not used as an standard technique for human diagnosis, recent progresses within academic environment are evolving in this direction [2,3]. In this context, the development of instrumentation aimed for the design of specific contrasts agents turns attractive. A prototype has been developed by modifiying a commercial field-cycling relaxometer and including/replacing additional hardware (to enable MRI experiments), that is, a field-cycling "MRI" relaxometer [4]. The instrument was based on a first variable-geometry wide-bore electromagnet designed for concept testing using phantoms and small animals [5], with a gradient unit of own design including optimized longitudinal gradient coils [6]. Recent experiments show the possibilities of such instrument for testing concepts related to both physical and chemical contrasts. In this talk, we will discuss field-cycling dynamic images [7] and the concept of active contrast [8]. Worth to mention is the fact that low magnetic field conditions were used in these experiments. The presentation will also address some features related to MRI at low-field/low-homogeneity condtions in general, provided the huge attention and the explosive grow that is expected for this topic in the near future (specially for non-cycled solutions) [9]. Differences between "dynamic" and "active" contrasts will also be discussed.

Acknowledgements

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MOLECULAR DYNAMICS BY MEANS OF NMR RELAXOMETRY

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The great advantage of NMR relaxometry is the ability to vary the magnetic field (and, hence, the resonance frequency) over a broad range, encompassing at least four orders of magnitude 9from about 4kHz to 40MHz, referring to ^{1}H resonance frequency). As a result, one can probe molecular motion on a timescale from ms to ns in a single experiment. Moreover (or one should rather say, most importantly), NMR relaxometry gives access to the mechanism of motion –the shape of frequency dependencies of relaxation rates (often referred to as relaxation dispersion profiles) make it possible to unambiguously distinguish not only between translational and rotational dynamics, but in this way one can identify the diffusion paths and determine their dimensionality or reveal anisotropy of molecular tumbling.
The exceptional potential of NMR relaxometry has rendered it to be one of the most prominent methods to study dynamical properties of molecular and ionic systems, ranging from "simple" liquids, via polymeric and protein systems, food products and tissues, to complex solids.
To exploit the potential of NMR relaxometry a considerably effort should be put into theoretical modelling of relaxation processes. In this lecture an overview of applications of NMR relaxometry to various systems with focus on the theoretical challenges and achievements will be presented.

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PROTON LOW-FIELD NMR FOR THE STUDY OF (BIO)MACROMOLECULAR DYNAMICS
or:
UNDERSTANDING ^{1}H T_2

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Polymeric materials (including biomolecular samples) are characterized by molecular dynamics covering a large timescale range, often accompanied by a complex morphology, comprising phases with different molecular packing and vastly different mobility. High-resolution, mostly ^{13}C-based solid-state NMR can play out its full power in elucidating these complexities, but at the expense of long experimental times. This tutorial summarizes the options provided by proton low-resolution (possibly low-field) NMR in gathering relevant structural and dynamic information. This method may appear rather limited, but I will show that sometimes, compared to ^{13}C NMR, even *more* quantitive information can be gleaned, thanks to the full control over the detected signal ("counting protons").

Dynamic information is mostly encoded in transverse spin evolution governed by the dipolar coupling network [1]. Therefore, methods as simple as *transverse relaxation measurements* can be used to characterize the dynamics, illustrated on the example of helical jump motions in polymer crystallites [2]. In systems with large-scale chain motion far above the glass transition, the dynamics is characterized by power-law time correlation functions and local motional anisotropy, the latter leading to finite residual dipolar couplings in the ten to few hundred Hz range. Also these can be probed quantitatively and be used to back up or disprove models in polymer physics aimed at an understanding of e.g. mechanical properties. To this end, *proton multiple-quantum NMR* has established itself as a more advanced, better analyzable version of transverse relaxometry [3,4]. The two signal functions provided by this technique can be processed and fitted rather reliably, allowing for a separation of coherent spin evolution due to (residual) dipolar couplings and incoherent transverse relaxation effects [5]. This is demonstrated on a recent example concerning the chain dynamics in transient associating networks [6].

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ARE RELAXATION TIME USEFUL IN MEDICINE?

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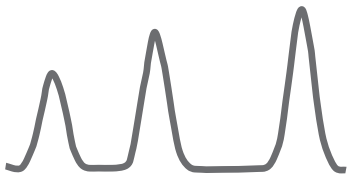
T_1 and T_2 weighted images provide the exquisite soft tissue contrast which has made MRI the technique of choice for most clinical applications in brain. Since the early days of MRI, numerous investigators have believed that accurate measurement of T_1 and T_2 times could provide additional more specific information about brain pathology. While there has been significant progress, relaxation time measurements are still rarely, if ever, used in the clinic although there have been many interesting research applications.

This talk will cover the most accurate ways to measure T_1 and T_2 times, review current understanding about what determines these times and highlight a few interesting applications of relaxation time measurements.

Finally, there will be an attempt to answer the question posed in the title.

PARTICIPANTS POSTERS



AMPERE **NMR** School

High-Resolution Inversion of 1D and 2D Magnetic Resonance Relaxation Data

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The exponential analysis of magnetic resonance relaxation signals determines complex molecular dynamics in multicomponent and heterogeneous materials. Computational methods that transform multiexponential data into the relaxation time distributions directly affect the quality of information that may be acquired. We developed a high-resolution 1D and 2D inversion method based on fast iterative shrinkage-thresholding algorithm (FISTA) to study complex fluid dynamics in multicomponent liquid mixtures and chalk rocks. The developed method successfully detects narrow relaxation time distribution peaks in a variety of samples with excellent repeatability. This inversion toolbox will be publicly available to other researchers in the future.

SOLUTION NMR AS A TOOL TO PROBE COORDINATION OF PARAMAGNETIC METALS DISSOLVED FROM BATTERY CATHODES

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Solution NMR is a tool commonly used to study the degradation products formed in electrolyte solutions of rechargeable batteries.[1-2] Lithium-ion batteries employ transition metal oxide cathodes, which are known to leach paramagnetic cations into the electrolyte solution, a process that contributes to battery failure.[3-4] Electrolyte degradation products are often present in small amounts, which can make observation via NMR challenging, and paramagnetic species are known to cause broadening of NMR signals.[5] This work examines the effect of dissolved Mn^{2+} and Ni^{2+} on the observation of degradation products in nonaqueous $LiPF_6$ electrolyte solution, and in so doing probes how these metals are coordinated. ^{1}H, ^{19}F, and ^{31}P NMR spectra reveal that anionic inorganic degradation products (formed from $LiPF_6$) are greatly affected by the presence of paramagnetic metals, but organic degradation products (formed from carbonate solvents) are less affected. ^{1}H and ^{19}F relaxation measurements confirm that dissolved Mn^{2+} and Ni^{2+} preferentially coordinate to the degradation product $PO_2F_2^-$ over the PF_6^- salt and carbonate solvents: thus, not only are degradation species present in small amounts, but many of them also show preferential binding to the metals that cause signal broadening. It is shown that this spectral loss of degradation products can be mediated through careful selection of deuterated solvents, as solvents that competitively coordinate the transition metals (e.g., dimethyl sulfoxide) facilitate the observation of degradation products over solvents that do not (e.g., acetonitrile).

Acknowledgements
This work was supported by the Faraday Institution Degradation project (EP/S003053/1, FIRG001) and by the Natural Sciences and Engineering Research Council of Canada.

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UNRAVELING THE UNDERLYING SOURCES OF DIFFUSION KURTOSIS IN FOCAL ISCHEMIA BY CORRELATION TENSOR MRI

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Stroke is a leading cause of long-term disability and death worldwide [1]. Currently, imaging techniques lack the specificity to resolve infarct core from penumbra and assess functional recovery. Here, we harness Correlation tensor MRI (CTI) [2] – a method capable of resolving the sources of non-Gaussian diffusion – to enhance specificity and improve acute ischemic lesions characterization.

A photothrombotic stroke was induced in mice, followed by brain extraction at 3h post-illumination. Diffusion MRI data were acquired on a 16.4 T Bruker Aeon (*ex-vivo*) and a 9.4T Bruker Biospec scanner (*in-vivo*) using CTI protocols [3].

Our CTI experiments (Fig. 1) revealed that microscopic kurtosis (μK) substantially contributes to the total kurtosis excess (K_T), and that anisotropic kurtosis (K_{aniso}) decreases substantially, consistent with predictions for neurite beading [5,6]. Edema was relatively small as evidenced by isotropic kurtosis (K_{iso}). CTI also enhanced the sensitivity towards stroke detection.

We demonstrated CTI's ability to resolve sources of non-Gaussian diffusion without prior microstructural assumptions. Such characterizations are pivotal in resolving the long-standing debate on the origins of diffusion sensitivity to acute stroke lesions [4-5]. Future studies aim to resolve the ischemic core and penumbra using CTI metrics. CTI-driven local changes in anisotropy [2], diffusivity distributions, and increases in structural disorder and restriction are promising for more sensitive detection of specific microstructural changes post-ischemia, which bodes well for novel characterizations and treatment efficacy.

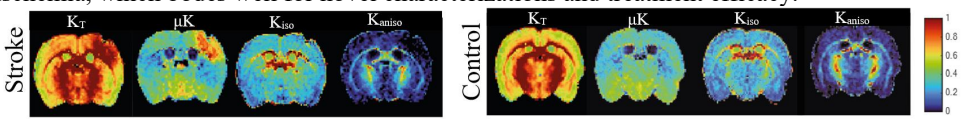

FIGURE 1. CTI metrics for a stroke and control *ex vivo* brains. In stroke, the ipsilesional hemisphere shows higher K_T in gray matter. A clear distinction between hemispheres is observed in μK, showing greater intensities in white and gray matter. K_{aniso} presents lower values within the ipsilesional hemisphere.

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CROSS-CORRELATED SPIN RELAXATION IN NMR STUDIES OF PROTEINS

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Intrinsically disordered proteins (IDPs) represent a considerable part of all naturally occurring proteins. They can exist in multiple forms and various transiently-adopted structures, while performing many biological functions [1]. It is crucial to develop reliable methods of examination of the structure of such proteins.

Cross-correlated relaxation of nuclear spins (CCR) is based on constructive or destructive interference of correlated relaxation mechanisms, which leads to changes in intensity of peaks in NMR spectra [2]. Intensity ratio of peaks in "auto" spectrum (without cross-correlation) to peaks in "cross" spectrum (with cross-correlation) includes information about particular dihedral angles within a molecule. A broad set of such NMR experiments allows to determine the values of the dihedral angles, which can be used to study the structure of proteins, including the flexible elements of IDPs [3]. The aim of conducted research was to design new multidimensional NMR experiments utilising CCR phenomena, which could be used to study structures of IDPs.

Three new CCR NMR experiments have been designed which allow to measure interference of three different dipole-dipole interactions: $N_iH^N{}_i$-CA_iHA_i, $N_iH^N{}_i$-$CA_{i-1}HA_{i-1}$ and $N_iH^N{}_i$-$N_{i-1}H^N{}_{i-1}$. The experiments have been programmed and verified by recording spectra of ubiquitin, a structured protein which consists of 76 aminoacids. A set of CCR rates has been obtained and compared with values established based on published structures of ubiquitin, the results proved to be sufficiently consistent. The next step of the research will be an application of the new experiments to determine the transient structures of intrinsically disordered proteins.

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NMR STUDIES ON POLY(IONIC LIQUID)-BASED AEROGELS FOR CO_2 CAPTURE AND CONVERSION

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Climate change mitigation relies on the generation of new materials that work at the same time as CO_2 sorbents and catalysts. Until now, the existing strategies for CO_2 capture (CC) require high energy consumption or are affected by the presence of impurities.[1,2]

Ionic liquids (ILs) are salts composed of organic cations with organic or inorganic anions, whose properties are tuneable towards the applications through structural changes. Furthermore, poly(ionic liquid)s (PILs) combine the unique characteristics of ILs with a macromolecular framework.[3]

In this work, high pressure NMR (HP-NMR) studies using model ILs provide us a molecular view on the interactions between IL anions/cations and CO_2. The identification of the most successful IL structural features are then incorporated in PIL-based aerogels. Aerogels are light-weight nanostructured materials with high porosity and specific surface area. They can be obtained from biopolymers such as chitosan, a biodegradable starting material that is also a biomass residue.[4,5] Based on this, we are developing chitosan-based aerogels with PILs (*AEROPILS*) aiming to improve gas diffusion and permeability. Preliminary tests as well as the evaluation of CC capacity show promising results.

Acknowledgements
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OPTIMIZATION OF THE ALGORITHM FOR NMR RESONANCE ASSIGNMENT OF INTRINSICALLY DISORDERED PROTEINS

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Intrinsically disordered proteins (IDPs), despite the lack of a rigid structure, show strictly defined functions in the body – they often regulate the activity of cells and are responsible for molecular recognition. NMR, being able to study such dynamic objects, is the method of choice here, however it also faces some difficulties. Rapid changes in the chemical environment cause chemical shift averaging, which results in high peak overlap. The solution to this problem is to use three-dimensional (3D) spectra in combination with four- and five-dimensional (4D and 5D) ones [1].

Resonance assignment based on high-dimensional spectra is easy and reliable. It can be performed automatically by the TSAR [2,3] (Tool for SMFT-based Assignment of Resonances) program.

The goal of the present work was to support the assignment with information on predicted chemical shifts for a given protein obtained the Potenci program[4]. The highly automatic testing system allowed also to identify some problematic procedures in the original algorithm. TSAR with the new procedures was tested on a group of 20 natively unstructured proteins from the Biological Magnetic Resonance Bank database. A decrease in the number of incorrectly assigned residues from 0.94% to 0.54% (decrease by 42%), with a slight increase in the number of correct assignments (from 88.05 % to 88.12 %), was achieved.

The last part of the work was to apply the improved program to the previously unassigned MetC [5] protein. The program correctly assigned 65% of the residues and incorrectly assigned 1% of the residues. The assignment was verified and completed manually - 68 % of the residues were assigned.

Acknowledgements

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FLOQUET PRETHERMALIZATION WITH LIFETIME EXCEEDING 90s IN A BULK HYPERPOLARIZED SOLID

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We report the observation of long-lived Floquet prethermal states in a bulk solid composed of dipolar-coupled ^{13}C nuclei in diamond at room temperature. For precessing nuclear spins prepared in an initial transverse state, we demonstrate pulsed spin-lock Floquet control that prevents their decay over multiple-minute long periods [1, 2]. We observe Floquet prethermal lifetimes $T'_2 \approx 90.9$s, extended >60,000-fold over the nuclear free induction decay times. The spins themselves are continuously interrogated for ∼10min, corresponding to the application of ≈5.8M control pulses. The ^{13}C nuclei are optically hyperpolarized by lattice Nitrogen Vacancy (NV) centers [3]; the combination of hyperpolarization and continuous spin readout yields significant signal-to-noise in the measurements. This allows probing the Floquet thermalization dynamics with unprecedented clarity. We identify four characteristic regimes of the thermalization process, discerning short-time transient processes leading to the prethermal plateau, and long-time system heating towards infinite temperature. This work points to new opportunities possible via Floquet control in networks of dilute, randomly distributed, low-sensitivity nuclei. In particular, the combination of minutes-long prethermal lifetimes and continuous spin interrogation opens avenues for quantum sensors constructed from hyperpolarized Floquet prethermal nuclei.

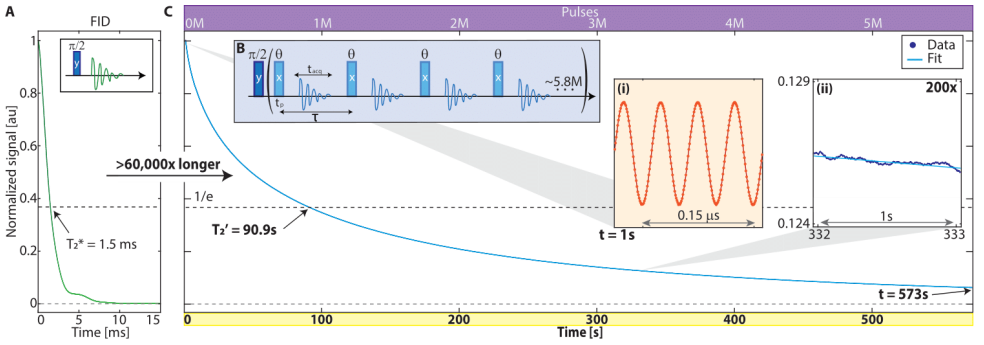

FIGURE 1. Floquet driving and lifetime extension. (A) Conventional ^{13}C free induction decay with $T_2^* \sim 1.5ms$. (B) *Floquet drive* consists of a train of θ-pulses applied spin-locked with the ^{13}C nuclei. Spins are interrogated in t_{acq} windows between the pulses (blue lines), the nuclear precession is sampled every 1ns. Pulse sequence not drawn to scale. (C) *Minutes-long lifetimes* of the transverse state under Floquet control ($\theta \sim \pi/2$). Data shows *single-shot* measurement of survival probability in the state $\rho_I \sim \varepsilon I_x$, and line is a fit to a sum of five exponentials. Here the *573s* period corresponds to *~5.8M* pulses. *Inset (i):* Raw data showing the ^{13}C spin precession, here at 1s into the decay. *Inset (ii):* Data zoomed *200x* in a *1s* window.

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STRUCTURE ELUCIDATION OF NOVEL (*N*-BOC-CYCLOAMINYL)-1,2-OXAZOLE AND PYRAZOLE-4-CARBOXYLATES BY NMR SPECTROSCOPY

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Undoubtedly, natural and synthetic amino acids are valuable precursors for the synthesis of more complex organic molecules, including heterocycles and synthetic peptides [1]. We have recently developed an efficient method for the conversion of various cyclic amino acids to the functionalized *N*-heterocyclic carboxylates [2]. The purpose of the current work was to obtain chiral and achiral building blocks from the cyclic amino acids, containing the 1,2-oxazole and pyrazole-4-carboxylate moieties and their structure elucidation using various NMR spectroscopy techniques.

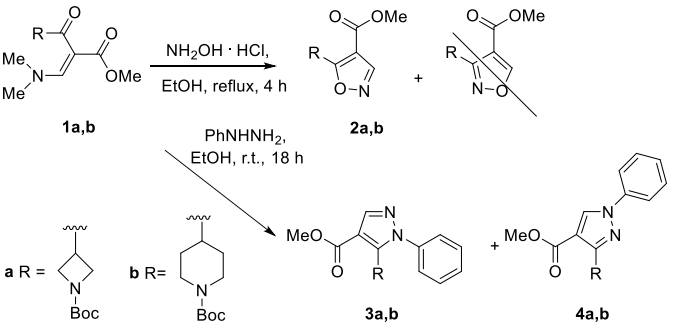

FIGURE 1. Conversion of cyclic amino acids to 1,2-oxazole and pyrazole derivatives.

When enaminone **1a,b** was heated with hydroxylamine hydrochloride or phenylhydrazine in ethanol, the target *N*-heterocyclic carboxylates formed in a moderate yield. The reaction can proceed through two different paths, producing different isomeric products [3]. The structures of all synthesized compounds were confirmed by detailed NMR spectroscopy and HRMS investigations.

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OXIME-FUNCTIONALIZED NANODIAMONDS AS A PLATFORM FOR TREATMENT OF ORGANOPHOSPHATE POISONING

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Nanoscience drastically changed our classical understanding of chemistry, biology, physics, and molecular interactions, allows us to try new pathways for drug delivery and use of bioactive compounds for nanomedicine.

In recent years, different carbon nanoparticles (CNP) were used for biomedical applications [1]. They were studied as a potential treatment, as well as a drug delivery platform. In this study, we used the nanodiamonds (NDs) which showed the least toxicity amongst other CNP [2]. The nanopowder made of ca. 5nm diamond particles with large accessible surface and tailorable surface chemistry shows extraordinary optical, mechanical, electronic, and thermal properties on the nanoscale. Inert and biocompatible NDs could be used in nanomedicine and biotechnology to improve the therapeutic value of various drugs [3]. The coating of drugs on NDs increases their bioavailability, solubility, retention time, efficacy, tolerability, and drug therapeutic index [1].

We tried to design the most beneficial method of the preparation of drug-coated harmless NDs for treatment of organophosphate poisoning. Developing potent antidotes towards acetylcholinesterase (ACHE) inhibited by OP in the central nervous system remains a challenging task. The pre-treated detonated NDs bear carboxylic groups on their surface. This allowed us to perform coating using traditional methods of organic synthesis. Continuing our previous work on design of oxime-functionalized reactivators [4], we synthesized several bioactive compounds and purified them, followed by attaching/coating on NDs' surface. The modified NDs were studied by different techniques (ssNMR, FTIR, DLS, SEM) and tested in the collaborator's biomedical laboratories for reactivity towards inhibited ACHE and toxicity screening against the mammalian cells. Its permeability across the blood-brain barrier was carefully investigated.

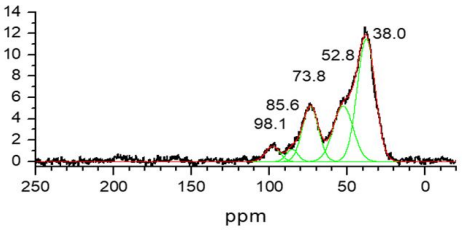

FIGURE 1. *The ^{13}C MAS NMR spectrum of the starting Nanodiamonds.*

Acknowledgements
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SHARPER BENCHTOP NMR: IMPROVING SNR BY APPROACHING NATURAL LINEWIDTHS

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The SHARPER (Sensitive, Homogeneous And Resolved PEaks in Real time) pulse sequence compensates for magnetic field inhomogeneity while also decoupling the heteronuclear couplings for the nucleus of interest, producing extremely narrow singlet signals. The selective variation of the pulse sequence, *sel*-SHARPER, further removes any homonuclear couplings from the signal of interest. Previous results applied SHARPER sequences at high field (400 MHz) to a range of reactions and showed the benefits for reaction monitoring in inhomogeneous magnetic fields, for example those caused by gas bubbling. [1] The greater inhomogeneity of the magnetic field on benchtop instruments means that the SHARPER technique has a wider scope for improvement in reduced linewidth and increased signal:noise (SNR) than at high-field even for standard samples.

This work adapts the SHARPER and *sel*-SHARPER sequences for benchtop NMR systems (Figure 1), producing several alternatives depending on spectrometer capabilities, such as the availability of pulse-field gradients. Figure 1 shows the application of one such sequence to 2,2,3,3,3-pentafluoropropanol achieving linewidths <0.4 Hz, comparable to the standard lineshape sample [2] and improved SNR.

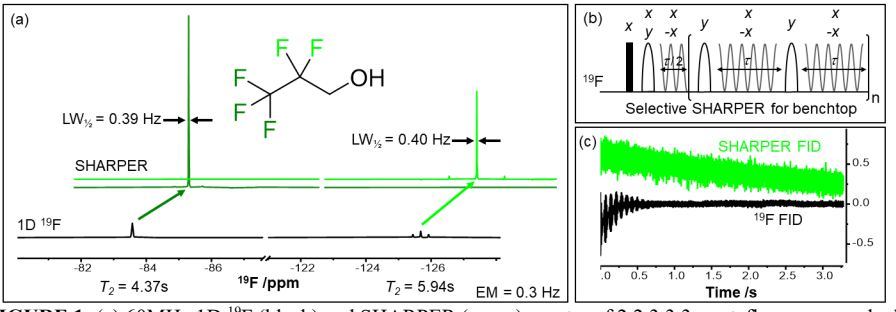

FIGURE 1. (a) 60MHz 1D ^{19}F (black) and SHARPER (green) spectra of 2,2,3,3,3-pentafluoropropanol, all spectra have the same vertical and horizontal scale. LW½ = 0.21 Hz for SHARPER spectra before the application of line broadening (0.3 Hz exponential). (b) The *sel*-SHARPER sequence used to record the data in (a) where τ = 20ms. (c) Real components of 1D ^{19}F and one SHARPER FID from the data shown in (a).

Acknowledgements

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HIGH-PRESSURE GAS ANALYSIS WITH BENCHTOP NMR

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NMR analysis of highly pressurized samples are already of interest since the pioneering days of NMR. However, most of the high-pressure (HP) setups used are usually complicated, highly specialized and lack versatility of applications. Here, a novel setup for high-pressure NMR spectroscopy and relaxation analysis with benchtop NMR is presented [1]. It is easy to operate, simple and small in size. It can be used to analyze the NMR properties of pure gases, to produce and quantify gas mixtures and to analyze gas-solid and gas-liquid interactions. The pressure can be adjusted freely between 0 to 200 bar independently of the sample gas filling pressure with the help of a two-chamber piston cylinder. The piston cylinder can be filled with sample gas in one chamber and by applying pressure to the second chamber on the other side of the cylinder one can manipulate the pressure of the sample gas within seconds.

The versatility of this novel HP setup is demonstrated by different applications. First, proton NMR relaxation times of gaseous and liquefied ethane are presented. Additionally, a three-component mixture comprising methane, ethane and hydrogen was mixed inside the piston cylinder and analyzed *in situ* with ^{1}H NMR spectroscopy at 60 MHz. The component concentrations quantified with NMR spectroscopy are in excellent agreement with gas chromatography data. Furthermore, the setup was used to visualize the effect of pressurized gas on a solid as well as a liquid substance. For this, solid PVC powder was filled into the tube prior to pressurization with gaseous and supercritical CO_2. The ^{1}H NMR spectra of PVC changed with the CO_2 pressure in terms of the peak integral and linewidth. In another experiment, benzene was pressurized with methane, and in a matter of minutes the molar fraction of dissolved methane could be determined by ^{1}H NMR spectroscopy.

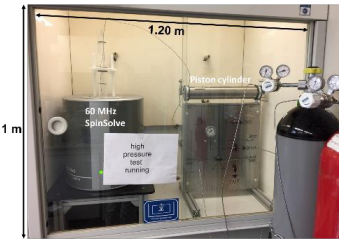

FIGURE 1. Photograph of the presented HP setup inside a chemical laboratory fume hood.

Acknowledgements
We acknowledge the financial and technical support from Equinor, Gassco and Baker Hughes.

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ANALYSIS OF DEPOSITS FROM FUELS AND BIOFUEL-MIXTURES USING BENCHTOP NMR

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The reduction of CO_2 emissions has become a ubiquitous goal for many fields of research. One consequence of this approach is that non-petroleum products like ethanol are added to gasoline or fatty acid methyl ether (FAME) to diesel and heating oil. Yet, the blending of petroleum based oil and FAME leads to a buildup of deposits which are damaging to many components. Unfortunately, the formation and structure of these deposits is curently far from understood.

In this context, we report the use of benchtop NMR spectroscopy and relaxometry to gain more details about the formation of these deposits and their composition during various aging procedures applied to blended heating oils. During an aging experiment, oils were stored for 18 months at 8 °C or 40 °C and their ^{1}H spectra and relaxation times have been analyzed periodically. In another experiment, oil has been pumped through a self-built test rig, which simulates an oil heating system with exception of the ignition process and instead redirects the oil back into the reservoir. The rig was disassembled after 1000 h or after a hardware-related failure and deposits found in the individual parts were analyzed by benchtop NMR.

During the long term storage of oil, solid deposits were found in some of the oils stored at 8 °C. They were identified as accumulations of wax-like esters of glycerol. Apparently, they are intermediates from the FAME production and are dissolved at room temperature. Pure FAME stored at 40 °C shows significant trends of integrals (Fig. 1). The oils examined with the test-rig produced a large amount of residues on several hardware components. Exact identification and quantification of the deposites is far from trivial as the oils are complex mixtures containing refined natural products. Nevertheless, a comparison of the integral ratios of the different spectral regions to the ratio found in the pure oil gives insights on the nature of the deposits and indications from which component they may have originated from.

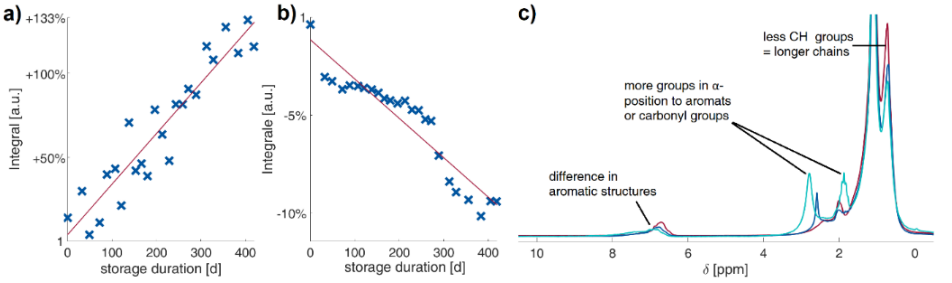

FIGURE 1. Integral change of a) aldehyde and b) double-bond spectral region of pure FAME during storage at 40 °C. c) Visualization of the spectral changes in the ^{1}H spectrum of a heating oil after 1000 h in the test-rig.

Acknowledgements

LOW-COST IDENTIFICATION AND QUANTIFICATION OF PVC PLASTICIZERS BY COMPACT NMR

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NMR instruments with stronger magnetic fields enable NMR spectra with better signal-to-noise-ratio and spectral resolution. Therefore it is not surprising that the quantification of plasticizers extracted from polyvinyl chloride (PVC) was so far only tackled by research groups who have access to high-field NMR devices [1]. Yet, since PVC and its plasticizers are virtually everywhere and several plasticizers are banned from usage in the European Union, an inexpensive and easy to use approach allowing a high analytical throughput is required for the identification and quantification of plasticizers.

In this context, we propose a new, low-cost and simple method based on benchtop NMR spectroscopy and the use of non-deutarated solvents [2]. Figure 1a shows representative ^{1}H spectra of plasticizer solutions with concentrations of 1 vol% acquired using non-deuterated n-hexane. All plasticizers show distinct spectral characteristics above 2.5 ppm which can be easily used for identification and quantification purposes. The aliphatic region is quite similar for all plasticizers. Thus, an appropriate non-deuterated solvent having signals below 2.5 ppm can be used. A limit of detection of 2.49 wt% of a plasticizer in a PVC product was achieved within just 1 minute measuring time. This detection limit is only three times higher compared to the more conventional approach using high-field NMR [1]. A further decrease in the quantification limits can be readily achieved, with the use of compact NMR device working at a higher static magnetic field (Figure 1b). The reliability of the proposed method using non-deuterated solvents is demonstrated by comparisons of the spectra recorded for the the same plasticizer dissolved in deuterated chloroform and by investigating the type of plasticizers and their content in PVC samples with unknwon histories.

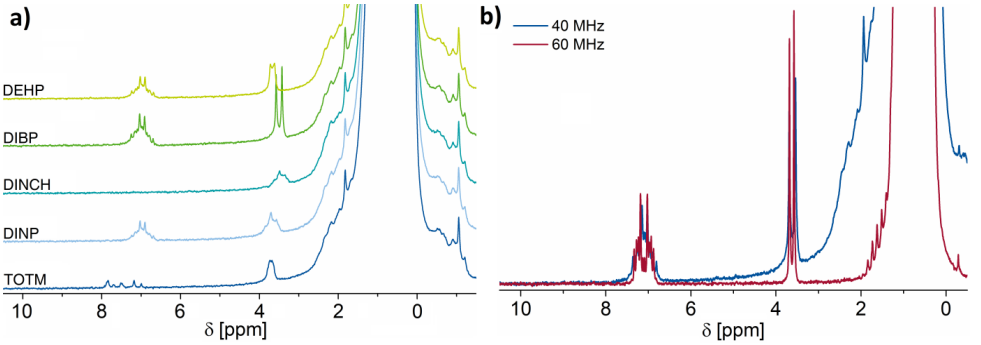

FIGURE 1. a) Comparison of ^{1}H NMR spectra of five plasticizers dissolved in non-deuterated n-hexane acquired with 4 scans using a 40 MHz benchtop device. b) Comparison of ^{1}H spectra of diisobutyl phthalate dissolved in non-deuterated n-hexane acquired at 40 (blue) and 60 (red) MHz using the same number of scans.

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Exploring the Dynamics of Methyl Group Rotations in Halogen-Bonded Cocrystals via Deuterium Solid-State NMR Relaxation Studies

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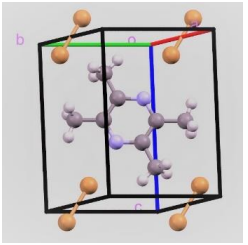
The changes in the free energy barrier and the dynamics of methyl groups are associated with their microenvironment and the supramolecular structures that are formed. It is therefore important to study how these chemically simple methyl groups' rotational barriers and dynamics are controlled by the microenvironment in order to understand the physics of larger molecular rotors that have extensive implications in nanotechnology, catalysis, drug design, etc. This work is focussed on how halogen bonding brings about changes in the deuterium NMR relaxation time constant and the activation energy of methyl group rotations. Our group has reported how deuterium NMR relaxation experiments can elucidate dynamics in solid 2,3,5,6-tetramethylpyrazine cocrystals formed through halogen bonds to iodinated donors.[i] Here, this work is expanded to examine the influence of halogen substitution on methyl group rotation, i.e., by working with brominated and chlorinated donors (e.g., Figure 1). The novel cocrystals synthesized were studied through deuterium NMR relaxation measurements and thereby we have extracted the T_1 relaxation time constants and calculated the activation energy of methyl group rotations.

Fig. 1: Crystal packing of 2,3,5,6-tetramethylpyrazine with Br_2



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Advanced NMR Cryoporometry Characterization of Mesoporous Solids

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Mesoporous solids are used in practical applications, including catalysis, sensing, separations, drug delivery, gas and energy storage, among others. Despite their wide use, the knowledge about their pore structure is still poor, especially for complex porous systems. Analysis of phase equilibria in confined spaces may provide vital structural details. While it is known that strong confinement effects are responsible for the alterations of fluid properties, many aspects of phase equilibria in mesopore spaces still remain matters of debates and uncertainty.

NMR cryoporometry is particularly suited for establishing pore architecture. Current studies utilise signal intensities of spin populations of pore liquid phases assessed using spin echo experiments over an entire experimental temperature range. The disadvantage with this routine approach is that, the well-known Gibbs Thomson equation which relates the freezing/melting point depression of a confined liquid to pore size accounts for a constant thickness of liquid non-frozen layers (nfl) existing between frozen cores and pore walls. However, new insights into the nfl thickness prove a variable thickness with temperature. In very small pores, thermal fluctuations render metastablility towards equilibrium. Additionally, disorder in pores are not revealed by this routine approach. All these shortcomings render the obtained pore size distributions quite inaccurate.

In this work, we provide important insights into the pore structure of small-pore mesoporous solids. We first determine signal amplitudes of liquid phases from the free induction decay (FID) signal by fitting them to a combination of lorentzian and gaussian functions. Accurate separation of the solid and liquid water phases are obtained, with the intensities of liquid free from the nuclear relaxation effects. In addition, a function of nfl thickness with temperature is also introduced to accurately determine the pore size distribution. Furthermore, we employ the recently developed serially-connected pore model (SCPM) which incorporates both thermodynamic equilibrium and metastable transition mechanisms to fully reproduce the experimental findings and obtain deeper insights into the microscopic mechanisms behind the complex phase behaviour. A more representative pore size distributions is, thus, obtained.

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INTERACTIONS BETWEEN HUMAN GPCR DRUGS AND MEMBRANE LIPIDS STUDIED BY EPR AND NMR SPECTROSCOPY

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G protein-coupled receptors (GPCRs) are the largest family of membrane proteins and mediate many cellular responses. As such, they are important in understanding molecular determinants of human diseases. Many ligands and clinical drugs that target GPCRs are poorly soluble in aqueous solutions. This motivates the question of how ligands and drugs access GPCR binding pockets. Related to this is the question of how interactions with the cellular membrane may alter GPCR drug availability and thus affect drug activity. To address these questions, we utilized both NMR spectroscoy and EPR spectroscopy to study interactions of membrane mimetics with drugs that target the human A_{2A} adenosine receptor (A_{2A}AR), a representative human class A GPCR. To study the interaction of A_{2A}AR drugs with the membrane environment, we chemically modified an established antagonist (inactivating ligand) xanthine amine cogener (XAC) by attaching TEMPO to a chemical group of the ligand that faces away from the binding pocket, forming TEMPO-XAC. TEMPO-XAC was verified to bind to A_{2A}AR with a similar affinity as XAC. EPR experiments with both empty lipid nanodiscs and nanodiscs containing A_{2A}AR showed that TEMPO-XAC interacts both with the receptor orthosteric pocket and lipids inside the nanodiscs. To study the interactions of TEMPO-XAC and A_{2A}AR, we recorded [^{15}N,^{1}H]-TROSY experiments with uniformly stable-isotope labeled ^{2}H,^{15}N A_{2A}AR in detergent micelles. The TROSY data confirmed that TEMPO-XAC binds both to A_{2A}AR specifically and also non-specifically interacts with the detergent molecules. These results provide evidence that even receptor ligands with relatively strong affinities for GPCRs may also simultaneously interact with membrane environments. This information provides useful context for future dynamic nuclear polarization (DNP) NMR experiments that utilize TEMPO-XAC and other specific ligands as polarization agents for *in situ* NMR experiments of GPCRs and other human membrane proteins.

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INCLUSION COMPLEXES OF NAPHTHALENE WITH THE NATURALLY OCCURRING CYCLODEXTRIN

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Naphthalene (NP) belongs to polycyclic aromatic hydrocarbons (PAHs). PAHs are very stable thermally and are resistant to degradation, therefore persist in nature for a long time. Naphthalene is included in the list of priority pollutants to be monitored on the basis of its toxicity and abundance. Though toxicity equivalency factor of naphthalene is 1000 lower than that of the most toxic PAH – benzopyrene, its toxicity is the highest among low molecular weight PAHs. Therefore, as complete physicochemical characterization of NP as possible is of great importance.

Cyclodextrins are macrocyclic oligosaccharides composed of a number of glucopyranoside units bound together by α-1,4 bonds. The naturally occurring α-, β- and γ-cyclodextrins (αCD, βCD, and γCD) consist of six, seven, and eight monosaccharides, respectively. CDs, whose shape remains a truncated cone, contain a lipophilic central cavity and a hydrophilic outer surface thus facilitating solubility of partially or fully lipophilic guest molecules in water. Their complexes with harmful chemicals are regarded as one of the methods allowing to remove pollutants from the environment.

The association constants of the complexes formed between NP and cyclodextrins (CD) in water have been determined by several methods, such as vaporization or transport methods, UV absorption, fluorescence, or induced circular dichroism. The results concerning stoichiometry and association constants are contradictory. It has to be pointed out that the method best suited for studying molecular complexes, the nuclear magnetic resonance, has not been exploited due to poor naphthalene solubility in water and inherently low NMR sensitivity. Recently, NMR sensitivity has significantly increased due to technical progress making feasible the NMR titration experiments of NP@CD complexes.

NP@CD inclusion complexes were investigated by ^{1}H NMR with αCD, βCD, and γCD. NP concentration was kept constant (ca. 50 μM/l) while a CD was used as a titrant. Stoichiometries of complexes and their association constants K are summarized in the Table.

CD	Stoichiometry	K [l/M]
αCD	1 : 2	K_1: 10 K_2: 82
βCD	1 : 1	310±48
γCD	1 : 1	274±65

Such stoichiometries are a consequence of a naphthalene size too large to be fully hidden in the cavity of a single αCD molecule, but matching well to βCD and γCD ones.

Au/Ti THIN FILMS PHYSICALLY DEPOSITED ON SiO_2/Si SUBSTRATES IN N/MEMS FABRICATION PROCESS – OPTICAL LITHOGRAPHY IN POSITIVE AND NEGATIVE TECHNOLOGY

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Photolithography is one of the most extensively utilized technologies due to its usage in fabricating microchips in the semiconductor industry. In essence, this technique involves the fabrication of a thin film on the top of a substrate (usually a silicon wafer), in which UV light is passed through a photomask that has the desired micropattern [1].

Reactive ion etching (RIE) is a plasma process where radiofrequency (RF) discharge-excited species (radicals, ions) etch substrate or thin films in a low-pressure chamber. RIE is a synergistic process between chemically active species and energetic ion bombardment. RIE is faster than either pure physical ion bombardment or spontaneous chemical etching [2].

The main purpose of the work was to improve procedures employed in lithography steps (in both: positive and negative systems), to achieve the expected result: 3D N/MEMS (resonators). In the poster, we will present the effects of a multi-level optical lithography process, with the use of AFM topography analysis, PVD Magnetron Sputtering technology during fabrication (well-deposited Au/Ti nano/microstructures on SiO_2/Si substrates). RIE with O_2 plasma was mainly used to clean the substrate from organic remains and to prepare the silicon wafer for the next step of preparation: the wet etching process in hydrofluoric acid.

Keywords: AFM, PVD, RIE, Optical Photolithography, M/NEMS, 3D structures, Resonators.

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Karol Załęski, NanoBioMedical Centre, Adam Mickiewicz University in Poznań.
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ION MOBILITIES IN POLYMER COMPOSITES FOR ELECTROMAGNETIC SHIELDING

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Polymer composite materials based on poly(vinylidene-co-hexafluoropropylene) (PVdF-HFP) have recently been studied from a morphological and electro-magnetic point of view, regarding their potential use as electro-magnetic shielding materials [1,2]. Here, we present an NMR study of composites based on PVdF-HFP, an electrolyte solution made of $LiBF_4$ dissolved in $EMIMBF_4$ (for ionic conductivity), multi-walled carbon nanotubes (providing an electron conducting network) and $BaTiO_3$ nanoparticles exhibiting a high permittivity [2].

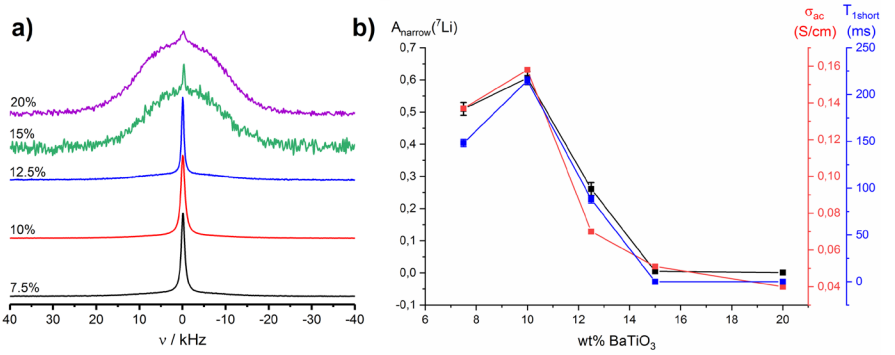

FIGURE 1. (a) ^{7}Li NMR spectra of polymer composites containing variable amounts of $BaTiO_3$ and (b) correlation between the ^{7}Li fraction with a narrow peak, the corresponding T_1 values and AC conductivity σ_{ac}.

Insight into Li^+, 1-ethyl-3methylimidazolium and BF_4^- ion dynamics was obtained by means of multinuclear NMR spectroscopy and relaxometry. For each ionic species, the NMR spectra reveal the presence of two populations, which differ significantly in their dynamics. This effect is especially pronounced for ^{7}Li, where a narrow line is superimposed on a broad hump, which is broadened by dipolar and quadrupolar couplings. The fraction of immobile Li ions increases upon raising the $BaTiO_3$ concentration. Additionally, we find a good correlation between the ^{11}B spinning-sideband intensity with the broad ^{7}Li fraction, which shows that both ions undergo the same dynamic restrictions upon increasing the $BaTiO_3$ concentration.

Acknowledgements
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Broadband Adiabatic-Inversion Cross-Polarization for Acquiring Ultra-Wideline NMR Spectra of Quadrupolar Nuclei

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Large anisotropic NMR interactions can give rise to ultra-wideline (UW) NMR patterns ranging from 250 kHz in breadth to upwards of 10 MHz, making it difficult to acquire uniform spectra [1,2]. Traditionally, short, high-powered, rectangular pulses are used to acquire NMR spectra; however, these are often insufficient for the acquisition of uniformly excited UW NMR patterns. As an alternative, the wideband uniform-rate smooth-truncation (WURST) pulse [3], a type of frequency swept (FS) pulse, can be used for the efficient acquisition of UWNMR patterns via broadband excitation and refocusing of spin polarization. The development of the WURST-CPMG [4] and broadband adiabatic inversion cross polarization (BRAIN-CP) [5] pulse sequences allows for the acquisition of UWNMR patterns using low power pulses through both direct excite (DE) and indirect excite (IE) methods, respectively. Currently, there are very few examples of CP used for the acquisition of central transition (CT, $+1/2 \leftrightarrow -1/2$) UW patterns of half-integer quadrupolar nuclei under static conditions [6]; by contrast, CP to half-integer quadrupoles under MAS conditions has been thoroughly explored [7,8,9,10].

In this work, we discuss the optimization of conditions for the acquisition of CT UWNMR patterns for a series of half-integer quadrupolar nuclides, including ^{35}Cl ($S = 3/2$), ^{55}Mn ($S = 5/2$), ^{59}Co ($S = 7/2$), and ^{93}Nb ($S = 9/2$). Comparisons of spectra acquired with DE and IE methods (with and without WURST pulses) are made, with a focus on relative signal enhancements and improved pattern uniformities. It was found that BRAIN-CP sequences consistently outperform conventional CP experiments in these respects. Additionally, the RF amplitudes of the WURST pulses, which are lower than those in standard CP experiments, scale as $(S+1/2)^{-1}$, resulting in reduced power requirements for higher-spin nuclei. The mechanisms of BRAIN-CP are investigated via numerical simulations, and correlated with experimental results. These methods can enable the study of a wide range of unreceptive quadrupolar nuclei and may be applied to dynamic nuclear polarization (DNP) under static and MAS conditions.

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CHARACTERIZATION OF AMMONIUM IONIC LQIUIDS BY MEANS OF SELF-DIFFUSION AND ITS MICROSTRUCTURE

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Exact characterization of microscopic interactions by terms of self-diffusion coefficient and evaluation of microscopic and macroscopic structure of two ammonium ILs' families (with varied cation) solutions was the aim of this work. In this way, we prepared alkyltriethylammonium and alkylcyclohexyldimethylammonium-based ionic liquids with varied alkyl chain length and bis(trifluoromethylsufonyl)imide anion. Features like low vapor pressure, wide range of liquid state, high thermal, electrochemical and chemical stability, and the ability to dissolve many organic and inorganic compounds, including some polymers are properties of ionic liquids (ILs), organic/inorganic salts with a melting point below 100 ºC.

Minor differences between ionic liquids (alkyl chain length, type of anion, etc.) can have a big influence on their properties. Changes in their chemical structure, affect all physicochemical properties: viscosity, density, hydrophility/hydrophobity, miscibility with solvents, electrochemical properties, thermal stability etc. This projectability is their main added value, differentiating them from conventional solvents, and this is the reason ILs are often considered as green solvents used in laboratory and industrial processes. The specific properties its use, and these depend exclusively on the construction of the anion and cation. Importantly the properties of bulk ILs are affected not only by their chemical structure but also the intra- and inter-molecular interactions, temperature, and the presence of gaseous, liquid or solid impurities. Self-diffusion coefficient is a parameter, which is not dependent on time and, therefore, can be used to characterise the translational mobility (diffusivity) of a certain type of molecule under certain conditions (temperature, pressure, molecular interactions).

After structure confirmation by means of NMR and FTIR, phase transition temperatures were determined by means of differential scanning calorimetry technique. Finally, self-diffusion coefficients were determined for series of the prepared ILs measurements at 14,4 T Agilent NMR spectrometer techniques (DOTY DSI-1372, gz=28 T/m, VnmrJ 4.2; Pulse sequence: PGSE (Pulsed Gradient Spin-Echo).

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^{17}O NMR relaxation measurements of GMO-MnO system

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Oxygen has only one NMR-active stable isotope, ^{17}O. This nuclide has a natural abundance of only 0.038%, a nuclear spin quantum number I = 5/2, and a Larmor frequency ω_0 = 54.22 MHz (at 400 MHz for 1H). The isotropic chemical shift range of ^{17}O spans about 1500 ppm. Water is often used as the solvent in ^{17}O NMR studies.

The nuclear Zeeman energy levels for a spin 5/2 nucleus such as $_{17}$O are six energy levels and five single-quantum transitions. The central transition (CT) between the + 1/2 and − 1/2 levels is the one usually observed in the NMR experiments of such nuclei.

Relaxation is a key phenomenon for magnetic resonance and there is an extensive literature covering its theoretical and experimental aspects. The longitudinal or spin–lattice relaxation rate $R_1 = 1/T_1$ determines the minimum time delay needed between two acquisitions. The transverse or spin-spin relaxation rate $R_2 = 1/T_2$ determines the widths of the resonances in the spectrum. Relaxation can provide valuable information on molecular motions and intramolecular distances and is extensively exploited in MRI.

The relaxivity of an MR contrast agent reflects how the relaxation rates of a solution change as a function of concentration [C]. Since a contrast agent may affect the two relaxation rates ($1/T_1$ and $1/T_2$) individually, there are two corresponding relaxivities, denoted r_1 and r_2. The relaxation rates of a contrast agent in solution are obtained by graphing changes in relaxation rates ($1/\Delta T_1$) and ($1/\Delta T_2$) at different concentrations. The slopes of the lines represent r_1 and r_2.

High-spin Mn(II) (S=5/2) is a potent T_1-relaxation agent that can be used to generate contrast in magnetic resonance imaging (MRI), and understanding hydration is key to understanding function.

Aim of this study was to determine the number of hydration state/water ligands (q) and relaxivity in cubosomes' systems with MnO nanoparticles. Relaxivity was calculated by dividing the MnO imparted increase in $1/T_2$ relative to the concentration of the paramagnetic ion in mM. About 600 µl of 4 samples were prepared at varied concentration in mM: 0,075; 0,1875; 0,375; 0,5625; 0,750 and 1.

NMR spectra were recorded on Avance DMX 400 Bruker spectrometer equipped with a 5 mm broadband probe. 1D 1H NMR One-pulse sequence was used with following parameters: p1 (90° pulse) at 25µs; 1024 number of scans (ns); d1 (relaxation delay) – 100ms. The transverse relaxation times of ^{17}O were measured through the linewidth of NMR signal at half-height at temperature range (21-65°C). After each temperature change there was a 10-15 minute break for thermal stabilization and after that shimming.

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Enthalpies of formation of Cu and Co doped FeNi: theoretical approach

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Enthalpies of formation of FeNi in various structural states were determined using thermodynamic modeling proposed by Miedema [1]. Solid solution and amorphous phase were mainly considered for parent equiatomic composition and for Cu- and Co-doped alloys. It is known that in meteorites $L1_0$ phase forms in contact between bcc- and fcc-FeNi [2]. Both bcc- and fcc- solid solutions exhibit soft magnetic properties. Contrarily, $L1_0$ is a hard magnetic phase [3], due to tetragonal distortion and layered arrangement of Fe and Ni atoms. Possibility of formation of amorphous phase was excluded on the basis of calculations, simultaneously corroborating ability of formation of solid solution. Cu was found to further decrease the enthalpy of formation of solid solution, with Co having negligible effect.

This work was co-financed by the project "Środowiskowe interdyscyplinarne studia doktoranckie w zakresie nanotechnologii" No. POWR.03.02.00-00-I032/16 under the European Social Fund – Operational Programme Knowledge Education Development, Axis III Higher Education for Economy and Development, Action 3.2 PhD Programme.

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LIFETIMES OF ^{31}P NMR SINGLET STATES BETWEEN CHEMICALLY EQUIVALENT NUCLEI

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NMR singlets between ^{31}P nuclei may be relevant to understanding phenomena such as certain biological processes [1]. We report the generation and lifetime measurement of a ^{31}P singlet for a chemically equivalent case in tetrabenzyl pyrophosphate (TBPP, Figure 1a).

The parameters of the unusually complex ^{31}P and ^{1}H spectra, caused by magnetic inequivalence, were extracted by comparing experimental results with simulations using the *Spinach* MATLAB package [2] (Figure 1b). We modeled the compound as two ^{31}P nuclei J-coupling with 4 ^{1}H nuclei three bonds away and found excellent agreement between simulation and experimental results.

Magnetic inequivalence enables access to a ^{31}P singlet state. We measured spin-lattice (R_1) and singlet (R_s) relaxation rates at 9.4 T at various temperatures using inversion-recovery and spin level induced crossing (SLIC) [3] sequences, respectively (Figure 1c). Surprisingly, R_s exceeded R_1 at all temperatures measured, contrary to expected results. Using molecular dynamics (MD) simulations with AMBER and calculating chemical shift anisotropy (CSA) tensors with Gaussian, we found that the short relaxation rates could be explained by the very large and highly anticorrelated CSA tensors present between ^{31}P nuclei in TBPP. It is therefore likely that singlet lifetimes are significantly longer at low magnetic fields. Future work will focus on ^{31}P singlets in other compounds, including biological species.

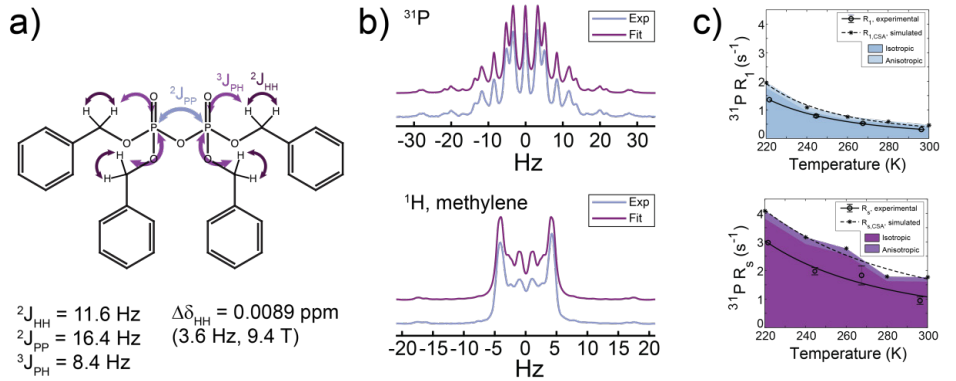

FIGURE 1. (a) Structure of tetrabenzyl pyrophosphate and chemical shift/coupling parameters from simulation. (b) Experimental NMR spectra and overlaid fits from simulation. (c) Measured R_1 and R_s relaxation rates as a function of temperature, along with values from molecular dynamics simulations of CSA.

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SIZE DEPENDENT TOXICITY AND DISTRIBUTION OF POLYMERIC NANOPARTICLES IN HUMAN CELLS

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One of the biggest challenges facing cancer biology nowadays is the development of new safe carriers that are able to transport therapeutics inside tumour cells. In the past few years nanoparticles have become a prime candidate for a new drug and RNA delivery system to human cells. In most cases they seem to be non-toxic to cells by direct contact as well as after internalization to cellular matrix.

Despite many years of studying nanostructures, the question of how they enter cells is still unclear. In this study on model polydopamine (PDA) nanoparticles we want to evaluate if size of nanoparticle can change it's toxicity and ability to penetrate cellular membrane. We used cytotoxicity assays to determine if PDA nanoparticles toxicity is connected to their size on four different cell lines – two cancerous and two described as non-cancerous. Next step is to block different paths how NPs can enter the cells and how it change with increase of nanoparticles size.

Acknowledgements
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Pore structures and connectivity of metakaolin-based geopolymers detected by ^{129}Xe NMR methods

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Geopolymers are cement like materials which are also good inorganic adsorbents and catalysts with disordered three-dimentional framework. These materials can also be thought of as X-ray-amorphous zeolites. The understanding of geopolymers' pore structure is important for its development and use as an adsorbent and catalyst [1]. Here, six metakaolin-based geopolymers with different water-to-solid (*w/s*) ratios were prepared and their pore structures and connectivity were studied by ^{129}Xe NMR, which has been shown to be a good method to study the pore structure in various porous systems [2]. The temperature dependent ^{129}Xe NMR spectra were used to detect pores with different sizes (Fig. 1a) and the average pore diameters as well as heats of adsorption (Fig. 1b and c). The connectivity of pores was studied by quantifying the exchange rates of ^{129}Xe moving between different environments using selective IR experiments (Fig. 1d and e). The highest water content sample was found to have only one pore type, higher average pore diameter, heat adsorption and exchange rates than the four samples with medium water contents, while the lowest water content sample did not form a good pore structure as it is the theoretical lowest *w/s* ratio.

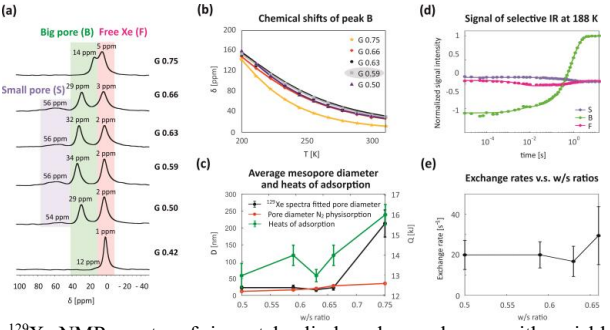

FIGURE 1. (a) The ^{129}Xe NMR spectra of six metakaolin-based geopolymers with variable *w/s* ratios at room temperature. (b) The ^{129}Xe chemical shifts as a function of temperature. (c) The average mesopore diameter and heats of adsorption fitted from ^{129}Xe spectra. (d) The signal of selective IR of G0.59 at 188 K. (e) The fitted exchange rates from selective IR data as a function of *w/s* ratios at 188 K.

Acknowledgements
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DENDRITES ON LITHIUM-METAL BATTERIES: A NUMERICAL APPROACH TO THE EFFECT OF THE MORPHOLOGY ON THE NMR SPECTRUM

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Lithium metal is considered the ultimate anode for lithium based batteries, due to, for instance, its high theoretical capcity (3860 $mAhg^{-1}$) compare with the currently used graphite [1]. However, this electrode presents several drawbacks that compromise the battery security and performance, such as the growth of lithium microstructures (dendrites) during the charge/discharge process. Great efforts are made to overcome those issues by proposing strategies for dendrite suppression and regulation. Therefore, it is crucial to have characterization tools to monitor the growth of dendrites throughout the cycles. In this sense, Nuclear Magnetic Resonance emerges as a powerful non-invasive technique capable of detecting the formation of dendrites [2]. Although it is possible to detect the presence of microstructures, interpretation of the spectra is not straightforward because different morphologies can lead to similar results [3]. The shape of the spectrum depends on the perturbations of the local magnetic field produced by the dendrites in presence of the static magnetic field, and a better interpretation of the spectrum can be achieved by means of numerical calculations of such perturbations. In this work, we performed those computations using the fourier based method developed by Salomir et al. [4]. This calculations allow us to study the effect of the dendrites on the NMR spectrum as a function of the density, height and width of the mictrostructures. We found that the most important factor is the density of dendrites, as suggested by Küpers et al. [3]. This simulations can also be useful for the assessment of multipulse sequences, such as proposed by Ilott et al [5], which could be used to enhance the microstructures signal.

Acknowledgements
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CHARACTERIZATION OF DYNAMIC REGIMES AND MEMBRANE ELASTICITY OF FLEXIBLE LIPOSOMES USING FAST-FIELD-CYCLING NMR

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At present, the development of nanometric vesicles for drug delivery in the human body by different routes has become of great interest for academic and industrial research groups. Flexible liposomes have proved to be useful for transdermal transport, therefore it is important to understand the underplaying mechanisms associated to the molecular dynamics, that influence the membrane elasticity.

In previous works [1-5], a model was presented to interpret the spin-lattice relaxation rate dispersion of protons, obtained with the fast field cycling nuclear magnetic relaxometry technique (FFC), for unilamellar liposomes. In addition to providing general information on the lipid dynamics, this model allows us to infer about the elastic properties of the liposomes by means of the elastic constant κ, which is one of the physical parameters involved in the model. This model has been validated from measurements of κ for cholesterol doped vesicles and for surfactant doped vesicles, in order to increase or decrease the value of the constant respectively.

In order to study the influence of the temperature and the surfactant's characteristics in the behavior of the molecular parameters in flexible liposomes, experiments using elastic vesicles were performed in this work. Specifically, we analyzed experimental relaxation dispersions obtained at four different temperatures (291-328K) for liposomes of radius of 50nm, composed of SPC (soy phosphatidylcholine) with four different detergents added to modulate the membrane flexibility, at concentrations up to 20%mol.

For the lowest temperature the elastic modulus of the membrane decreases with the addition of surfactant, as expected. Also, after a correlation analysis, a great influence of some properties of the surfactant on specific physical parameters of the system was found. In addition comparing with previous works [4; 5], a different dynamical properties a molecular level have been observed depending on the purity of the used lipids. Finally, a deeper analysis about how the relationship between the elastic constant and the diffusion constant of the membrane can describe changes in the deformability performance of a liposome formulation was made.

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A ^{19}F NMR APPROACH TO STUDY A CO-CHAPERONE-BASED QUALITY CONTROL SYSTEM

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Cells need reliable mechanisms to detect and remove misfolded proteins as these are aggregation-prone, thereby causing cell stress or death and the malfunctioning of protein quality control underlies many neurodegenerative diseases such as Parkinson's disease. In addition, lots of newly synthesised proteins contain hydrophobic regions for membrane insertion that must be shielded from the aqueous cytosol.

Involved in both processes are co-chaperones like SGTA, which delivers tail-anchored membrane proteins, denoted by a C-terminal hydrophobic helix to the endoplasmic reticulum. In conjuction with the Bcl2-associated anthanogene 6 (BAG6) complex SGTA also controls the fate of misfolded or mislocalised proteins by promoting their refolding and direction to the ER membrane or targeting them for proteasomal degradation [1].

The hydrophobic substrates are recognised and bound by SGTA's C-terminal domain, but the mechanism of distinction between the good and the bad is not understood. SGTA has three self-contained domains and the N-terminal dimerisation and TPR domains of known structure bind to BAG6 and proteasomal receptors, respectively. The C-terminal domain was studied using an integrative approach [2] and the quality control mechanism to sort hydrophobic proteins for rescue or degradation was investigated by ^{19}F NMR spectroscopy.

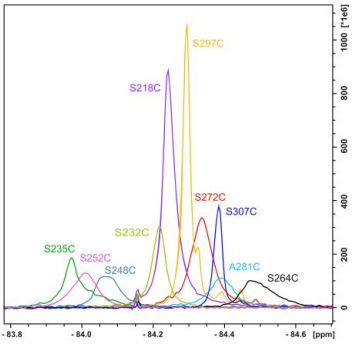

FIGURE 1. Overlaid 1D ^{19}F NMR spectra of individual SGTA C-terminal domain mutants.

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Dr R.A. Atkinson and the Centre for Biomolecular Spectroscopy, King's College London

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PHOTOLUMINESCENT PROPERTIES OF ZNO NANOPARTICLES GROWED ON THE ELECTROSPUN POLYMER NANOFIBERS

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One-dimensional (1D) ZnO nanomaterial is the object of the interest in a variety of studies due to its unique photoluminescent (PL) properties, high surface to volume ratio, biocompatibility, non-toxicity, and stability. 1D ZnO nanocomposites can be produced by a combination of several independent techniques, such as electrospinning and atomic layer deposition (ALD). Among the various methods, electrospinning is a simple, controllable, and inexpensive method of making flexible polymer mats with uniform nanofiber lengths and diameters [1, 2]. These attributes make polymer nanofibers ideal candidates for coating by a thin layer of ZnO, in order to obtain nanomaterials with the desired characteristics and properties.

In present study, we report on the investigation of the structural and optical properties of four different electrospun polymer (PVA, PA6, PVDF, PLLA) nanofibers covered by the ALD ZnO. It was found that only PVDF/ZnO nanofibers exhibit stable room temperature PL that may be the result of a higher ZnO content in the sample. In addition, PL measurements were conducted as a function of excitation power and temperature in order to establish the main PL mechanisms and parameters for the PVDF/ZnO sample, as the most promising candidate for the biophotonic application.

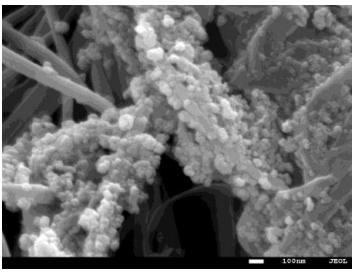

FIGURE 1. PVDF/ZnO nanocomposite made by a combination of electrospinning and atomic layer deposition methods.

Acknowledgements

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UNDERSTANDING THE MICROSTRUCTURAL EVOLUTION OF CEMENTED PASTE BACKFILL WITH LOW FIELD NMR RELAXATION

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Cemented paste backfill (CPB) comprising mineral tailings, binders and mixing waters is an important potential support material in the mining industry. As the mechanical properties of CPB are significantly influenced by its microstructural characteristics the development of measurement tools to better understand its pore structure evolution is important for its increased utilisation. Based on our recent study [1], this poster reports the application of low-field nuclear magnetic resonance (NMR) relaxation measurements to characterise the microstructural evolution of CPB, contrasting common tap water and hypersaline water (~22 wt% salt) as mixing waters. Both T_1 and T_2 relaxation times were found to correlate with the uniaxial compressive strength (UCS) of our CPB materials, facilitating the formulation of a predictive correlation function between NMR relaxometry and mechanical properties.

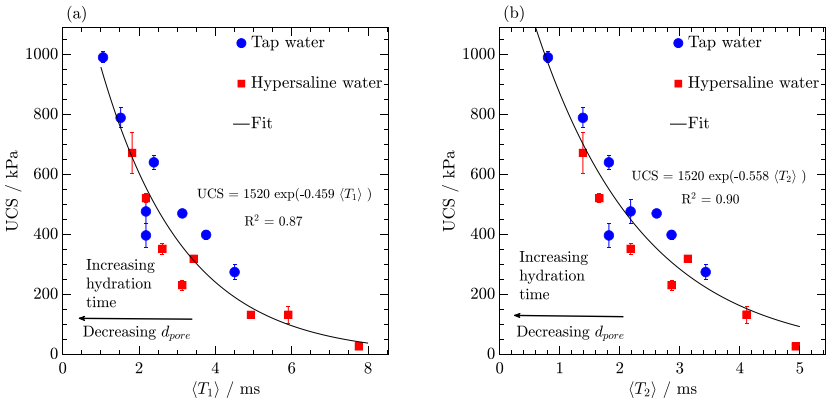

FIGURE 1. Correlation of (a) <T_1> and (b) <T_2> NMR relaxation times against CPB UCS data. Data from materials containing both 5 wt% and 9 wt% binder content are overlaid. A model exponential decay is detailed in each case and discussed further in the main poster.

Acknowledgements
Razyq Nasharuddin acknowledges the PhD financial support from the Australian Government Research Training Program (RTP) Scholarship.

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New Experimental Observations of the Behavior of Sodium Ions in Saturated Rock Samples

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^{1}H NMR relaxometry of saturated rock samples has become a useful tool for the characterization of porosity and transport phenomena of enclosed fluids. The pore size can be measured using the difference between inverse relaxation values of protons absorbed by the saturated rock and that present in the bulk fluids. These experiments are usually performed at low magnetic fields to reduce the influence of the diffusion on the relaxation values in the presence of Internal Gradient Fields. Recently, sodium ions have become objects of investigation. However unlike protons, sodium ions can have anisotropic properties like appearance of the bi-exponential relaxation and residual quadrupolar distribution, which can lead to complex behavior of spins inside the pores. Here, we describe eight ^{23}Na NMR experiments at 9.39 T external magnetic field, in which we have investigated the behavior of sodium ions in 4 saturated rock samples[1]. Comparing Spin Echo and CPMG experimental data, we demonstrate that the CPMG data can be incorrectly interpreted when weak pulses are used in the presence of Internal Gradient Fields (Figure 1a). We show that the reduction of spin-spin relaxation value with unchanged of spin-lattice relaxation value of sodium ions is caused by anisotropic motion and cannot be explained in the same way as for protons (by influence of the diffusion in the presence of Internal Gradient Fields). There can be a link between free diffusional and motional averaging regimes regardless of the size of environment in which the measured spin ions are (Figure 1b). This work demonstrates unique model for the behavior of ions inside pore media, which is different than known models (Brownstein-Tarr model and "agarose gel model").

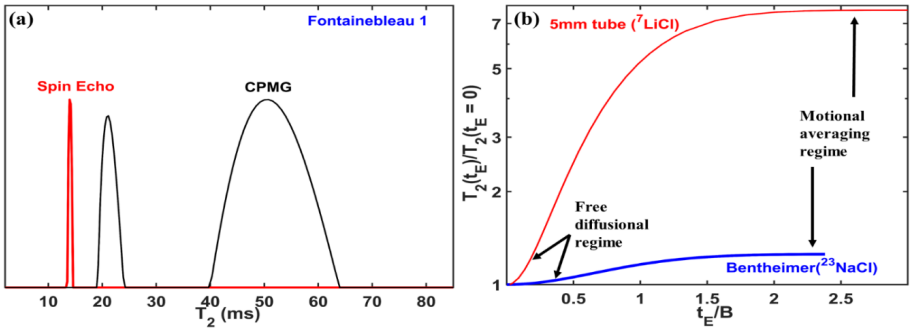

Figure 1. (a) Probability distribution of T_2 of ^{23}Na Spin Echo (red line) and CPMG (black line) experiments in Fontainebleau 1 sandstone. Fontainebleau 1 has a single pore distribution. (b) The dependence of T_2 values (normalized with T_2 at $t_E = 0$) on the Spin Echo time (t_E) of ^{23}NaCl in Bentheimer sandstone and ^{7}LiCl solution in 5 mm tube (red line). The normalized parameter B depends on the diffusion coefficient and the free mean path.

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HYDROGEN BONDING OF METHYLATED ADENINE ANALOGUES STUDIED BY NMR SPECTROSCOPY

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Hydrogen bonds (H-bonds) play a key role in the determination of structure and proper function of nucleic acids (NAs). There are two main types of base pairing geometry in NAs – Watson-Crick type, introduced in 1953 [1], and Hoogsteen type, discovered a few years later [2].

Adenine molecule is able to form both base pairing types mentioned above, but the situation differs when it undergoes methylation. N^6-methylation of adenine has been considered absent in human genome until 2018 [3]. Nowadays, this modification is related to obesity and Alzheimer's disease [4]. The methyl group in position 6 can adopt two orientations because the bond between (methyl)amino group and purine ring is of an order higher than one, and the rotation around this bond is restricted. Each of the conformers (rotamers) has a different hydrogen bonding pattern, and the steric hindrance of methyl group discriminates Watson-Crick or Hoogsteen base-pairing site.

In this work we present NMR and DFT study of the intermolecular hydrogen bonds of (methylated) adenine analogues with their complemental partner, thymine. We found out that the *N*-methylation stabilizes Hoogsteen base pairing. This pairing is also preferred for those derivatives, which are able to form both complex types (Hoogsteen and Watson-Crick). Watson-Crick geometry is preferred when three hydrogen bonds can be formed between the adenine derivative and thymine. We described and compared the intermolecular interactions of four adenine derivatives and their rotamers with thymine in different solvents using low-temperature NMR spectroscopy.

Acknowledgements
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DMSO/IL solvent systems for cellulose dissolution: binary or ternary mixtures?

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The mechanism of cellulose dissolution in ionic liquid - dimethylsulphoxide solvent systems has attracted a lot of attention fuelled by the prospect of using this biopolymer as a green alternative to synthetic materials.[1]

Ternary [C_4mim]Cl/DMSO/H_2O mixtures were studied applying Nuclear Magnetic Resonance (NMR) experiments and molecular dynamics (MD) simulations. Titration of binary and ternary solvent systems with water and DMSO disclosed a relation between the molar fraction of the mixture components, and the proton chemical shift of H_2O. Three main regions were observed: (i) a chemical shift similar to water in DMSO; (ii) intermediate values of H_2O chemical shift; (iii) a chemical shift similar to pure water. An 'ideal' range for the IL/DMSO/H_2O ratio was observed, which was tested in cellulose dissolution and rationalized using IL – cellobiose NOE interactions.

The MD simulations support this theory demonstrating the presence of molar composition-dependent interactions. In low x_{IL} a sandwich-like structure is formed where the water molecules tend to be positioned between the cation and anion. In intermediate concentrations of IL, the organization changes, a complex 3D network of DMSO and IL cations exists, and both water and chloride can be found in equivalent regions of space around the cation, suggesting a looser IL networks. The NMR NOE analysis on cellobiose dissolved in these solvents supported this hypothesis by showing interactions between cellobiose and the IL cation that could be switched on and off by varying the solvent composition.[2]

Acknowledgements
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POSTER N^{O} 35

STUDY OF AMYLIN MUTANTS PROPERTIES USING NMR SPECTROSCOPY

Aleksandra Pawlak

The aim of the study is to obtain chemical shifts for modified amylin analogues. The tested peptides have amyloidogenic properties. Chemical shifts were assigned based on a series of two-dimensional NMR spectra. The work covers the biological aspects of protein aggregation and emphasizes the essence of research on amylins as well as the use of computer programs for spectral analysis. As part of the work, chemical shifts of five amylin analogues were obtained. The lack of sequence signals in the ROESY spectra made it impossible to unambiguously assign the signals in the case of amino acids occurring multiple times in the peptide sequence. The resulting chemical shifts were compared with the chemical shifts simulated under the assumption that the peptides lacked structure. Chemical shifts were also used to check whether these peptides had a secondary structure.

2D DISPEL-TOCSY: USING 2D NMR TO QUANTIFY ^{13}C ENRICHMENT IN METABOLOMICS SAMPLES

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DISPEL (Destruction of Interfering Satellites by Perfect Echo Low-pass filtration) is a NMR technique that supresses one-bond ^{13}C satellites in ^{1}H spectra [1]. While satellites normally appear at 0.54% intensity compared to the parent peaks and often have a negligible impact on the appearance of spectra, they can be much more intense in spectra of metabolites prepared using ^{13}C labelled feedstock for tracking metabolic pathways of microorganisms. [2]. This greatly increases the intensity of ^{13}C satellites in ^{1}H spectra causing them to overlap and obscure the ^{1}H-^{12}C signals. Simplification of the spectra achievable via DISPEL makes it a useful technique for identification of metabolites in such samples. For simple mixtures, the use of 1D DISPEL can also assist with quantification of the levels of ^{13}C incorporation. Nevertheless, in highly complex mixtures even this simplification is insufficient.

A further development of the DISPEL technique aimed to quantify site-specific ^{13}C enrichment of metabolites presented in complex mixtures, is described here. The combination of DISPEL with the commonly used 2D ^{1}H-^{1}H TOCSY sequence, shown in Figure 1b, can lead to selective suppression of signals arising from transfer between protons bonded to different carbon isotopes. This depends on where DISPEL is implemented in the TOCSY experiment. If the DISPEL sequence is included before the TOCSY mixing, transfer will only be observed from protons bonded to ^{12}C. Likewise, if DISPEL is included after the mixing time, the only transfer observed will be to protons bonded to ^{12}C. If DISPEL is implemented before and after the TOCSY mixing, the spectrum will only contain signals that started and ended up on ^{12}C bonded protons. By running all four combinations of DISPEL on/off and TOCSY in a single interleaved experiment, the resulting spectra can be manipulated to only show peaks arising from and ending up on specific sites, such as $^{12}CH \rightarrow {}^{12}CH$, $^{12}CH \rightarrow {}^{13}CH$, $^{13}CH \rightarrow {}^{12}CH$ and $^{13}CH \rightarrow {}^{13}CH$. After correcting for differences in relaxation of ^{12}C- and ^{13}C-attached protons, the cross peaks in DISPEL-TOCSY spectra can be integrated to determine the levels of ^{13}C enrichment of individual metabolites in a site-specific manner.

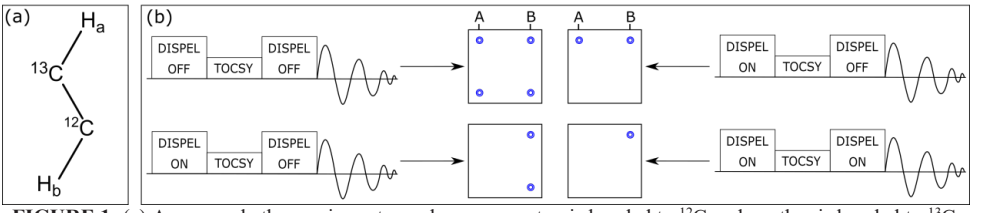

FIGURE 1. (a) An example three spin system where one proton is bonded to ^{12}C and another is bonded to ^{13}C. (b) Possible combinations of the DISPEL and TOCSY sequences showing the transfers that would be observed for the system in (a).

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FIRST ALGAL WHOLE CELL ^{13}C SOLID-STATE NMR GLYCAN ASSIGNMENT AND QUANTIFICATION

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In cells, carbohydrates can exist as pure carbohydrate polymers for storage, or associated with lipids or proteins, in a wide variety of cellular compartments such as the cell wall, cytoplasm or other organelles[1]. For example, the microalgae *Parachlorella CK-5* and *Chlamydomonas reinhardtii* have been extensively studied for photosynthesis and food supplementation - two fields of research closely associated with glycan production. To investigate glycosylation in living organisms, it is necessary to detect and quantify glycans in native cellular environment but the most used technique to analyze glycan is mass spectrometry and needs sample destruction.

In this work, we present how ^{13}C ssNMR can be used to precisely determine glycan compositions in whole-cell samples, mainly using ^{13}C-^{13}C INADEQUATE, due to low dispersion of glycan resonances. Here we describe acquisition and processing conditions in which INADEQUATE[2] is the most quantitative in cell to monitor glycan variations. We show that whole-cell SS-NMR of *P. CK-5* and *C. reinhardtii* reveals a large amount of glucose mostly contained in starch grains. Also, using *C. reinhardtii* mutants, we demonstrate that whole-cell ssNMR is able to follow starch synthesis in cell[3]. Moreover, in *P. CK-5* we assigned several other glycans probably associated to the cell wall of *P. CK-5*. Altogether, this work is the first to propose an atomistic view of glycan composition of whole algal cells. This approach could be applied to a variety of other biological samples, including human cells.

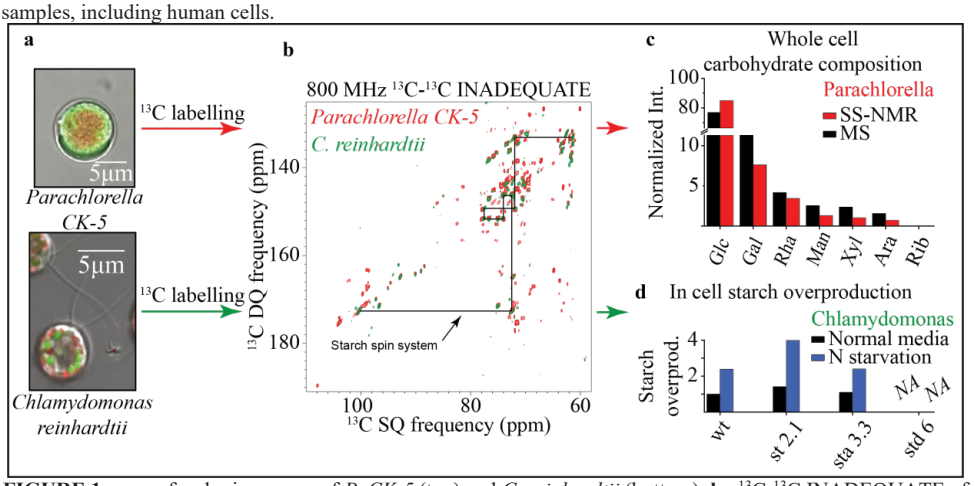

FIGURE 1. a- confocal microscopy of *P. CK-5* (top) and *C. reinhardtii* (bottom). **b**– ^{13}C-^{13}C INADEQUATE of whole cell *P. CK-5* (red) and *C. reinhardtii* (green). **c**– Quantifications of glycans using MS and SS-NMR in *P. CK-5*. **d**- Comparison of starch accumulation in different *C. reinhardtii* strains using whole cell SS-NMR. Wild-type (*wt*), amylopectin-rich (*st 2.1*), amylose-rich (*std 3.3*) and starchless (*std 6*) strains.

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Characterization of ciprofloxacin water solution confined in single wall carbon nanotubes

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One of the promising materials for drug delivery are carbon nanotubes, due to their specific physicochemical properties which depends on their size, metallic properties or length. These nanomaterials can be combined with various drugs or antibiotics, which makes them great tools for nanomedicine[1]. In this work we study the confined water solution of ciprofloxacin in single wall carbon nanotubes of two diameter – 1.7 nm and 30 nm. Many publications recently show that CNTs reveal antimicrobial effect. To characterize carbon nanotubes and influence of ciprofloxacin on their properties we used isotherm adsorption method, Raman spectroscopy and FT-IR method. The determination of pore volume was done using adsorption isotherm. Analysis of Raman spectroscopy of the carbon nanotubes shows that the higher amorphousness of carbon nanotubes, which increase bio-capability of materials, is associated with their semiconductor's properties. We used FT-IR spectroscopy to define the structure of confined ciprofloxacin, and we can assume what vibrations of functional group in the ciprofloxacin molecule have been suppressed by confining in the pores. On the basis of our FTIR results we can design the most likely alignment of the molecule of ciprofloxacin confined in single wall carbon nanotubes. We observed the effect of extinguishing of the molecule vibrations in the core of the molecule [2], while the biologically active bonds vibrations are slightly shifted towards lower frequencies relatively to the CPX bulk.

Acknowledgements
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STUCTURAL EFFECTS INDUCED BY GADOLINIUM IONS IN ALUMINOSILICATE CORE-SHELL STRUCTURES STUDIED BY SOLID-STATE NMR AND EPR SPECTROSCOPIES

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New nanostructured core-shell aluminosilicate microspheres doped with different concentrations of gadolinium ions are reported. Chemical synthesis of structures is based on Stöber method for silica core, and electrostatic attraction for nucleation of shell [1]. Structural changes determined in samples by increasing the content of gadolinium were studied by several solid-state NMR spectroscopy methods (e.g ^{29}Si, ^{29}Si CP, ^{27}Al, ^{27}Al 3Q MAS NMR) and EPR spectroscopy. Stable amorphous microspheres with gadolinium ions on the outermost layer of structures were obtained, being promissing materials that can be used as contrast agents in magnetic resonance imaging (MRI).

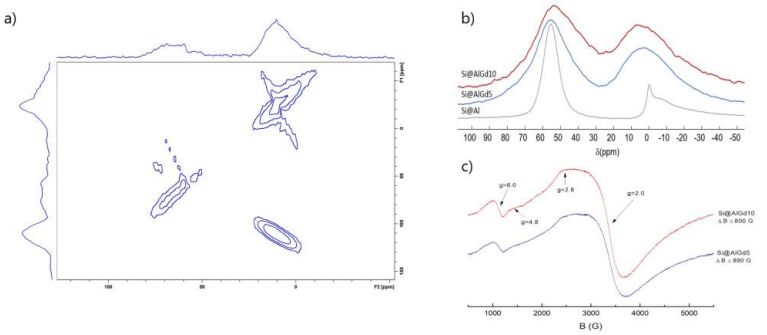

FIGURE 1. ^{27}Al 3Q MAS NMR of AlGdx shell (a), ^{27}Al MAS NMR (b) and EPR spectra (c) of core-shell structures with different content of gadolinium.

When strong physical interactions take place, wonderful stories can born, and NMR spectroscopy helps us to understand them! Today was also helped by his sister, EPR spectroscopy.

Acknowledgements
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The effect of water and impurities traces on properties of DBU-based protic ionic liquids

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Protic ionic liquids (PILs) are an environmentally friendly alternative to conventional organic solvents due to their unique physicochemical properties such as low flammability, low vapor pressure, high ionic conductivity, and high thermal stability. Usually, PILs are synthesized by a simple neutralization route. In theory, this simple one-step preparation method does not require demanding purification steps. However, the present work evidenced a strong impact of the purification method on the properties of the PILs samples. The PILs studied were based on the 1,8-diazabicyclo-[5,4,0]-undec-7-ene (DBUH) cation and two anions from the strong acids (trifluoromethanesulfonate, TFO^-) and bis(trifluoromethanesulfonyl)imide $TFSI^-$). The unpurified PILs were synthesized by a neutralization reaction. Subsequently, the purification method using activated charcoal and ethyl acetate as solvent was performed to produce the purified samples. The natural abundance ^{15}N NMR spectra reveal that the impurities and/or traces of water affect the residence time of the acidic proton in the DBUH cation only in the imide system. PGSE-NMR diffusion experiments pointed out that the transport properties of the DBUH-TFO are influenced by impurities and/or traces of water. Conversely, the diffusion profile of DBUH-TFSI samples remains constant in both circumstances. The nature of such differences in terms of molecular interactions and transport properties is currently under further investigation in our laboratories.

NMR and EPR characterization of V_2O_5 as a cathode material for high-capacity Li-ion batteries

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Li-ion batteries are the key technology for the electrification of the transport sector. Their enhancement requires fundamental understanding of the battery chemistry involving solid-state and interface reactions and processes. NMR and EPR were succesfully applied to investigate battery materials in many cases [1,2], however, mostly independent from each other. Here, both techniques are applied to investigate the cathode material V_2O_5. We exploit the strengths of EPR to target dilute surface defects and monitor redox reactions, and the strengths of NMR to identify phase transitions and the local surrounding of the nuclei under investigation.

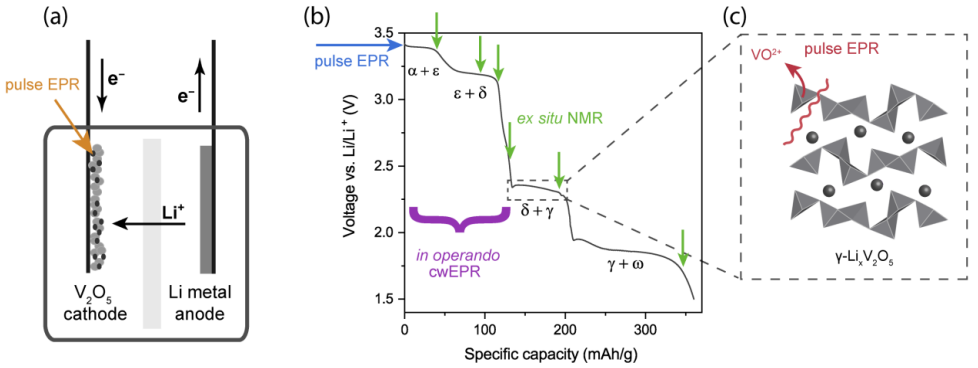

FIGURE 1. Overview of electrochemical testing of V_2O_5 cathodes in Li-ion batteries. Applied magnetic resonance techniques are indicated in color. (a) Schematic of the used battery setup with a V_2O_5 working electrode, a lithium metal counter and reference electrode, and a seperator. EPR-active defects are introduced through cathode film preparation. (b) Electrochemical discharge profile showing voltage plateaus that indicate distinct phase transitions. $Li_xV_2O_5$ phases are indicated with greek letters. (c) Upon the δ -> γ phase transition, vanadyl ions are released from the bulk cathode.

Acknowledgements
We thank P. P. M. Schleker and E. Veroutis for initial help with NMR experiments.

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CAN MORPHINE BE REPLACED? STRUCTURAL STUDIES OF MODIFIED OPIOID PEPTIDES

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Opioid peptides are compounds that bind to opioid receptors, causing, inter alia, an analgesic or narcotic effect. The first opioid peptide - enkephalin - was discovered by J. Hughes and H. Kosterlitz in 1975 [1]. As a result interest in opioid peptides began. Among them were dermorphins, endomorphins and morphiceptins. Nowdays we know the biological activity of opioid peptides is similar to morphine and often devoid of its side effects. Different strategies have been used to improve the properties of the peptides to make them more amenable as therapeutics, such as cyclization or incorporation of unnatural amino acids within the peptide sequence.

The compounds we study are cyclic analogues of enkephalin, dermorphine, endomorphine and morficeptin. Our peptides were cyclized through aromatic rings. The structures of modified opioid peptides were calculated with XPLOR [2] basing on through-space restraints obtained from analysis of two-dimensional NMR spectra. The elucidated structures of the modified opioid peptides are part of structure-activity relationship studies. Our goal is to understand what structural features govern the opioid peptide selectivity and the activity towards a particular opioid receptor. The linkage of the calculated structures to the biological activity of these peptides can determine the "active conformation", which is the conformation that peptides assume when binding to the opioid receptor. The end goal of these studies is to help in designing new, better drugs.

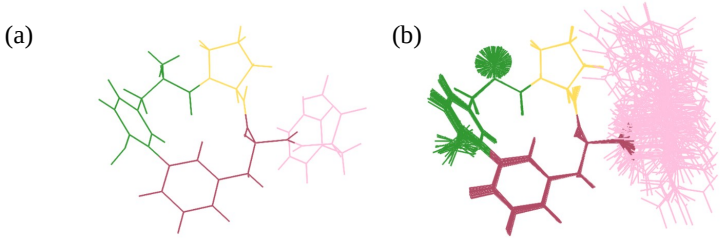

FIGURE 1. The lowest-energy conformer (a) and ensemble of the 50 lowest-energy structures (b) of endomorphin analog H-(cyklo-o,m)-[Tyr-Pro-Phe-Phe]-NH_2 . Proline – yellow, tyrosine – green, phenylalanine – pink (the former phenylalanine is darker and the subsequent one is brighter).

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RELAXATION DISPERSION EXPERIMENTS USING LONG-LIVED NUCLEAR SPIN ORDER

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Given their enhanced lifetime, long-lived states(LLS)[1] and coherences(LLC's)[2] can open new time windows for the study of exchange phenomena by relaxation dispersion. LLS can have relaxation time constants T_{LLS} of tens of seconds in the case of protons and up to tens of minutes for heteronuclei, which favours the investigations of slow exchange dynamics in molecules[3]. We show the results of of our investigationsof relaxation dispersion methods coupled with long-lived spin order as the initial magnetization source. LLS were sustained using eitherCarr-Purcell-Meiboom-Gill [4], [5] -type pulse sequences or spin-locking with variable amplitudes[6] in order to compare the efficiency of refocussing and locking pulse sequences.

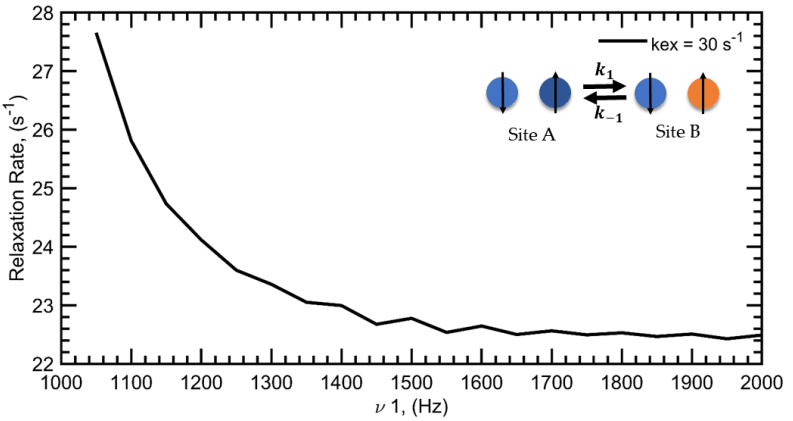

FIGURE 1. Simulated relaxation dispersion profile of a long-lived state generated in a BPTI's Tyr35 model of two-spin system ($\Delta\upsilon^{A}_{IS}$=36 Hz; $\Delta\upsilon^{B}_{IS}$=356 Hz; J_{IS}=8.3 Hz) sustanined with continuous radio-frequency and different amplitudes of the locking field (ν1). The color for the two spins in different sites indicates similar or different chemical shifts.

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REGULATION OF CONFORMATIONAL DYNAMICS OF THE HUMAN A_{2A} ADENOSINE RECEPTOR BY ENDOGENOUS PHOSPHOLIPIDS AS VIEWED BY ^{19}F NMR IN AQUEOUS SOLUTIONS

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G protein-coupled receptors (GPCRs) are sensory proteins that regulate many physiological processes and are targeted by one-third of current FDA-approved drugs. The function of GPCRs is closely related to their structural plasticity, which has been investigated by NMR spectroscopy [1]. Using ^{19}F NMR in aqueous solutions, we investigated the structural mechanisms by which anionic lipids regulate the conformational dynamics of the human A_{2A} Adenosine Receptor ($A_{2A}AR$) in lipid nanodiscs. Precise control of the lipid composition within the nanodiscs permitted NMR measurements over a large range of different ratios of zwitterionic and anionic lipids. Lipid compositions were verified by ^{31}P NMR measurements, and the receptor was confirmed to be folded and functional across the entire range of lipids in nanodiscs. In previous NMR studies of $A_{2A}AR$ in detergent micelles, a ^{19}F probe located at the intracellular surface of helix VII was shown to be sensitive to changes in the receptor conformation associated with different efficacies of bound drugs [2]. Using the same NMR probe, we measured ^{19}F spectra for $A_{2A}AR$ complexes with drugs of different efficacies across a range of different lipid compositions. While spectra of $A_{2A}AR$-antagonist complexes did not change as a function of lipid composition, we observed striking differences in spectra of $A_{2A}AR$-agonist complexes upon varying the amount of anionic lipids present in nanodiscs. A structural basis for this observation was investigated with $A_{2A}AR$ variants containing amino acid replacements of charged residues present at the intracellular surface. ^{19}F NMR data of tertiary complexes of $A_{2A}AR$ and a partner G protein further corroborate the role of charged lipids in regulating function-related conformational dynamics. The presented data show an intriguing synergy among drug efficacies, lipid composition and partner signaling proteins that provide a new window into GPCR signaling in cellular environments.

Acknowledgements
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Block copolymer poly(ethylene oxide)-b-polystyrene studied by NMR, DSC, WAXS and AFM

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The development of lithium-ion batteries has greatly contributed to the advanced development of the broad field of electronics [1]. Their definitely higher efficiency, lower mass and the output current density significantly exceeded the older technologies (e.g. nickel-cadmium cells) [2,3]. The research goal of this project is to use various NMR methods to characterize the phase behavior and self-assembly in selected block copolymers in the context of further studies of lithium-doped systems. Ultimately this could contribute towards development of novel, efficient and environment-friendly (without organic solvent) batteries.

In order to study the self-organization of block copolymers, structural studies were carried out using two complementary techniques i.e. NMR spectroscopy and AFM to determine the sizes of the domains. Moreover, DSC and WAXS techniques were used to examine the phase transitions and the degree of crystallinity. The conducted research will allow for further structural analysis of systems based on admixtures of lithium ions in order to develop more ecological and non-toxic dry electrolytes for applications in the field of lithium-polymer batteries.

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"Zakopane"
AMPERE NMR SCHOOL 2021
VIRTUAL EVENT

PROGRAMME
June 21-23 2021, Poznan, Poland

Country/Time Zone					MONDAY, June 21st	TUESDAY, June 22th	WEDNESDAY, June 23rd
USA/Canada(west)(CEST-9)	Brazil (CEST-5)	USA/Canada(east)(CEST-6)	India (CEST+3:30)	CEST			
23.45-0.00	3.45-4.00	2.45-3.00	12.15-12.30	8.45-9:00	**Stefan Jurga** *welcome*		
0.00-0.40	4.00-4.40	3.00-3.40	12.30-13.10	9.00-9.40	**Bernhard Blümich** Molecular Dynamics by NMR for Materials Testing: Relaxation, Exchange and MRI	**Ilya Kuprov** Why are some nuclei non-spherical and what does that have to do with spin?	**Beat Meier** Fast MAS and Biomolecules
0.40-1.20	4.40-5.20	3.40-4.20	13.10-13.50	9.40-10.20	**David Lurie** Basic Physics of MRI and Research on Fast Field-Cycling MRI	**Matthias Ernst** Residual Line Width in MAS Solid-State NMR	**Jadwiga Tritt-Goc** NMR studies of solid cellulose-based proton conductors
1.20-2.00	5.20-6.00	4.20-5.00	13.50-14.30	10.20-11.00	**Julia Krug** The higher, the better?! The promises and perks of ultra-high field MRI at 22.3 T	**Olivier Lafon** Solid-state NMR of quadrupolar nuclei and their neighbors	**Wiktor Koźmiński** High dimensionality and high resolution NMR experiments for biomolecules
2.00-2.40	6.00-6.40	5.00-5.40	14.30-15.10	11.00-11.40	**Daniel Topgaard** Diffusion-relaxation correlation MRI	**Janez Dolinšek** High-entropy alloys	**Fabien Ferrage** Exploring scalar couplings and chemical exchange from low to ultra-high fields
3.00-4.00	7.00-8.00	6.00-7.00	15.30-16.30	12:00-13.00	***Poster Session***		***Break***
4.00-5.00	8.00-9.00	7.00-8.00	16.30-17.30	13.00-14.00	***Lunch***		
5.00-5.40	9.00-9.40	8.00-8.40	17.30-18.10	14.00-14.40	**Anton Duchowny** NMR Studies of Complex Samples Using Compact Instruments	**Michał Bielejewski** The kinetics of proton transport and thermal processes in anhydrous nanocomposite ptoton conductor based on cellulose	**Esteban Anoardo** Field-Cycling MRI relaxometry
5.40-6.20	9.40-10.20	8.40-9.20	18.10-18.50	14.40-15.20	**Janez Stepišnik** Insight into the details of molecular translation dynamics in liquids by NMR gradient spin echo method	**Claudia Schmidt** NMR studies of polymer gel electrolytes	**Danuta Kruk** Molecular dynamics by means of NMR relaxometry
6.20-7.00	10.20-11.10	9.20-10.00	18.50-19.30	15.20-16.00	**Ville-Veikko Telkki** Ultrafast multidimensional relaxation and diffusion measurements	**Vladimir Chizhik** Conformational and aggregation behavior of some surfactants in aqueous solutions by 1H and 13C NMR	**Kay Saalwaechter** Proton low-field NMR for the study of (bio)macromolecular dynamics or: understandin 1H T2
7.00-7.40	11.00-11.40	10.00-10.40	19.30-20.10	16.00-16.40	**Siegfried Stapf** Measuring NMR relaxation times – What can possibly go wrong ?	**Anja Böckmann** Carbon- and proton-detected solid-state NMR sequential assignments and applications to fibrils and membrane proteins	**Alex MacKay** Are Relaxation Times Useful in Medicine?
							CONFERENCE CLOSING