Conference Program (as of March 27, 2023)

This Conference Program is subject to change. It is intended to serve as a reference guide only.

The online planner and mobile app tools will be available in late March.
These tools are for registered attendees and will be updated regularly as changes occur.

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64th ENC PROGRAM (as of March 27, 2023)
This program is subject to change.

SATURDAY, APRIL 15

Pre-Conference Vendor Meetings
Times & Locations Vary

All-day, **IVAN Meeting**, Oak Shelter bldg on Asilomar campus.

13:00-17:00, **Bruker Workshops**, Monterey Plaza & Spa - **Learn More & RSVP Today**

SUNDAY, APRIL 16

Pre-Conference Vendor Meetings
08:00-14:15

8:30 - 12:30, **Bruker Symposium**, Monterey Plaza & Spa, **Learn More & RSVP Today**

10:30, **JEOL NMR Mini-Symposium**, Fred Farr bldg on Asilomar Campus - **Learn More & RSVP Today**

12:15-14:15, **ACD/Labs Meeting**, Nautilus bldg on Asilomar Campus.

Conference Check-in/ Name Badge Pick Up
10:00-18:00, Triton

YOUNG SCIENTISTS' SYMPOSIUM (YSS)
14:30-16:00, Merrill Hall

YSS 14:30, **Jihyun Kim** (Weizmann Institute of Science) New cross-polarization schemes for heteronuclear transfers involving labile protons in biomolecular NMR		YSS 15:15, **Shannon L Eriksson** (Duke University) Improving SABRE Polarization Through Three-Dimensional Magnetic Field Manipulation
YSS 14:45, **Wenkai Zhu** (University of Pittsburgh) Visualizing Proteins in Mammalian Cells by 19F NMR Spectroscopy		YSS 15:30, **Haiyan Mao** (University of California, Berkeley) Scalable nanoporous networks for carbon capture via solid-state NMR spectroscopy
YSS 15:00, **Nuwandi M. Ariyasingha** (Wayne State University) Efficient Polarization Re-distribution in Hyperpolarized Propane Produced Via Pairwise Parahydrogen Addition		YSS 15:45, **James J. Kimball** (Florida State University) Broadband Adiabatic Inversion Cross-Polarization: Theory and Applications

WELCOME RECEPTION with Exhibit Booths & Young Scientist Posters – **All Attendees Invited**
16:00-18:00, Fireside Pavilion

ASILOMAR LODGER DINNER for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees may purchase a lunch ticket bundle or dinner ticket online by April 7.
18:00 – 19:00, Crocker Dinning Hall

OPTIONAL Vendor Hospitality Suites (following reception)

MONDAY, APRIL 17, 2023

Early Morning Lecture Series (for Students & ALL who wish to learn!)
07:00-07:50, Merrill Hall

07:00-07:50 **Understanding NMR Spectroscopy** Part 1 of 4

Presenter: James Keeler

Would you like to deepen or brush up your understanding of NMR theory? Join us for a morning lecture series by **James Keeler** (University of Cambridge), author of *Understanding NMR Spectroscopy*. These lectures assume only a modest prior knowledge of NMR theory and will cover some key topics of wide interest to budding NMR spectroscopists. Topics will include: Energy levels, Hamiltonians and operators; Introducing and using product operators; Relaxation; Coherence selection by phase cycling and field gradients

ASILOMAR LODGER BREAKFAST for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees cannot purchase breakfast tickets, but you can purchase lunch and dinner tickets by April 7.
07:30-09:00, Crocker Dining Hall

MOA: Laukien Prize Session (plenary session)
08:45 -10:10, Merrill Hall
Len Mueller and Daniel Raftery presiding

COFFEE BREAK with Exhibit Booths and Posters, Fireside Pavilion
10:10-10:45

MOD: Hyperpolarization & Eclectica (parallel session)
10:45-12:35, Merrill Hall

10:45-11:10 **Tracking Degradation in Commercial Li Batteries with High Chemical and Temporal Resolution**

Presenting Author: Lauren Marbella

Lauren Marbella (Columbia University)

Anode-free batteries offer the highest specific energy density for Li-based batteries, but practical application is plagued by the growth of high surface area Li deposits. The presence of these Li filaments is strongly correlated with the formation of dead (electrochemically inactive) Li that leads to low Coulombic efficiency (CE) and serious safety concerns. Yet, electrifying large-scale modes of transportation will rely on energy dense technologies. Commercial batteries present unique challenges because the way that electrodes are stacked inside of a multilayer cell impact Li deposition due to differences in pressure in the system. I will discuss our efforts to use operando NMR spectroscopy to probe buried interfaces in these systems and quantitatively detect Li growth, dead Li, and electrolyte decomposition.

MONDAY, APRIL 17, 2023 - *continued*

11:10-11:30 **Toward Efficient SABRE Hyperpolarization of Tin-117 Nuclear Targets for Neutron Optics-Based Time-Reversal Symmetry Investigations**

Presenting Author: Abubakar Abdurraheem

Abubakar Abdurraheem (Wayne State University); Shahabuddin Alam (Southern Illinois University); Anthony Petrilla (Southern Illinois University); Boyd Goodson (Southern Illinois University); Roman Shchepin (South Dakota School of Mines and Technology); Michale Snow (Indiana University/CEEM); Eduard Chekmenev (Wayne State University)

Tin-117 has been identified as a nucleus of interest for envisioned neutron-optics based searches for time-reversal invariance violation (TRIV). However, such experiments would require the sustained production of large quantities of hyperpolarized 117Sn nuclei on a neutron beam line. We report on our pilot efforts to prepare HP states in another spin-1/2 tin isotope, 119Sn, using SABRE hyperpolarization and detection with a benchtop NMR spectrometer capable of detecting both tin isotopes. The spectrometer detects 119Sn with greater sensitivity, facilitating substrate hyperpolarization screening with the rationale that our observations may be expanded to 117Sn hyperpolarization. 119Sn polarization is relayed through 1H substrate spins, and can be hyperpolarized in tin-functionalized pyrimidine, imidazole, and oxazole rings; oxazole derivatives yielded the highest proton polarization.

11:30-11:50 **First demonstration of SABRE hyperpolarized 1-13C Pyruvate metabolism detected in vivo**

Presenting Author: Austin Browning

Austin Browning (North Carolina State University); Keilian MacCulloch (North Carolina State University); Matt Rosen (MGH/Martinos Center); Eduard Chekmenev (Wayne State University); Boyd Goodson (Southern Illinois University); Thomas Theis (North Carolina State University); Patrick TomHon (Vizma Life Sciences); Carlos Dedesma (Vizma Life Sciences); Yi-Fen Yen (Harvard Medical School); David Bedoya (Harvard Medical School)

The first SABRE hyperpolarized in-vivo signal is shown here with 1-13C Pyruvate as the metabolite. The 1-13C Pyruvate signal last for over a minute, with a 20° flip angle, while the metabolic conversion to 1-13C Lactate is highlighted, with a 30° flip angle. This work was performed using our variable field MRI, set to 1.5 T. The next step in this work is moving to a biocompatible solution to allow for survival based studies utilizing SABRE and CSI to monitor diseased and healthy models for location and conversion rates of pyruvate and lactate. What this current work shows is a clear "giant leap" and path forward for the use of the simple and cost-effective method of SABRE for in-vivo applications.

11:50-12:10 **Co-hyperpolarized [13C,15N2]Urea + [1-13C]Pyruvate for Perfusion and Metabolic Imaging of Human Abdomen**

Presenting Author: Yaewon Kim

Yaewon Kim (University of California); Hsin-Yu Chen (University of California); Tanner Nickles (University of California); Jeremy Gordon (University of California); Peder Larson (University of California); Xiaoxi Liu (University of California); Louise Magat (University of California); Philip Lee (University of California); Daniel Gebrezgiabhier (University of California); Cornelius von Morze (Washington University); Daniel B. Vigneron (University of California); Michael A. Ohliger (University of California)

[13C,15N2]Urea and [1-13C]pyruvate were co-polarized using dynamic nuclear polarization and injected into healthy volunteers for simultaneous imaging of perfusion and metabolism in the abdomen. Whole-abdomen dynamic images were successfully obtained, and the distribution of urea was compared to pyruvate and its metabolites. While relative intensities in various organs were similar between urea and pyruvate, the time-to-peak of urea was earlier than pyruvate in kidneys and spleen. These effects may reflect differences in vascularity, permeability and metabolism for the two agents. This study investigated the first use of co-polarized HP pyruvate and urea in the human abdomen and demonstrated clinical-research value for simultaneous perfusion and metabolic imaging in abdominal organs and future MR molecular imaging of tumors and metabolic diseases.

12:10-12:35 **MRI of Roots in the Greenhouse and Agricultural Field**

Presenting Author: Hilary Fabich

Hilary Fabich (ABQMR, Inc.)

Analyzing plants above the ground is routine in plant breeding programs. Though the roots are an essential part of the plant, there is no convenient method for studying intact roots in natural soil. We have developed low-field MRI systems for use both in the greenhouse and agricultural field. The systems can be transported around the agricultural field and acquire 3D images of intact roots in natural soils. In addition to hardware development, we have been working to optimize pulse sequences to separate the signal of soil water from that of root water. Being able to image the root architecture provides useful information for plant breeders as they breed plants for specific climates and disease resistance.

MONDAY, APRIL 17, 2023 - *continued*

MOE: MRI: Methods and Applications (parallel session)
10:45-12:30, Chapel

10:45-11:10 **K2S Challenge: From Undersampled K-Space to Automatic Segmentation**

Presenting Author: Valentina Pedoia

Valentina Pedoia (UCSF)

Image reconstruction and downstream tasks have typically been treated independently by the image processing community, but we hypothesized performing them end-to-end facilitate further optimization. To these ends, UCSF organized the K2S challenge, where challenge participants were tasked with segmenting bone and cartilage from 8X undersampled knee MRI acquisitions. Top challenge submissions produced high-quality segmentations maintaining fidelity to ground truth, but strong reconstruction performance proved not to be required for accurate tissue segmentation. This, in conjunction with there being no correlation between reconstruction and segmentation performance, confirmed reconstruction algorithms can be optimized for downstream tasks in an end-to-end fashion.

11:10-11:30 ***in vivo* Imaging Stroke via 3D Hyperpolarized Xenon MRI with hundreds micron resolution**

Presenting Author: Haidong Li

Ming Zhang (National Center for Magnetic Resonance in Wuhan, Wuhan Institute of Physics and Mathematics, Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Haidong Li (National Center for Magnetic Resonance in Wuhan, Wuhan Institute of Physics and Mathematics, Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Hongchuang Li (National Center for Magnetic Resonance in Wuhan, Wuhan Institute of Physics and Mathematics, Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Xiuchao Zhao (National Center for Magnetic Resonance in Wuhan, Wuhan Institute of Physics and Mathematics, Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Xiaoling Liu (National Center for Magnetic Resonance in Wuhan, Wuhan Institute of Physics and Mathematics, Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Yeqing Han (National Center for Magnetic Resonance in Wuhan, Wuhan Institute of Physics and Mathematics, Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Xianping Sun (National Center for Magnetic Resonance in Wuhan, Wuhan Institute of Physics and Mathematics, Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Chaohui Ye (National Center for Magnetic Resonance in Wuhan, Wuhan Institute of Physics and Mathematics, Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Xin Lou (Department of Radiology, Chinese PLA General Hospital); Xin Zhou (Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences)

Stroke is the second leading cause of death worldwide. 1H MRI has been increasingly used in the diagnosis and management of acute stroke. However, 1H MRI has a large background signal from biological tissue. Here, we demonstrated the feasibility of isotropic 3D high-resolution hyperpolarized 129Xe brain MRI and its potential for assessing the perfusion abnormalities caused by stroke. Permanent middle cerebral artery occlusion (MCAO) was performed on the rat, then 129Xe and 1H MRI were performed in sequence. Finally, a 3D high spatial-resolution129Xe brain image was obtained within 5 min, and hypoperfused areas observed with 1H and 129Xe images were well-matched. These results indicate that 3D 129Xe brain MRI is a promising method for stroke diagnosis.

11:30-11:50 **Using HP 129Xe MRI to Validate Proteomics Biomarkers in Early Cystic Fibrosis Lung Disease**

Presenting Author: Zackary Cleveland

Zackary Cleveland (Cincinnati Children's Hospital Medical Center); Abdullah S. Bdaiwi (Cincinnati Children's Hospital Medical Center); Matthew Siefert (Cincinnati Children's Hospital Medical Center); Emily Skala (Cincinnati Children's Hospital Medical Center); Assem G. Ziady (Cincinnati Children's Hospital Medical Center)

Cystic fibrosis (CF) is a progressive, historically fatal disease that has seen lifespans increase significantly due to improved therapies. However, these improvements have created a "victim of success" situation, where standard-of-care tools to can no longer monitor lung disease severity in many patients. Hyperpolarized (HP) 129Xe MRI can detect impaired ventilation years before changes the standard clinical tool—spirometry—but it is limited to a handful of specialize academic medical centers. Proteomic biomarkers from high-precision mass spectrometry can accurately forecast CF lung disease progression, but these biomarkers have only been validated in patients with spirometrically detectable lung disease. Here we use HP 129Xe MRI to validate proteomic markers in early severity and progression in CF lung disease.

MONDAY, APRIL 17, 2023 - *continued*

11:50-12:10 **The Effect of Elimination of Systematic Errors on the Distributions of Diffusion Tensor Imaging Metrics in Automatically Segmented Regions of White and Grey Matter of The Left and Right Hemisphere**

Presenting Author: Weronika Mazur

Weronika Mazur (AGH University of Science and Technology); Julia Lasek (AGH University of Science and Technology); Artur Krzyak (AGH University)

Neurodiseases can be assessed by diffusion tensor imaging (DTI) metrics in certain white (WM) and grey matter(GM) structures. In this study, we examined how the elimination of systematic errors (by using B-matrix spatial distribution, BSD) and enhancing SNR (by increasing number of excitations from 4, to 16 and 64, called NEX4, NEX16 and NEX64, respectively) change the distributions of mean diffusivity (MD) and fractional anisotropy (FA) in 95 automatically segmented WM and GM regions applying deep learning algorithm depending on the b-value. Results showed that without BSD application, systematic errors can cause artificial asymmetry between MD and FA values in the right and left hemisphere and GM and WM. b-value=2000 s/mm2 introduces strong kurtosis and skewness eliminated by BSD.

12:10-12:30 **Accurate Tomographic Magnetic Resonance T1 Mapping at Ultra-low Field**

Presenting Author: Sheng Shen

Sheng Shen (1. A. A. Martinos Center for Biomedical Imaging, Department of Radiology, MGH, 2.Harvard Medical School); Neha Koonjoo (1. A. A. Martinos Center for Biomedical Imaging, Department of Radiology, MGH, 2.Harvard Medical School); Stephen E. Ogier (1.University of Colorado, 2. National Institute of Standards and Technology); Kalina V. Jordanova (National Institute of Standards and Technology); Kathryn E. Keenan (National Institute of Standards and Technology); Matthew S. Rosen (1. A. A. Martinos Center for Biomedical Imaging, Department of Radiology, MGH, 2.Harvard Medical School, 3. Dept. of Physics, Harvard University)

Motivated by the growing contemporary interest in low field (<100 mT) and ultra-low field (<10 mT) MRI scanners, we have developed an accurate T1 mapping approach for use at ultra-low field that maintains its accuracy even in the low signal-to-noise ratio (SNR) regime at 6.5 mT. We describe here the use of a variable flip angle (VFA) method combined with accurate B1 correction. A hybrid B1 mapping method was used to increase the accuracy of low flip angle maps and thus increase the accuracy of T1 maps. We validated that our T1 mapping approach in a human tissue-mimic phantom has a deviation lower than 10% when compared to the ground truth obtained using an inversion recovery (IR) spectroscopic method.

MOF: Small Molecules: Emerging Methods and Applications (parallel session)
10:45-12:35, Nautilus

10:45-11:10 **Insights into Fluorinated Drug Substance and Drug Product via 19F Solid-State NMR Spectroscopy**

Presenting Author: Joe Lubach

Joe Lubach (Genentech, Inc.)

High resolution characterization of pharmaceutical solid dosage forms represents an ever-challenging and ever-changing problem facing pharmaceutical scientists. 19F solid-state NMR is becoming increasingly utilized around the community due to its sensitivity, exquisite selectivity, and prevalence in modern drug candidates. We will examine a few of the advantages, and drawbacks, of 19F solid-state NMR, and a variety of ways it can be exploited in solid form analysis. These include simple crystal form identification, crystallographic inequivalency evaluation, water content determination, and quantitative solid form measurements in complex drug products. Deeper understanding of drug particles in the presence of an excipient matrix offered by fluorine NMR can provide valuable insight into dosage form design for more robust drug products and processes.

11:10-11:30 **SHARPER-DOSY: Sensitivity Enhanced Diffusion-Ordered NMR Spectroscopy**

Presenting Author: Dusan Uhrin

George Peat (University of Edinburgh); Patrick J. Boaler (University of Edinburgh); Claire L. Dickson (Oxford Instruments); Guy C. Lloyd-Jones (University of Edinburgh); Dusan Uhrin (University of Edinburgh)

A liquid-state NMR method, which increases the sensitivity of the existing techniques for the measurement of diffusion coefficients of pure compounds by a factor of 10-100, is reported. The associated 10²-10⁴-fold time saving is achieved by removing the chemical shift separation and splittings due to J couplings. Diffusion coefficients are measured from a narrow singlet (which is invariant to magnetic field inhomogeneity) obtained by signal acquisition embedded within short spin-echoes. The proposed experiment incorporates solvent suppression and signal selection. Using high field cryoprobe NMR spectrometers, SHARPER-DOSY makes it possible to measure in a matter of minutes diffusion coefficient of medium-size organic molecules using as little as few hundred nanograms of material.

MONDAY, APRIL 17, 2023 - *continued*

11:30-11:50 **Long-lived states in achiral aliphatic chains**

Presenting Author: Geoffrey Bodenhausen

Kirill Sheberstov (ENS); Anna Sonnefeld (ENS); Geoffrey Bodenhausen (ENS)

Long-lived states involving typically four protons of neighbouring CH2 groups in achiral aliphatic chains can be excited by selective irradiation at one or several frequencies. Such experiments open interesting perspectives of delocalised long-lived states that are relatively insensitive to paramagnetic relation agents, can be boosted by hyperpolarization, generalised to mixtures for drug screening, and be unravelled by chemical shift MRI methods.

11:50-12:10 **Barriers to Proton Resolution in Solids**

Presenting Author: Daria Torodii

Daria Torodii (EPFL); Pinelopi Moutzouri (EPFL); Bruno Simes De Almeida (EPFL); Lyndon Emsley (EPFL)

We address experimentally the factors that limit proton linewidths in ultrafast MAS solid-state NMR experiments, which include residual dipolar coupling, anisotropic bulk magnetic susceptibility (ABMS), and distributions of chemical shifts. We find that in many organic solids the dipolar contributions account for about 20-40% of the 1H linewidth at 100 kHz MAS and can be reduced or removed by faster MAS or pure isotropic approaches. The remaining 60-80% of the linewidth is dominated by inhomogeneous interactions including ABMS and chemical shift distributions which are explored by dilution, deuteration and two-dimensional 1H-1H correlation experiments at 100 kHz MAS. Our findings open up a series of new pathways to ultra-high resolution 1H NMR in solids.

12:10-12:35 **Fast prediction of solution-state NMR parameters across a wide 3D chemical space**

Presenting Author: Craig Butts

Ben Honore (University of Bristol); Calvin Yiu (University of Bristol)

We present our next generation machine learning system, IMPRESSION-G2, capable of predicting isotropic and anisotropic NMR parameters. It achieves accuracies comparable to the best quantum chemical methods, but can complete predictions in milliseconds per molecule rather than hours or days. Crucially it accounts for stereochemical and conformational effects, making rapid 3D structure elucidation and verification practical and robust.

ASILOMAR LODGER LUNCH for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees may purchase a lunch ticket bundle or dinner ticket online by April 7.
12:45-14:00, Crocker Dinning Hall

POSTER SESSION with Exhibit Booths
14:00-15:45, Fireside Pavilion
See Poster Listings at end of document.

MOG: BioSolution: Condensed or Cellular (parallel session)
16:00-17:50, Merrill Hall

16:00-16:25 **Quenched H/D exchange NMR of inclusion bodies reveals significant native-like protein structure**

Presenting Author: Elizabeth Meiering

Elizabeth Meiering (University of Waterloo)

Protein aggregation is at the nexus of molecular processes crucial to aging, disease, and employing proteins for biotechnological and medical applications. While there has been considerable recent progress in determining structural features of protein aggregates formed in cells, owing to prevalent heterogeneity in aggregation, many aspects remain obscure. We report high-resolution analysis by quenched amide hydrogen/deuterium exchange NMR and complementary methods of cellular inclusion body aggregates for ALS-associated mutants of superoxide dismutase, engineered target binding proteins known as Adnectins or monobodies, and myoglobin. In contrast to prior studies reporting prominent amyloid in inclusion bodies, we find evidence for significant native-like structure. The results indicate an ensemble of protein self-association processes may contribute to IB formation.

MONDAY, APRIL 17, 2023 - *continued*

16:25-16:45 **Real-Time NMR Multiplexed GEF Assay Allowing the Monitoring of Multiple sGTPases Nucleotide Exchange from Cells and Organoids, Simultaneously**

Presenting Author: Genevieve Seabrook

Genevieve Seabrook (University Health Network-Princess Margaret Hospital); Teklab Gebregiworgis (Department of Biochemistry); Christopher B. Marshall (University Health Network-Princess Margaret Hospital); Tadateru Nishikawa (JEOL); Nikolina Radulovich (University Health Network-Princess Margaret Hospital); Ming Tsao (University Health Network-Princess Margaret Hospital); Mitsuhiko Ikura (University Health Network-Princess Margaret Hospital)

Small GTPases are regulators mediating important cellular functions. These sGTPases are often mutated in human cancers. We have developed a real-time multiplex NMR assay allowing the following of several sGTPases nucleotide exchange in a single experiment. Along with sGTPase proteins strategically selectively labeled, time-shared NMR methodology was used to reduce acquisition time. Analysis of sGTPases amides chemical shift changes, allowed us to identify residues that have been perturbed during the nucleotide exchange and the resulting structural changes within the sGTPases. A mixture of six sGTPases was used to assay GEF activities present in cells lysates and in organoids lysates. A combination of selective isotopic labeling and real-time, time-shared NMR experiments can be extended to other biological processes.

16:45-17:05 **Decoupling Protein Concentration and Aggregate Content Using Diffusion and Water NMR**

Presenting Author: Mark I. Grimes

Mark Grimes (University of Cambridge); Matthew Cheeks (AstraZeneca); Jennifer Smith (AstraZeneca); Fabio Zurlo (AstraZeneca); Mick D. Mantle (University of Cambridge)

Water proton nuclear magnetic resonance (wNMR) utilises the water transverse relaxation rate [$R_2(^1H_2O)$] to gain understanding about solutes. It has been used in this manner to obtain information about solution protein concentration and aggregate content, under both static and flow conditions. However, $R_2(^1H_2O)$ is influenced by both characteristics, so is unable to differentiate between them. In this work, the water diffusion coefficient [$D(^1H_2O)$] is used in conjunction with $R_2(^1H_2O)$ to separate these values. It has been demonstrated using three different model protein systems, in concentration ranges relevant for the maximum antibody titres found in fed batch bioreactors. This method allows for the rapid and facile determination of both protein concentration and aggregate content in a non-invasive manner.

17:05-17:25 **Long-lived Spin Order for Magnetization Transport: Overhauser Transfer and Stroboscopic Follow-up of Redox Reactions**

Presenting Author: Paul Vasos

Paul Vasos (ELI-NP/IFIN-HH); Florin Teleanu (ELI-NP/IFIN-HH); Adonis Lupulescu (ELI-NP/IFIN-HH); Adrian M. Voda (ELI-NP/IFIN-HH); Aude Sadet (ELI-NP/IFIN-HH)

Long-lived coherences (LLC's) and long-lived states (LLS) extend available timescales for magnetization transfer. Rotating-frame Overhauser transfer can be enhanced using LLC configurations. We measured LLC-ROE transfer between Ala-Hα and Gly-Hα1,2 in AlaGly. LLC-ROE transfer becomes more intense than via classical ROE for molecular rotational correlation times τc > 10 ns. LLC-ROE applications will be discussed for different symmetry configurations in peptides and for protein Lysozyme. Stroboscopic LLS is introduced for on-the-fly detection of molecular transformations on time-scales of tens of seconds. This is demonstrated for the follow-up of glutathione GSH/GSSG redox conversion starting from the initial polarization of GSH-Gly-Hα1,2, where lifetimes T_LLS of 16 s are reached. The stroboscopic LLS method is adapted for use with dissolution-DNP enhanced magnetization.

17:25-17:50 **Combining Laser and NMR for the Surface Analysis of Intrinsically Disordered Proteins**

Presenting Author: Jung Ho Lee

Jung Ho Lee (Seoul National University)

We present a photo-chemically induced dynamic nuclear polarization (photo-CIDNP) experiment suitable for the analysis of intrinsically disordered proteins (IDPs). Pulse stretching of laser pulses, band-selective decoupling of 13Cα, and simultaneous application of radiofrequency and laser pulses were implemented to quantitatively analyze the IDP surface at ultrahigh resolution. Comparative analysis with other surface accessibility methods validated the newly developed method and emphasized the importance of dye charge. Using the neutral riboflavin dye, surface accessibilities were measured to be nearly identical throughout the alpha-synuclein sequence. Divalent cations were shown to induce compaction of the C-terminal region and release of the N-terminal region of alpha-synuclein. This new method can be used as an orthogonal and independent method for investigating the overall IDP conformation.

MONDAY, APRIL 17, 2023 - *continued*

MOH: BioSolids I (parallel session)
16:00-17:45, Chapel

16:00-16:25 **Oxygen-17 NMR Studies of Proteins: Opportunities and Challenges**

Presenting Author: Gang Wu

Gang Wu (Queen's University)

One of the long-standing challenges in 17O NMR studies of proteins is the production of site-specifically or uniformly 17O-labeled proteins [1]. Recently, we demonstrated that it is feasible to achieve amino acid-type specific 17O-labeling of proteins via recombinant expression in an auxotrophic Escherichia coli strain [2]. This new approach allows incorporation of 17O isotopes into both the protein backbone and side chains. This opens up new opportunities for 17O NMR studies of proteins. In this talk, we will discuss this new 17O-labeling approach and present new 17O NMR data obtained with the latest NMR technologies including the uses of an ultra-high magnetic field (35.2 T) and a CryoProbe.

[1] G. Wu, Prog. Nucl. Magn. Reson. Spectrosco. 114/115, 135 (2019).

[2] B. Lin, I. Hung, Z. Gan, P.-H. Chien, H. L. Spencer, S. P. Smith, and G. Wu, ChemBioChem 22, 826 (2021).

16:25-16:45 **Biomolecular MAS NMR: some good reasons for spinning faster**

Presenting Author: Zhiyu Sun

Zhiyu Jeff Sun (Centre de Rsonance Magntique Nuclaire Trs Hauts Champs, Institut des Sciences Analytiques (UMR 5280 CNRS, Ecole Normale Suprieure de Lyon, Universit Claude Bernard Lyon 1), Universit de Lyon, 69100 Villeurbanne, France); Tanguy Le Marchand (Centre de Rsonance Magntique Nuclaire Trs Hauts Champs, Institut des Sciences Analytiques (UMR 5280 CNRS, Ecole Normale Suprieure de Lyon, Universit Claude Bernard Lyon 1), Universit de Lyon, 69100 Villeurbanne, France); Claire Ollier (Centre de Rsonance Magntique Nuclaire Trs Hauts Champs, Institut des Sciences Analytiques (UMR 5280 CNRS, Ecole Normale Suprieure de Lyon, Universit Claude Bernard Lyon 1), Universit de Lyon, 69100 Villeurbanne, France); Kumar Tekwani Movellan (Department of Chemistry and Biochemistry, University of Delaware, Newark, DE 19716, USA; Pittsburgh Center for HIV Protein Interactions, University of Pittsburgh, Pittsburgh, PA 15260, USA); Brent Runge (Department of Chemistry and Biochemistry, University of Delaware, Newark, DE 19716, USA; Pittsburgh Center for HIV Protein Interactions, University of Pittsburgh, Pittsburgh, PA 15260, USA); Kristof Grohe (Bruker BioSpin); Jochem Struppe (Bruker BioSpin); Sebastian Wegner (Bruker BioSpin); Angela Gronenborn (Department of Structural Biology, University of Pittsburgh School of Medicine, Pittsburgh, PA, 15261, USA); Tatyana Polenova (Department of Chemistry and Biochemistry, University of Delaware, Newark, DE 19716, USA; Pittsburgh Center for HIV Protein Interactions, University of Pittsburgh, Pittsburgh, PA 15260, USA); Guido Pintacuda (Centre de Rsonance Magntique Nuclaire Trs Hauts Champs, Institut des Sciences Analytiques (UMR 5280 CNRS, Ecole Normale Suprieure de Lyon, Universit Claude Bernard Lyon 1), Universit de Lyon, 69100 Villeurbanne, France)

The switch from the traditional MAS NMR approaches with 13C and 15N detection to 1H has accelerated the site-specific analysis of complex immobilized biological systems and opened the way to samples of higher molecular weight and only available in limited amounts.

We will take the moves from a critical analysis of recent literature data, share our first results on a prototypical Bruker 0.4 mm probe capable of rates exceeding 150 kHz and discuss the expected impact of fast MAS on resolution and sensitivity of NMR experiments on different classes of biomolecular samples.

16:45-17:05 **1H-31P Cross Polarization in White Matter Tissue: Towards New Contrast Mechanisms for MRI and Beyond!**

Presenting Author: Cariad-Arianna Knight

Cariad Knight (University of British Columbia); Alex Ensworth (University of British Columbia); Carl Michal (UBC)

MRI is in dire need of better myelin characterization tools. We present the use of solid-state NMR techniques to investigate myelin bilayers through direct detection of phospholipid phosphorous (31P) via dipolar coupled protons (1H) by cross polarization (CP), magnetization transfer (MT), and a hybrid MT-CP sequence. Results from porcine white matter indicate that CP provides a highly effective and specific myelin solid 31P filter in both static and magic angle spinning experiments. MT-CP experiments demonstrate magnetization exchanged between solid and aqueous 1H is detectable in subsequent 31P CP spectra. Additional experiments including wideline separation, spin echo double resonance and relaxation measurements provide further insight to myelin lipid health. This work enthusiastically supports development of a CP-based MRI contrast mechanism.

MONDAY, APRIL 17, 2023 - *continued*

17:05-17:25 **Structure and Dynamics of Microtubule- and Membrane-Bound Tau Protein from Solid-State NMR**

Presenting Author: Nadia El Mammeri

Nadia El Mammeri (MIT); Aurelio J. Dregni (MIT); Pu Duan (MIT); Olivia Gampp (MIT); Mei Hong (MIT)

The intrinsically disordered protein tau associates with and stabilizes microtubules to maintain neuronal health. Tau is also thought to interact with lipid membranes, which may play a role in the propagation of neurofibrillary tangles in Alzheimer's disease (AD). To understand the mechanism of the early misfolding events in AD, we have used magic-angle-spinning ssNMR to investigate the structure and dynamics of tau bound to microtubules and lipid bilayers. We present a new paradigm of tau's microtubule binding properties, and we reveal interesting mechanistic insights from membrane-induced tau amyloid fibrils. These results have important implications for the functional and pathological states of tau in diseases and demonstrate the power of solid-state NMR to investigate complex biomolecular assemblies in neurodegenerative diseases.

17:25-17:45 **High-Resolution 17O Solid-State Nuclear Magnetic Resonance of Peptides: 1H Detected HCO and HNO MQMAS**

Presenting Author: Zhehong Gan

Ivan Hung (Florida State University); Eric G. Keeler (New York Structural Biology Center); Wenping Mao (NHMFL, Florida State University); Peter Gorkov (NHMFL, Florida State University); Robert Griffin (Massachusetts Institute of Technology); Zhehong Gan (NHMFL, Florida State University)

Oxygen is an integral component of proteins but remains sparsely studied because its only NMR active isotope, 17O, has low sensitivity, low resolution, and large quadrupolar couplings. These issues are addressed here with efficient isotopic labeling, high magnetic fields, fast sample spinning, and 1H detection in conjunction with multidimensional experiments to observe oxygen sites specific to each amino acid residue. Sequential assignments and long-range distance restraints are demonstrated by using 3D 1H/13C/17O and 1H/15N/17O experiments, suggesting that such methods can become an essential tool for biomolecular structure determination. The use of 17O for initial polarization is found to provide better sensitivity per unit time compared to 1H.

MOI: Inorganic and Hybrid Materials (parallel session)
16:00-17:50, Nautilus

16:00-16:25 **Octahedral Tilt Engineering: Atomic-Level Picture of Stabilized α-FAPbI3**

Presenting Author: Dominik J. Kubicki

Tiarnan A. S. Doherty (Cavendish Laboratory, University of Cambridge,); Satyawan Nagane (Cavendish Laboratory, University of Cambridge,); Dominik Kubicki (University of Warwick); Young-Kwang Jung (Department of Materials Science and Engineering, Yonsei University); Duncan N. Johnstone (Department of Materials Science and Metallurgy, University of Cambridge); Affan N. Iqbal (Cavendish Laboratory, University of Cambridge); Dengyang Guo (Cavendish Laboratory, University of Cambridge); Kyle Frohna (Cavendish Laboratory, University of Cambridge); Mohsen Danaie (Electron Physical Science Imaging Centre, Diamond Light Source Ltd.); Elizabeth Tennyson (Cavendish Laboratory, University of Cambridge); Stuart Macpherson (Cavendish Laboratory, University of Cambridge); Anna Abfalterer (Cavendish Laboratory, University of Cambridge); Miguel Anaya (Cavendish Laboratory, University of Cambridge); Yu-Hsien Chiang (Cavendish Laboratory, University of Cambridge); Philip Crout (Department of Materials Science and Metallurgy, University of Cambridge); Simone Ruggeri (Laboratories of Organic and Physical Chemistry, Wageningen University and Research); Sean M. Collins (School of Chemical and Process Engineering and School of Chemistry, University of Leeds); Clare P. Grey (Yusuf Hamied Department of Chemistry, University of Cambridge); Aron Walsh (Department of Materials, Imperial College London); Paul A. Midgley (Department of Materials Science and Metallurgy, University of Cambridge); Samuel D. Stranks (Cavendish Laboratory, University of Cambridge)

Metal halide perovskites are used in optoelectronics research across the board and determining their atomic-level structure has been a growing area of solid-state NMR research over the past 5 years. I will give an overview of the recent progress, challenges and future directions in this area. I will focus particularly on the ternary formamidinium lead iodide as this material has excellent optical characteristics but is thermodynamically unstable and much of the current effort focuses on stabilizing its photoactive phase. We have developed a new stabilization strategy which relies on surface templating and results in remarkable phase stability. Solid-state NMR, NQR and scanning electron diffraction were key to understanding the atomic-level mechanism of this approach.

MONDAY, APRIL 17, 2023 - *continued*

16:25-16:45 **Migrating Solvation Structures in Li-ion Battery Electrolytes Revealed by Electrophoretic NMR**

Presenting Author: David Halat

David Halat (UC Berkeley & LBNL); Julia Im (University of California, Berkeley); Chao Fang (University of California, Berkeley); Aashutosh Mistry (Argonne National Laboratory); Saheli Chakraborty (University of California, Berkeley); Darby Hickson (University of California, Berkeley); Venkat Srinivasan (Argonne National Laboratory); Rui Wang (University of California, Berkeley); Nitash Balsara (University of California, Berkeley); Jeffrey Reimer (University of California, Berkeley)

The rapid (dis)charge capability of Li-ion batteries, an important metric for electric vehicle adoption, strongly depends on relative transport of ions and solvent within the electrolytic phase. Electrophoretic NMR (eNMR), wherein PFG experiments are synchronized with an applied electric field, can directly quantify the direction and magnitude of cation (⁷Li), anion (¹⁹F), and solvent (¹H) motion with spectroscopic specificity in Li-ion battery electrolytes. We report ¹H eNMR measurements of solvent velocity in model LiTFSI/tetraglyme electrolytes that reveal cation–solvent coordination, complementing molecular dynamics (MD) snapshots depicting specific cationic solvation structures. We also extend eNMR measurements to multivalent systems, i.e., polyanionic electrolytes, where we measure negative cation velocities that suggest Li⁺ moves the "wrong way" under electric field application.

16:45-17:05 **Probing the Local Environments of Cu(I) in Metal-Organic Frameworks via 63/65Cu Solid-state NMR Spectroscopy**

Presenting Author: Wanli Zhang

WANLI ZHANG (Western University); Bryan E.G. Lucier (Western University); Victor V. Terskikh (National Research Council Canada); Yining Huang (Western University)

For the first time, a series of Cu(I)-containing metal-organic framework (MOFs) with Cu sites in different coordination environments have been examined by using 63/65Cu wide-line NMR at 21.1 T. The 63/65Cu wide-line spectra were successfully acquired by Hahn-echo/WURST-CPMG sequences combined with VOCS (variable-offset cumulative spectra) method. The diversity of local environments of Cu(I) centers leads to a wide range of the quadrupolar coupling constant, ranging from 18.8 to 74.8 MHz depending on the Cu(I) local geometry. The NMR parameters were calculated by using plane-wave DFT calculations to aid the structural refinements of MOF systems. We demonstrate that the combination of 63/65Cu solid-state NMR and theoretical calculations can provide valuable structure information on local environment of copper metal ions/clusters within MOFs.

17:05-17:25 **Decoding the Microscopic Structure and Dynamics in Honeycomb-layered Sodium-ion Conductors: Na2MgxZn2-xTeO6 (x = 0 – 2) Using Solid-state NMR Spectroscopy**

Presenting Author: Diganta Sarkar

Diganta Sarkar (University of Alberta); Amit Bhattacharya (University of Alberta); Vladimir K. Michaelis (University of Alberta)

Sodium-ion batteries are emerging as a prospective alternative to lithium-ion batteries by offering low cost, improved safety, and superior sustainability. Solid-state NMR spectroscopy can provide a robust understanding of atomic-level structure and local Na-ion conduction to achieve their enhanced bulk performance. Here, we discuss a solid-state NMR method for comprehensive structure-property characterization of the honeycomb-layered solid electrolytes, Na2MgxZn2-xTeO6 ($0 \leq x \leq 2$), complemented by powder X-ray diffraction. The progression of Zn/Mg mixing in honeycomb layers and the influence of Zn/Mg order/disorder on bulk activation energies will be featured in this presentation. Furthermore, we study the variable-temperature 23Na spin-lattice relaxation to probe local Na-ion migration energies, and to determine how certain compositions may influence local Na-ion dynamics.

MONDAY, APRIL 17, 2023 - *continued*

17:25-17:50 **Combination of 17O NMR and computational modelling for the characterization of structure and dynamics in various systems containing COO groups**

Presenting Author: Christel Gervais

Christel GERVAIS (LCMCP - Sorbonne Universite); christian bonhomme (Sorbonne University); Hung Ivan (National High Magnetic Laboratory); Zhehong Gan (National High Magnetic Laboratory); Vinicius Martins (The University of Western Ontario); Bryan Lucier (The University of Western Ontario); Yining Huang (The University of Western Ontario); Jun Xu (National Institute for Advanced Materials, Nankai University); Thomas-Xavier Metro (IBMM, Universite de Montpellier); Cesar Leroy (ICGM, Universite de Montpellier); Ieva Goldberga (ICGM, Universite de Montpellier); Jessica packova (ICGM,Universite de Montpellier); Chia-Hsin Chen (ICGM, Universite de Montpellier); Danielle Laurencin (CNRS)

17O NMR spectroscopy is a very interesting characterization technique since oxygen can exhibit a wide variety of bonding in many molecules and materials. Thanks to selective 17O-enrichment, high-resolution 17O ssNMR spectra can be recorded and interpreted thanks to the use of DFT calculations : this combined experimental/theoretical approach allows a precise positioning of hydrogens and the nature of the H-bonding network to be established as well as binding modes of ligands. This will be illustrated in various systems including Zn and Al-based MOFs. In addition to the validation of structural models, 17O NMR data can also help to probe local dynamics as observed for instance in ibuprofen. Motions related to carboxylic groups were investigated with the help of computationnal modeling.

ASILOMAR LODGER DINNER for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees may purchase a lunch ticket bundle or dinner ticket online by April 7.
18:00 – 19:00, Crocker Dinning Hall

Vendor Hospitality Suites
From 19:00 onward

AMMRL Meeting
19:00-20:30, Chapel

TUESDAY, APRIL 18, 2023

Early Morning Lecture Series (for Students & ALL who wish to learn!)
07:00-07:50, Merrill Hall

07:00-07:50 **Understanding NMR Spectroscopy** Part 2 of 4

Presenter: James Keeler

Would you like to deepen or brush up your understanding of NMR theory? Join us for a morning lecture series by **James Keeler** (University of Cambridge), author of *Understanding NMR Spectroscopy*. These lectures assume only a modest prior knowledge of NMR theory and will cover some key topics of wide interest to budding NMR spectroscopists. Topics will include: Energy levels, Hamiltonians and operators; Introducing and using product operators; Relaxation; Coherence selection by phase cycling and field gradients

ASILOMAR LODGER BREAKFAST for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees cannot purchase breakfast tickets, but you can purchase lunch and dinner tickets by April 7.
07:30-09:00, Crocker Dining Hall

TOA: BioSolids II (parallel session)
8:45 - 10:10, Merrill Hall

08:45-09:10 **The Mechanisms of Lipid-targeting Antibiotics**

Presenting Author: Markus Weingarth

Markus Weingarth (Utrecht University)

Antimicrobial resistance is a global health threat, calling for new antibiotics. Good candidates could be compounds that target special lipids that only exist in bacterial, but not in human cell membranes. These drugs kill pathogens without detectable resistance. This has generated huge interest.

Using ssNMR and microscopy, our group has introduced approaches to study lipid-targeting antibiotics at different length-scales in membranes(1,2). Recently, we determined the killing mechanism of teixobactin(3), considered the first new antibiotic in 30 years. We showed that teixobactin kills bacteria by forming supramolecular fibrils that compromises the bacterial membrane. In addition, we show the mechanism of Clovibactin, a new antibiotic from 'unculturable' bacteria.

1. Medeiros-Silva, Nature Communications (2018)

3. Shukla, Nature Communications (2020)

2. Shukla, Nature (2022)

09:10-09:30 **NMR Crystallography of Tyrosine Phenol Lyase: Refining the Crystal Structure and Highlighting the Active Site Chemistry**

Presenting Author: Rittik K Ghosh

Rittik Ghosh (Department of Biochemistry, University of California - Riverside, CA 92521); Maria Luiza Caldas Nogueira (The University of Florida); Frederic M. Vigier (National High Magnetic Field Laboratory, Florida State University, Tallahassee, FL 32310); Joanna Long (University of Florida); Len Mueller (University of California Riverside)

We make use of an integrative combination of DNP enhanced solid-state NMR, X-ray crystallography, and first principles computational chemistry to solve for the atomic-resolution, three-dimensional structure of the active site for the quinonoid intermediate of tyrosine phenol lyase, a 206 kDa pyriodoxal-5'-phosphate dependent enzyme. Most importantly, this NMR crystallographic approach allows us to delineate the mechanistically significant protonation states that define the active site chemistry and reconcile inconsistencies between previous X-ray crystal structures. Our refined active site structure satisfies both the experimental chemical shift restraints and the active site electron density for the bound substrate, which was previously poorly fit by the reported structural model.

TUESDAY, APRIL 18, 2023 - *continued*

09:30-09:50 **Solid-state NMR analysis of the Pseudomonas aeruginosa biofilm matrix**

Presenting Author: Courtney Reichhardt

Courtney Reichhardt (Washington University)

Most bacteria live as multi-cellular communities termed biofilms, which protects bacteria against harsh conditions. Within biofilms, bacterial cells are entangled in a biopolymer-rich extracellular matrix that is rich in biopolymers. Previously, it was determined that up to three structurally distinct exopolysaccharides as well as several proteins and extracellular DNA contribute to Pseudomonas aeruginosa biofilm matrices. The presence of different exopolysaccharides in P. aeruginosa biofilm matrices results in varying viscoelastic properties of the biofilms, which is related to the virulence of the bacteria. We hypothesized that these viscoelastic properties arise due to different dynamics and interactions of exopolysaccharides within the biofilm matrix. To test this hypothesis, we used solid-state NMR to investigate the composition and dynamics of P. aeruginosa biofilm matrices.

09:50-10:10 **Magic-Angle Spinning NMR and Integrated Approaches for Structure Determination of Microtubule-Associated Proteins Assembled with Microtubules**

Presenting Author: Changmiao Guo

Changmiao Guo (Department of Chemistry and Biochemistry, University of Delaware); Chunting Zhang (Department of Chemistry and Biochemistry, University of Delaware); Raymundo Alfaro-Aco (Department of Molecular Biology, Princeton University); Ryan W. Russell (Department of Chemistry and Biochemistry, University of Delaware); Caitlin M. Quinn (Department of Chemistry and Biochemistry, University of Delaware); Mingyue Li (Department of Chemistry and Biochemistry, University of Delaware); Angela M. Gronenborn (Department of Structural Biology, University of Pittsburgh School of Medicine); Sabine Petry (Department of Molecular Biology, Princeton University); John C. Williams (Department of Molecular Medicine, Beckman Research Institute of City of Hope); Tatyana Polenova (Department of Chemistry and Biochemistry, University of Delaware)

We demonstrate magic-angle spinning (MAS) NMR and integrated approaches for structure elucidation of microtubule-associated proteins assembled with microtubules, including kinesin-1 motor domain (KIF5B) and active domain of a microtubule nucleation factor. We determined an all-atom NMR structure of KIF5B in complex with microtubules by integrating MAS NMR restraints with the medium-resolution cryo-EM density map. Additionally, we report the structure of the targeting protein for Xklp2 C-terminal domain involved in a condensate on microtubules using 1H-detected fast MAS NMR and molecular modeling. These studies provided atomically detailed insights unavailable from other methods, such as binding interfaces with microtubules, and "invisible" dynamically disordered regions of biological assemblies.

TOB: Instrumentation I (parallel session)
8:45 - 10:15, Chapel

08:45-09:10 **Silicon-chip based small NMR spectrometers**

Presenting Author: Donhee Ham

Donhee Harvard (Harvard University)

Over the past ca. 15 years, we have been developing miniaturized nuclear magnetic resonance (NMR) spectrometers by conflating small permanent magnets and silicon radio-frequency transceiver integrated circuits. They have been able to perform a diverse set of multi-dimensional NMR relaxometry and spectroscopy experiments, resolving J-coupled spectra for small molecules, and magnetic resonance imaging (MRI). These portable, affordable, and low-maintenance NMR spectrometers may enable in-field, on-demand, or online applications for small molecular fingerprinting, chemical reaction monitoring, biomolecular sensing, quality control, subsurface exploration, and imaging of ex vivo biological tissues as well as artificial organoids, small organisms, and organic material systems. In this presentation, I will review these small NMR platforms and their future perspectives.

09:10-09:30 **190 GHz Single Chip Dynamic Nuclear Polarization Microsystem**

Presenting Author: Nergiz Sahin Solmaz

Nergiz Sahin Solmaz (Ecole Polytechnique Federale de Lausanne); Reza Farsi (Ecole Polytechnique Federale de Lausanne); Giovanni Boero (Ecole Polytechnique Federale de Lausanne)

We report on a single chip DNP microsystem operating at 190 GHz. The ESR detector consists of a 190 GHz oscillator with oscillation amplitude detection. The frequency of the oscillator is tunable up to 5 GHz. The NMR detector is a receiver-only chain with 75 dB overall gain, consisting of a 10 turns microcoil with a diameter of about 200 um, a broadband LNA operating up to 1 GHz, a mixer, and a LF amplifier with 4 MHz bandwidth. An external coil is used for NMR excitation. At the conference, ESR, 1H NMR, and 1H DNP-NMR spectra acquired with solid samples at about 6.8 T at room temperature as well as at low temperature will be presented.

TUESDAY, APRIL 18, 2023 - *continued*

09:30-09:50 **A flexible low cost microchip-based NMR system for micro-scale applications**

Presenting Author: Kathryn Marable

Kathryn Marable (Annaida Technologies); Marco Grisi (Annaida Technologies SA); Guillaume Gruet (Annaida Technologies); Giulia Sivelli (Annaida Technologies); Gaurasundar Conley (Annaida Technologies)

Micro-NMR is a growing area within the field of NMR that allows increased sensitivity with reduced sample volume demands [1]. We present here a scalable micro-NMR, user-friendly, and broadband probe system. The probe can be used with standard NMR spectrometers in measurements of samples with volumes from 10 nL to 100 pL at frequencies between 150 MHz and 600 MHz. The use of additive manufacturing allows customization and adaptation for a wide range of magnets, spectrometer systems, and applications. Furthermore, the CMOS-based design allows for simultaneous acquisition from multiple sensor coils on the same device. This presentation discusses the system architecture, features, and performance, as well as prospects for future developments.

09:50-10:15 **Zero-dead-time detection and other advances in oscillator-based NMR and EPR**

Presenting Author: Jens Anders

Jens Anders (University of Stuttgart); Michal Kern (University of Stuttgart); Bernhard Bluemich (RWTH Aachen); Klaus-Peter Dinse (Helmholtz-Zentrum Berlin fuer Materialien und Energie); Klaus Lips (Helmholtz-Zentrum Berlin fuer Materialien und Energie)

In this invited talk, after a brief introduction to the working principle of VCO-based MR, including a detailed explanation of its ability for zero-deadtime-detection, we will present our latest research results in this field together with a discussion of the advantages and drawbacks of this method compared to classical MR detection. We will close the talk with a brief outlook on future research directions in VCO-based MR.

COFFEE BREAK with Exhibit Booths and Posters, Fireside Pavilion
10:20-10:45

TOD: Impacts of Metabolomics (parallel session)
10:45-12:30, Merrill Hall

10:45-11:10 **NMR and the Brazilian flora: a successful combination in the identification of Leishmania donovani nucleoside hydrolase inhibitors**

Presenting Author: Luzineide Wanderley Tinoco

Bruno Clemente B. Marques (Universidade Federal do Rio de Janeiro); Gregorio Torres Rangel (Universidade Federal do Rio de Janeiro); Guilherme S. Caleffi (Universidade Federal do Rio de Janeiro); Joao Avelar (Universidade Federal do Rio de Janeiro); Paulo Roberto Ribeiro Costa (Universidade Federal do Rio de Janeiro); Luzineide Tinoco (Universidade Federal do Rio de Janeiro)

Nucleoside hydrolases are a strategic target for drug development to treat leishmaniasis, a neglected disease. The identification of flavonoids as inhibitors of Leishmania donovani nucleoside hydrolase (LdNH) emerged from the biological screening of 214 extracts from Brazilian plants. Three plants were selected for their results and lack of previous phytochemical description: Leandra amplexicaulis, Urvillea rufescens, and Ormosia arborea. The use of NMR combined with chemometrics allowed the identification of two new proanthocyanidins as LdNH inhibitors before isolation, directing the best strategy to purify them. From this identification, a series of flavonoids was synthesized and their activities determined. STD and Waterlogsy data, obtained for the most potent flavonoids, combined with docking studies, provided information on structure-activity relationships.

11:10-11:30 **A New Limit for Blood Metabolite Analysis Using 1H NMR Spectroscopy**

Presenting Author: G. A. Nagana Gowda

G. A. Nagana Gowda (University of Washington); Vadim Pascua (University of Washington); Daniel Raftery (University of Washington)

The relatively small number of blood metabolites accessible by a simple 1D NMR method has restricted the scope of NMR applications in the metabolomics field. Enhancing the limit of identified metabolites in blood will therefore greatly impact NMR-based metabolomics. With a focus on addressing this challenge, and based on the comprehensive investigation of human blood and plasma using a combination of 1D/2D NMR techniques, we describe the identification of unknown metabolites and expanding the limits of quantifiable blood metabolites using NMR. The results provide access to nearly 90 metabolites, which is the highest to date for a simple 1D 1H NMR experiment that is widely used in the metabolomics field. The new findings are expected to greatly impact blood metabolomics.

TUESDAY, APRIL 18, 2023 - *continued*

11:30-11:50 **Mitochondrial Metabolism in Astrocytes is Essential for their Survival Against Neurotoxic Electrophile Exposure**

Presenting Author: Alexandra Crook

Alexandra Crook (University of Nebraska - Lincoln); Jordan Rose (University of Nebraska - Lincoln); Annadurai Anandhan (University of Nebraska - Lincoln); Christian Brian (University of Nebraska - Lincoln); Rodrigo Franco Cruz (University of Nebraska - Lincoln); Robert Powers (University of Nebraska - Lincoln)

Astrocytes regulate neuronal excitability and homeostasis, and they are the first line of defense against xenobiotics crossing into the brain. We demonstrate that astrocytes mitochondrial metabolism is essential for survival against neurotoxic electrophiles such as inorganic arsenic (iAs). Subtoxic iAs induced an increase in de novo GSH synthesis and a reduced intracellular environment in astrocytes. Targeted NMR metabolomics revealed that iAs induced the anaplerotic generation of glutamate via the TCA cycle, which contributes to GSH de novo synthesis. iAs exposure led to substantial extracellular glutamate accumulation that was mediated by reversal of the excitatory amino acid transporter 1. These results reveal that mitochondrial metabolism in astrocytes is required for the detoxification of neurotoxic electrophiles such as iAs.

11:50-12:10 **Pluronic F-127 as a gel matrix for in-cell NMR**

Presenting Author: Cale Thornton

Cale Thornton (Boise State University); Nicole Elizabeth Aughtry (Boise State University); Wesley Joseph Hirons (Boise State University); Lisa R. Warner (Boise State University)

In-cell NMR is a technique that can be used to analyze metabolic pathways in a wide variety of cells in vivo. However, cells larger than ~2 micrometers in diameter, tend to settle to the bottom of the NMR tube over the course of hours, no longer centered in the coil. This limits application of in-cell NMR to smaller cells, shorter experiment time, or specialty tubes. One approach to mitigate this problem is to suspend cells in a gelatinous medium. Pluronic F-127 is a biocompatible thermosensitive hydrogel that has potential as a gel matrix for in-cell NMR and has previously been used to study magnetically aligned Pf1 phage. Here, we examine Pluronic F-127 as a gel matrix for in-cell NMR experiments.

12:10-12:30 **Detection of Human Prostate Cancer using Multivoxel MR Spectroscopy Based Metabolomics Imaging**

Presenting Author: Leo Cheng

Leo Cheng (MGH Harvard Medical School)

Imaging detection of prostate cancer is still a challenging task in clinic. NMR-based metabolomics has demonstrated improved potential in disease diagnosis and characterization when compared with measurements of individual metabolites. Using a 7T human whole-body MRI system, we measured multivoxel MRS for 30 prostates removed from patients with biopsy-proven cancer. These 30 cases were divided into Training and Testing cohorts. Cancer discriminating canonical analysis was conducted on the Training cohort and applied to the Testing cohort. Metabolomics imaging profiles thus discovered demonstrate the superior capability when compared with parameters currently used in clinic. Although obtained from removed prostates, these results, measured on a whole-body MRI system, can be implemented directly in clinic, with its concept applicable to general clinical MR.

TOE: Simulations, Processing, and New Software (parallel session)
10:45-12:30, Chapel

10:45-11:10 **EasyNMR: Web-based data handling and NMR simulations**

Presenting Author: Thomas Vosegaard

Armin Afrough (Interdisciplinary Nanoscience Center, Aarhus University); Thomas Vosegaard (Interdisciplinary Nanoscience Center, Aarhus University)

We present a flow-based programming solution, EasyNMR, for numerical simulations in NMR and with a novel focus on cloud-based data storage and -sharing. With a web-based interface, EasyNMR needs no installation thus limiting the barrier for getting started yet maintains the versatility of more advanced NMR simulation software solutions.

TUESDAY, APRIL 18, 2023 - *continued*

11:10-11:30 **Spectroscopy as an inverse problem: Structure elucidation from 1D NMR spectral information via deep imitation learning**

Presenting Author: Eric Jonas

Eric Jonas (University of Chicago)

Small molecule structure elucidation has long been an area of active spectroscopic interest. Here we formulate structure elucidation as an inverse problem, akin to tomography or medical imaging, and develop new machine-learning techniques to elucidate small molecule structures entirely from shift data. We do this by leveraging an existing fast forward model (from structure to spectrum) and new graph-based machine learning methods. We can achieve 80% accuracy on structure recovery with carbon shifts and 40% accuracy with proton shifts. Additionally our method can produce quantified confidence estimates, saying when it "doesn't know" the correct solution for a given spectrum. We highlight future directions for this approach and quantify the overall identifiability of small molecule structures from shift spectra.

11:30-11:50 **Automated Large Scale 13-C & 1-H Chemical Shift Predictions of 40,000 Natural Products via DFT Calculations**

Presenting Author: Amy Jystad

Amy Jystad (Pacific Northwest National Laboratory); Jessica Bade (Pacific Northwest National Laboratory); Sean Colby (Pacific Northwest National Laboratory); John Cort (Pacific Northwest National Laboratory)

The gold standard for identification of novel natural products is structure elucidation by NMR spectroscopy. Unfortunately, most of this data is scattered in labs and print journals. This has spurred efforts to establish the Natural Products Magnetic Resonance Database (NP-MRD). NP-MRD is expected to contain ~350,000 structures, only a small portion (<15%) will likely have associated NMR data. To fill this gap, we are in the process of backfilling NP-MRD with DFT calculated NMR spectra. We have developed in silico Chemical Library Engine 2 (ISiCLE 2) to automate the prediction of chemical shifts, and Simulated NMR to Experiment Assignment Software LibrarY (SNEASY) to compare predicted and experimental shifts, resulting in the DFT chemical shift data of ~40,000 natural products.

11:50-12:10 **Rapid Prediction of Full Spin Systems using Uncertainty-Aware Machine Learning**

Presenting Author: Jake Williams

Jake Williams (University of Chicago); Eric Jonas (University of Chicago)

Accurate simulation of solution NMR spectra requires knowledge of all chemical shift and scalar coupling parameters. We present a novel machine learning technique which combines uncertainty-aware deep learning with rapid estimates of conformational geometries to generate Full Spin System Predictions with UnCertainty (FullSSPrUCe). We improve on previous state of the art in accuracy on chemical shift values, predicting protons to within 0.209 ppm and carbons to within 1.218 ppm. Further, we are able to predict all scalar coupling values, unlike previous GNN models, achieving 3JHH accuracies between 0.838-1.392 Hz on small experimental datasets. Uncertainty quantification shows a strong, useful correlation with accuracy, and we design a method to intelligently combine ab initio and experimental data.

12:10-12:30 **mrsimulator: A cross-platform object-oriented open-source software package for fast solid-state NMR spectral simulation and analysis**

Presenting Author: Philip Grandinetti

Deepansh Srivastava (Hyperfine, Inc); Matthew Giammar (Ohio State University); Lexi McCarthy (Ohio State University); Maxwell Venetos (University of California); Philip Grandinetti (Ohio State University)

The free and open-source Python package mrsimulator is a simple-to-use, easy-to-install, versatile library with a permissive license capable of simulating multi-dimensional solid-state NMR spectra of coupled spin systems under variable-angle spinning conditions. High simulation benchmarks are achieved using analytical solutions for transition frequencies and coherence transfers between transitions. This approach generalizes to multi-dimensional NMR spectra simulations using symmetry pathway concepts for describing multi-pulse NMR experiments. The efficiency gains with this approach are essential for the accurate spectral modeling of non-crystalline materials, where thousands of subspectra are needed for accurate line shape simulations. mrsimulator is fully documented with numerous examples (https://mrsimulator.readthedocs.io). It easily integrates with other scientific and machine-learning libraries to create new opportunities for data science with solid-state NMR spectroscopy.

TUESDAY, APRIL 18, 2023 - *continued*

ASILOMAR LODGER LUNCH for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees may purchase a lunch ticket bundle or dinner ticket online by April 7.
12:45-14:00, Crocker Dinning Hall

OR

UHF-NMR Lunch Meeting, RSVP by March 31
13:00-14:00

POSTER SESSION with Exhibit Booths
14:00-15:45, Fireside Pavilion
See Poster Listings at end of document.

TOG: Organic, Soft Matter and Biomaterials (parallel session)
16:00-17:45, Merrill Hall

16:00-16:25 **Developing models of extracellular matrix in health and ageing: NMR approaches**

Presenting Author: Melinda J Duer

Melinda Duer (University of Cambridge)

The extracellular matrix (ECM) forms the bulk of our structural tissues and provides them with their mechanical properties. Intriguingly, at the molecular level, the ECM provides the communication system between the cells and the signals that drive the individual behaviour of cells. Ultimately, if we can understand how the extracellular matrix molecular structure and dynamics dictates the behaviour of cells, then we can develop ways to combat the effects of ageing. However, understanding the molecular level properties of the extracellular matrix has been hampered by the lack of methods to study tissues at the atomic scale. In this talk, I will describe the NMR approaches that my group has taken and are continuing to develop to tackle these complex questions.

16:25-16:45 **Characterization of bound water in post-translationally modified amyloid-beta fibrils and a soil sample from Antarctica, using O-17 and H-2 NMR.**

Presenting Author: Liliya Vugmeyster

Liliya Vugmeyster (CU Denver); Dmitry Ostrovsky (CU Denver); Aryana Rodgers (CU Denver); Riqiang Fu (National High Magnetic Field Laboratory)

Water bound to interfaces of biological molecules or soils can have profound impact on functionality due to enhancement of motions and activation of interfaces. Using a combined approach with deuterium and oxygen-17 solid-state NMR, we present characterization of amounts and motions in bound water in two very distinct systems: amyloid-beta fibrils with a pyroglutamate post-translational modification and a soil sample from McMurdo Dry Valleys of Antarctica. Spectra, longitudinal and rotating frame relaxation times at two magnetic field strength values (18.8 and 9.4 T) and over a broad temperature range of 300 to 220 K provide means of obtaining amounts of free and bound water, as well as time scales of motions induced by interfacial interactions.

16:45-17:05 **Paramagnetic Guest Exchange Saturation Transfer (ParaGEST) Revealing Hidden Interactions in Supramolecular Host-Guest Systems**

Presenting Author: Elad Goren

Elad Goren (Mr.); Liat Avram (Dr.); Amnon Bar-Shir (Prof.)

The recently developed ¹⁹F-guest exchange saturation transfer (¹⁹F-GEST) approach adopts the principles of chemical exchange saturation transfer (CEST) for studying the binding kinetics of host-guest systems. Incorporating paramagnetic lanthanides to α- and β-cyclodextrins (creating Ln-α-CDs and Ln-β-CDs), to obtain ¹⁹F-paraGEST, allows to study faster exchange rates in NMR time scale.

Here we show that varying the pseudo-contact shift induced on an exchanging fluorinated guest provides insights into its dynamic interactions, not accessible by any other analytical tool. Specifically, benefiting from the enhanced spectral resolution of ¹⁹F-paraGEST, we could identify two different populations of a bound guest specific for Ln-β-CDs, implying different CD-binding geometries with similar activation energies. The results highlight the importance of ¹⁹F-paraGEST for studying "NMR-invisible" host-guest systems.

TUESDAY, APRIL 18, 2023 - *continued*

17:05-17:25 **Exploring Ion gating of conducting polymer PEDOT:PSS by Operando NMR Spectroscopy**

Presenting Author: Dongxun Lyu

Dongxun Lyu (University of Cambridge); Yanting Jin (University of Cambridge); Pieter Magusin (University of Cambridge); Evan Wenbo Zhao (University of Cambridge); Scott Keene (University of Cambridge); George Milliaras (University of Cambridge); Clare Grey (University of Cambridge)

The conducting polymer poly(3,4-ethylenedioxythiophene) poly(styrene sulfonate) (PEDOT:PSS) is regarded as the most promising organic mixed ionic-electronic conductors for applications in bioelectronics and energy storage. This has led to increasing interest in the development of new analytical methods to non-invasively visualise the transport and coupling of electronic and ionic charge carriers during device operation. Here we show that operando 1H and 23Na NMR spectroscopy can quantify cation and water movement during the doping/dedoping of PEDOT:PSS films. A distinct quadrupolar splitting is observed for sodium ions bound to anisotropic domains of the polymer films. Operando 23Na NMR studies reveal a close-to-linear correlation between the quadrupolar splitting and the charge stored in the film, which is quantitatively explained by a two-site exchange model.

17:25-17:45 **Decoupling the Effects of Thermal Cycling and Shear on Phase-Change Nano-Emulsions by NMR spectroscopy, Rheo-NMR, and MRI Velocimetry**

Presenting Author: Jungeun Park

Jungeun Park (The City College of New York); Ulrich Scheler (Leibniz Institute for Polymer Research); Robert J. Messinger (The City College of New York)

Organic phase-change material (PCM) nano-emulsions are formed by emulsifying oil in water in the presence of surfactant and can store or release thermal energy during phase transitions. However, PCMs nano-emulsions become unstable due to thermal cycling and shear in heat transfer systems. To better understand the molecular origins of these instabilities, liquid-state NMR measurements were applied to a model PCM nano-emulsion, enabling metastable supercooling effects to be monitored and revealing that the surfactant head group exists in multiple environments that change upon thermal cycling. 1H rheo-NMR and MRI velocimetry methods were also applied to measure the velocity profile and concentration distribution of oil within a Searle cell, revealing non-linear velocity profiles and shear-induced migration of the emulsion droplets.

TOH: BioSolution: Molecular Interactions and Energetics (parallel session)
16:00-17:45, Chapel

16:00-16:25 **The role of highly flexible regions in orchestrating the properties of multidomain proteins: insights from 13C detected NMR experiments**

Presenting Author: Isabella C Felli

Isabella Felli (University of Florence)

Highly flexible regions of complex multi-domain proteins introduce an additional dimension in protein function, still exploiting simple modules (globular domains and highly flexible regions themselves). This modular protein architecture is shared by many proteins involved in recognition, signaling and regulation, all processes in which structural and dynamic heterogeneity plays a fundamental role. Protein malfunction, linked to the onset of incurable diseases, is often related to highly flexible regions.

NMR represents a unique tool for their investigation at the atomic level. However when globular and disordered domains are simultaneously present in a protein, the NMR spectra can become quite complex. 13C detection offers an elegant approach to study them not only in isolation but also when part of complex multi-domain proteins.

16:25-16:45 **Quadruplex DNA Structures and Ligand Intercalation**

Presenting Author: Janez Plavec

Janez Plavec (National Institute of Chemistry)

DNA with its canonical duplex and alternative structures including quadruplex motifs are associated with many biological functions of DNA. NMR revealed structural details of four-stranded DNA architectures adopted by GGGAGCG repeats in the regulatory regions of genes responsible for neurological disorders. Their unique tetrahelical structures are distinctly different from G-quadruplexes. Complexes with bis-quinolinium ligand 360A exhibit intercalation between GAGA- and GCGC-quartets. On the other hand, Phen-DC3, one of the best-known G-quadruplex ligands causes dTAGGG(TTAGGG)3 to change its fold in KCl solution from a hybrid-1 to a chair-type structure, with the ligand intercalating between two G-quartets, ejecting a potassium ion. The unprecedented high-resolution NMR structure is the first to show true ligand intercalation into an intramolecular G-quadruplex.

TUESDAY, APRIL 18, 2023 - *continued*

16:45-17:05 **Pressure, Motion and Conformational Entropy in Molecular Recognition by Proteins**

Presenting Author: Josh Wand

Jose A. Caro (Texas A&M University); Kathleen G. Valentine (University of Pennsylvania); Taylor R. Cole (Texas A&M University); Josh Wand (Texas A&M University)

Molecular recognition is a determinant of biochemistry. A dynamic proxy using NMR-relaxation has revealed a richness in the contributions of conformational entropy (-TΔSconf) to binding thermodynamics. We examined the internal motion of barnase in a thermodynamic cycle over binding barstar and hydrostatic pressure. Motion in barnase, as evidenced by 15N and methyl deuterium relaxation, is conserved along the pressure-binding cycle. Binding has a -TΔSconf penalty of +11.7 kJ/mol at 1 bar. At 3kbar, the overall change in side chain motion (TΔSconf) is zero, suggesting a role for ΔSconf in the adaptation of proteins to extreme environments. Spatial clustering of the pressure sensitivity indicates that local contributions of ΔSconf can contribute to the thermodynamics of protein function.

17:05-17:25 **Investigation of the Allosteric Signaling Mechanism of a Thermostable GeoCas9 by Solution NMR**

Presenting Author: Helen Belato

Helen Belato (Brown University); George P. Lisi (Brown University)

CRISPR-Cas9 is a widely utilized biochemical tool. An understanding of the molecular motions that are critical for target DNA recognition, unwinding, and cleavage by Cas9 are unknown. In this study, we report an atomic level comparison of structural and dynamic properties of the Recognition lobe (Rec) and the HNH domain of a thermostable Cas9 (GeoCas9). We show that GeoHNH motions are regulated by fast (ps-ns) timescale dynamics. Furthermore, when the residue with the highest flexibility on the ps-ns timescale is mutated in GeoHNH, its protein solubility, thermal stability, and dynamic profile are drastically distorted. NMR studies of Rec show evidence of a contiguous millisecond timescale dynamic pathway throughout Rec, which links the adjacent GeoHNH domain for allosteric signaling.

17:25-17:45 **Modulation of co-translational protein folding on CRISPR/Cas9-engineered ribosomes investigated by solution NMR, cryoEM and MD simulations**

Presenting Author: Minkoo Ahn

Minkoo Ahn (University College London); Tomasz Wodarski (University College London); Alkistis Mitropoulou (University College London); Sammy Chan (University College London); Haneesh Sidhu (University College London); Elena Plessa (University College London); Thomas Becker (Ludwig-Maximilians-Universitt Mnchen); Nediljko Budisa (University of Manitoba); Christopher Waudby (University College London); Roland Beckmann (Ludwig-Maximilians-Universitt Mnchen); Anas Cassaignau (University College London); Lisa Cabrita (University College London); John Christodoulou (University College London)

To understand how co-translational protein folding is modulated by the narrow ribosome exit tunnel, we have rationally engineered three exit tunnel protein loops of the 70S ribosome by CRISPR/Cas9 gene editing, and studied the co-translational folding of an immunoglobulin-like filamin domain (FLN5). Our thermodynamics measurements employing 19F/15N/methyl-TROSY NMR spectroscopy reveal how the variations in the lengths of the loops (uL23, uL24) exert their distinct and concerted effects on the free energy of FLN5 folding and binding to the ribosome surface. This is highlighted by the opposite folding outcomes resulting from the loop extensions, and cryoEM and MD simulations show the structural basis of such changes, thereby providing principles for how to remodel them to elicit a desired folding outcome.

ASILOMAR LODGER DINNER for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees may purchase a lunch ticket bundle or dinner ticket online by April 7.
18:00 – 19:00, Crocker Dinning Hall

VENDOR HOSPITALITY SUITES
From 18:30

WEDNESDAY, APRIL 19, 2023

Early Morning Lecture Series (for Students & ALL who wish to learn!)
07:00-07:50, Merrill Hall

07:00-07:50 **Understanding NMR Spectroscopy** Part 3 of 4

Presenter: James Keeler

Would you like to deepen or brush up your understanding of NMR theory? Join us for a morning lecture series by **James Keeler** (University of Cambridge), author of *Understanding NMR Spectroscopy*. These lectures assume only a modest prior knowledge of NMR theory and will cover some key topics of wide interest to budding NMR spectroscopists. Topics will include: Energy levels, Hamiltonians and operators; Introducing and using product operators; Relaxation; Coherence selection by phase cycling and field gradients

ASILOMAR LODGER BREAKFAST for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees cannot purchase breakfast tickets, but you can purchase lunch and dinner tickets by April 7.
07:30-09:00, Crocker Dining Hall

WOA: Theoretical Treatment of Pulse Sequences and Experiments (parallel session)
8:45 - 10:10, Merrill Hall

08:45-09:10 **Pulse-Sequence Optimization Based on Effective Floquet Hamiltonians**

Presenting Author: Matthias Ernst

Matias Chavez (ETH Zurich); Matthias Ernst (ETH Zrich)

We present a new approach to pulse sequence optimization that is based on effective Floquet Hamiltonians and not state-to-state transfers using numerical simulations. Efforts to use effective Floquet Hamiltonians for pulse-sequence optimization have been hampered by the requirement to have a continuous transition from a resonant situation through a near-resonant and finally to a non-resonant effective Hamiltonian. Especially the near resonant case is difficult to describe using classical Floquet methods. We have introduced a frequency-continuous Floquet approach that allows us to obtain effective Hamiltonians in all three cases. Based on this approach, we will show first attempts to optimize pulse sequences for frequency-selective polarization transfer under MAS based on single-spin interaction-frame trajectories that can be calculated very efficiently.

09:10-09:30 **Low power optimal control pulses improve the performance of multidimensional Bio-molecular solution NMR experiments at ultrahigh-field 1.2 GHz (28.2 T) spectrometers**

Presenting Author: David Joseph

David Joseph (Max Planck Institute for Multidisciplinary Sciences, Gttingen, Germany); Christian Griesinger (Max Planck Institute for Multidisciplinary Sciences, Goettingen, Germany)

Bio-molecular NMR studies are usually limited by sensitivity and resolution, especially for concentration limited samples. At present, Bruker provides only a 3 mm CryoProbe for the 1.2 GHz spectrometers due to high power limitations. This restricts the study of concentration limited samples to a smaller sample volume. In this work, we have designed low power optimal control (OC) pulses for broadband universal rotation of 1H and 15N nuclei and introduce a new approach to designing OC band selective pulses. These pulses were utilized to construct low power 2D-OC-[1H,15N]-HSQC and 3D-OC-HNCO and their TROSY versions. These OC-sequences gave a performance improvement and we are confident that low power OC experiments can enable large volume measurements at the 1.2 GHz magnets.

09:30-09:50 **Towards more Robust Sparsity with Nonuniform Sampling in Challenging 2D NMR by Reducing Repeat Subsequences**

Presenting Author: Lucille E Cullen

Lucille Cullen (Bucknell University); Alan Marchiori (Bucknell University); David Rovnyak (Bucknell University)

Sampling noise and aliasing artefacts are a barrier to using sparser nonuniform sampling (NUS) in complex 2D NMR experiments. We find that weak aliasing artefacts are a growing concern in sparser 1D-NUS and can sometimes be misattributed to incomplete deconvolution of the broader point-spread function. As sparsity increases in NUS, we find that detrimental repeat sequences can occur early in the sampling schedule, correlating with aliasing artefacts in resulting spectra. By developing a convolutional screening approach to evaluate sampling schedules, these repeat sequences can be detected and characterized. Selecting schedules to avoid repeat sequences and using short periods of initial uniform sampling are effective at reducing these initial repeat sequences and enabling routine 25-33% 1D-NUS of challenging 2D-NMR experiments.

WEDNESDAY, APRIL 19, 2023 - *continued*

09:50-10:10 **SAND: Automated Time-Domain Modeling of NMR Spectra Applied to Metabolomics**

Presenting Author: Frank Delaglio

Yue Wu (Stanford University); Omid Sanati (University of Georgia); Mario Uchimiya (University of Georgia); Krish Krishnamurthy (Chempacker LLC); Art Edison (University of Georgia); Frank Delaglio (NIST IBBR)

NMR metabolomics offers great potential impact in drug discovery, diagnostics, and personalized medicine, but its exploitation requires detailed quantification of spectral features. Development of an automated, objective workflow for such quantification has been a long-standing challenge because of extensive spectral overlap and high signal complexity. To address this challenge, we introduce the software application SAND (Spectral Automated NMR Decomposition), for automated feature quantification in the time-domain. SAND follows upon the success of previous approaches to analyze spectra by time-domain modeling, adding a framework that provides automated quantification of entire spectra without the need for interactive region selection. We demonstrate SAND on metabolomics data including a urine spectral series spiked with differing amounts of a four-compound mixture.

WOB: BioSolution: Membrane Proteins (parallel session)
8:45 - 10:15, Chapel

08:45-09:10 **Structural and dynamic characterization of viral proteins: NS2B and domain III of glycoprotein E of Zika virus**

Presenting Author: Ana Paula Valente

Ana Paula Valente (Federal University of Rio de Janeiro)

We aim to explore fundamental issues associated with protein dynamics and macromolecular interactions in recognition processes using solution Nuclear Magnetic Resonance (NMR). We emphasize the study of NS2B and domain III of glycoprotein E of Zika virus. NS2B is a membrane protein responsible for regulating viral protease activity. We were able to elucidate its structure formed by four transmembrane helices and a hydrophilic portion that is compatible with the relaxation parameters and PRE data. The domain III of the glycoprotein E (DIII) is related to the binding of the virus to the cell receptor and neutralizing antibodies. DIII was studied free and in complex with glycosaminoglycan to mimic the interaction with the extracellular matrix.

09:10-09:30 **Bicelles Are an Effective Membrane Model for High-Resolution NMR Study of Curvature-Sensitive Molecules**

Presenting Author: Fang Tian

fang Tian (Penn State University College of Medicine); Yansheng Ye (Penn State University College of Medicine); Guifang Wang (Penn State University College of Medicine); Hong-Gang Wang (Penn State University College of Medicine)

Membrane curvature, a geometric measurement of the bending of the lipid bilayer, has been demonstrated to regulate enzymatic activity (e.g. ArfGAP1, Atg3, and Sar1) and direct the subcellular localization of some proteins (e.g. SpoVM and DivIVA). Our recent studies of human Atg3 (hAtg3) and VPS37A proteins demonstrated that, unlike micelles and nanodisc, bicelles are an effective membrane model for the structural study of curvature sensitive molecules, presumably because the loosely packed, dynamic planar surfaces of bicelles mimic a membrane with the type of packing defects that are required for interactions with these molecules.

09:30-09:50 **Ligands Tune the Local and Global Motions of Neurotensin Receptor 1 (NTS1): a Density-Functional Theory (DFT)-Guided Solution NMR Analysis**

Presenting Author: Joshua Ziarek

Joshua Ziarek (Indiana University)

Technical challenges have largely limited the application of NMR to the super-microsecond motional regimes of GPCRs. Focusing on a peptide-binding GPCR, the neurotensin receptor 1 (NTS1), we employed NMR and density functional theory to probe global sub-microsecond motions of 13Cε-methionine residues. Using this approach, known as methionine chemical shift-based global order parameters, we establish that the NTS1 solution ensemble includes substates with lifetimes on several discrete timescales. The longest-lived metastable states reflect those captured in agonist- and inverse agonist-bound crystal structures separated by large energy barriers. Individual methionine residues sense the rapid concerted motions superimposed within these long-lived states. The degree of these fast, global dynamics correlates with ligand pharmacology suggesting a role for conformational entropy in GPCR ligand discrimination.

WEDNESDAY, APRIL 19, 2023 - *continued*

09:50-10:15 **Conformational selection in GPCR activation and inhibition**

Presenting Author: Brian F Volkman

Brian Volkman (Medical College of Wisconsin); Shawn Jenjak (Medical College of Wisconsin); Roman R. Schlimgen (Medical College of Wisconsin); Andrew B. Kleist (Medical College of Wisconsin); Francis C. Peterson (Medical College of Wisconsin)

G protein–coupled receptors (GPCRs) recruit beta-arrestins to coordinate diverse cellular processes, but the structural dynamics driving this process are poorly understood. Atypical chemokine receptors (ACKRs) are intrinsically biased GPCRs that engage beta-arrestins but not G proteins, making them a model system to effector-specific signal transduction. 2D NMR experiments on native, 13C methionine–labeled ACKR3 revealed that beta-arrestin recruitment is associated with conformational exchange at key regions of the extracellular ligand-binding pocket and intracellular effector coupling region. Structure-function analysis of the nanobody-ACKR3 interface identified key contacts and reveal a novel mechanism for GPCR inactivation. Our data suggest that conformational selection guides □-arrestin recruitment by tuning receptor dynamics at intracellular and extracellular regions.

WOC: *in vivo* Spectroscopy (parallel session)
8:45 - 10:15, Nautilus

08:45-09:10 **MR Spectroscopy Development of Metabolic Imaging Biomarkers in Gliomas**

Presenting Author: Changho Choi

Changho Choi (Vanderbilt University Medical Center)

Proton MR spectroscopy (MRS) of 2-hydroxyglutarate (2HG) and glycine in gliomas will be presented. Development of 2HG MRS and dissemination of the MRS protocol will be briefly discussed. In our study in 35 glioma patients, glycine level was positively correlated with cell proliferation rate and inversely correlated with expression of glycine decarboxylase enzymes. 2HG level did not correlate with cell proliferation rate. High level of glycine was significantly associated with short patient survival, irrespective of isocitrate dehydrogenase mutational status. Our data suggest that aggressive gliomas reprogram glycine-mediated one-carbon metabolism to meet the biosynthetic demands for rapid cell proliferation. MRS evaluation of 2HG and glycine may provide metabolic imaging biomarkers that are predictive of tumor progression and clinical outcome.

09:10-09:30 **Lactate Spectroscopy and Spectroscopic Imaging on a portable 46 mT Halbach MRI scanner**

Presenting Author: Itamar Ronen

Chloe Najac (C.J. Gorter Center for MRI, Department of Radiology, LUMC); Tom O'Reilly (C.J. Gorter Center for MRI, Department of Radiology, LUMC); Andrew Webb (C.J. Gorter Center for MRI, Department of Radiology, LUMC); Itamar Ronen (Clinical Imaging Sciences Centre, Brighton and Sussex Medical School, University of Sussex)

Low-field MRI systems (with B0<0.1T) for point-of-care applications are becoming increasingly widespread and could become a turning point for low-income countries and intensive care units. For magnetic resonance spectroscopy in vivo at low field, low SNR for biologically relevant molecules and lack of spectral separability pose significant challenges. Here we demonstrate the possibility of obtaining J-spectroscopic images of lactate solutions at B0=46mT using a Carr-Purcell-Meiboom-Gill (CPMG) sequence added with phase encoding gradients. Lactate spectra fit well with data acquired without spatial encoding and with density matrix simulations. Further work will focus on translating this methodology in vivo and accounting for the complexity of acquiring data in the human brain and other anatomies.

09:30-09:50 **Neural Network Reconstruction of Human Density-Weighted Concentric Ring Trajectory MRSI Data acquired at 3T**

Presenting Author: Nicholas Farley

Nicholas Farley (Purdue Univeristy); Uzay E. Emir (School of Health Sciences, Purdue University / Weldon School of Biomedical Engineering, Purdue University); Matt Rosen (MGH/Martinos Center); Neha Koonjoo (MGH / Martinos Center)

The previous decade saw significant improvements in computing hardware and software development tools which led to an increased interest in deep neural networks and their applications in mathematical problems such as data classification, feature extraction, noise reduction, and function emulation. Our group has attempted to train a deep neural network to learn the relationship between in-vivo Density-Weighted Concentric-Ring-Trajectory Spectroscopic k-space data and its corresponding Cartesian image-domain spectroscopic data. We show a direct comparison of outputs generated from our standard reconstruction pipeline and our neural network's inference. The metabolite peaks of NAA (~2ppm) and tCr (~3ppm) are noticeably well approximated.

WEDNESDAY, APRIL 19, 2023 - *continued*

09:50-10:15 **Magnetic Resonance Applications of Methyl Sulfone in vivo**

Presenting Author: Lana Kaiser

Ioannis Pappas (University of Southern California); Ben A. Inglis (University of California); Lana Kaiser (University of California)

Various applications of a common dietary supplement, methyl sulfone (MSM), are investigated in vivo using NMR and MRI methods. NMR properties of MSM in the healthy human brain and in the blood are examined regarding using MSM in vivo. Some of the applications include the usage as a chemical shift standard in vivo, a marker of the brain/gut axis in healthy aging and neurological diseases and the marker of the viability of blood/brain barrier. Preliminary results of using MSM to measure pH and temperature in the mammalian brain are summarized.

COFFEE BREAK with Exhibit Booths and Posters, Fireside Pavilion
10:15-10:45

WOD: Eclectica (parallel session)
10:45-12:35, Merrill Hall

10:45-11:10 **Zero- to ultralow-field NMR: some resent developments and applications**

Presenting Author: Dmitry Budker

Dmitry Budker (Helmholtz Institute, JGU Mainz and UC Berkeley)

In zero- to ultralow-field (ZULF) NMR, one does not need magnets in some or all of the three stages of an experiment: polarization, encoding, and detection. This unusual NMR modality has witnessed rapid development since the advent of compact and sensitive noninductive sensors, especially, atomic magnetometers that are now available commercially. In this talk we will discuss several recent ZULF NMR experiments carried out by our group and collaborators, demonstrating applications in areas as diverse as searches for beyond-the standard-model particles and interactions, monitoring chemical reaction dynamics within metal catalytic reactors, and detection of breaking down of membranes of biological cells as a result of chemotherapy. ZULF NMR may be combined with hyperpolarization and radioactive detection overcoming the sensitivity limitations.

11:10-11:30 **Optically Detected NMR of Photochemically Hyperpolarized Molecules**

Presenting Author: Danila Barskiy

Liubov Chuchkova (1. Institut fr Physik, Johannes Gutenberg Universitt-Mainz, 2. Helmholtz-Institut Mainz, GSI Helmholtzzentrum fr Schwerionenforschung); Roman Picazo-Frutos (1. Institut fr Physik, Johannes Gutenberg Universitt-Mainz, 2. Helmholtz-Institut Mainz, GSI Helmholtzzentrum fr Schwerionenforschung); James Eills (Institute for Bioengineering of Catalonia); Oleg Tretiak (1. Institut fr Physik, Johannes Gutenberg Universitt-Mainz, 2. Helmholtz-Institut Mainz, GSI Helmholtzzentrum fr Schwerionenforschung); Yinan Hu (State Key Laboratory of Brain and Cognitive Science, Institute of Biophysics, Chinese Academy of Sciences); Danila Barskiy (1. Institut fr Physik, Johannes Gutenberg Universitt-Mainz, 2. Helmholtz-Institut Mainz, GSI Helmholtzzentrum fr Schwerionenforschung); Sven Bodenstedt (ICFO-Institut de Cincies Fotniques, The Barcelona Institute of Science and Technology); Jacopo de Santis (ICFO-Institut de Cincies Fotniques, The Barcelona Institute of Science and Technology); Michael C. D. Tayler (ICFO-Institut de Cincies Fotniques, The Barcelona Institute of Science and Technology); Dmitry Budker (1. Institut fr Physik, Johannes Gutenberg Universitt-Mainz, 2. Helmholtz-Institut Mainz, GSI Helmholtzzentrum fr Schwerionenforschung, 3. Department of Physics, University of California); Kirill Sheberstov (Ecole Normale Superieure Paris)

Photochemically induced dynamic nuclear polarization (photo-CIDNP) enables nuclear spin ordering by irradiating a sample with light. An approach for measuring photo-CIDNP using fast-field-cycling NMR at nanotesla to microtesla fields is presented here for a model system. We demonstrate that photo-CIDNP can be optically detected for compounds with millimolar concentrations without isotopic enrichment. Spin hyperpolarization of protons higher than 0.1% was achieved by irradiating the sample with light at 20 mT. The proposed approach opens a new spectroscopic modality for spin-chemistry applications. Being insensitive to susceptibility-induced magnetic inhomogeneity, the method may be used to study relaxation rates at low fields and may help resolve open questions regarding the nature of avian magneto sensing.

WEDNESDAY, APRIL 19, 2023 - *continued*

11:30-11:50 **Enabling Operando 17O NMR of Lithium-Oxygen Batteries to Study Degradation**

Presenting Author: James H. J. Ellison

James Ellison (University of Cambridge); Clare P. Grey (University of Cambridge)

Lithium-Oxygen batteries (LOB) promise exceptional energy density, however, cells are very short lived, due to breakdown caused by reactive oxygen species. Operando 17O NMR being quantitative, non-invasive and real-time is a tantalising technique to study LOB.

We demonstrate that using enrichment, optimised CPMG/DFS and cell design, we can acquire spectra on the timescale of minutes. Gaussian processes are then used to fit the data and provide uncertainty estimates. We test to what extent degradation differs between electrochemical cycling and "rest" periods as well as observing changes in the amount of discharge/breakdown products with depth of charge and voltage applied, thus offering mechanistic insight to guide development of longer-lived cells. The methods generalise to operando studies of other similarly challenging nuclei.

11:50-12:10 **Iron Oxide Nanoparticle Quantification in Cryopreserved Rat Kidneys using Ultra Low Frequency Longitudinally-Detected Electron Paramagnetic Resonance (LOD-EPR)**

Presenting Author: Saurin Kantesaria

Saurin Kantesaria (University of Minnesota); XUEYAN TANG (University of Minnesota, Center for Magnetic Resonance Research); Steven Suddarth (University of Minnesota, Center for Magnetic Resonance Research); John Bischof (University of Minnesota, Department of Mechanical Engineering); Michael Garwood (University of Minnesota, Center for Magnetic Resonance Research)

Currently there is no low-cost method to nondestructively track IONPs in organs across a wide concentration range (0.05-100 mg Fe/mL). To address this, our lab has developed a low-cost, LOngitudinally Detected Electron Paramagnetic Resonance (LOD-EPR) system that can detect electron moments in IONPs. This work aims to evaluate LOD-EPR performance in terms of IONP quantification accuracy and effects of IONP behaviors such as particle aggregation on signal in solution and in biopsies of IONP-perfused rat kidneys. Linearity is demonstrated for LOD-EPR spectral peak amplitude vs concentration in 0.05-10 mg Fe/mL IONP samples. Aggregation decreases LOD-EPR signal intensity by ~50%. LOD-EPR signal is seen in IONP-perfused kidney biopsy compared to the baseline level signal in a fresh rat kidney biopsy.

12:10-12:35 **DNP Tensor Polarization Enhancement for Nuclear Physics Targets**

Presenting Author: Elena Long

Elena Long (University of New Hampshire)

Dynamic Nuclear Polarization targets have allowed for many spin observables to be measured in nuclear physics experiments. In spin-1 materials, the tensor polarization can also be enhanced and used to probe quark and nuclear structure. Tensor enhancement can be achieved by applying an additional semi-selective saturation RF in addition to microwave DNP enhancement. Such as system has been built at the University of New Hampshire for use in upcoming experiments at Jefferson Lab. Any overview of the process and target DNP system will be discussed.

WOE: BioSolids III (parallel session)
10:45-12:30, Chapel

10:45-11:10 **Magnetic-Alignment of Nanodiscs and NMR Applications**

Presenting Author: Ayyalusamy Ramamoorthy

Ayyalusamy Ramamoorthy (University of Michigan); Samuel McCalpin (University of Michigan); Bankala Krishnarjuna (University of Michigan); Thirupathi Ravula (University of Michigan)

A major focus of our research has been on the development of membrane mimetics (such as bicelles and nanodiscs) to enable the applications of solution and solid-state NMR experiments to study the dynamics structures of membrane-associated peptides and proteins. In this talk, the development of the nanodisc technology for NMR-based studies to probe the structural interactions between membrane-bound proteins such as cytochromes (~16-kDa b5, ~57-kDa P450, ~80-kDa P450-reductase) and to characterize membrane stabilized oligomeric amyloid intermediates will be presented. In addition, the use of magnetically-aligned nanodiscs to measure residual dipolar couplings (RDCs) and residual quadrupolar couplings (RQCs) from water-soluble biomolecules (such as proteins, RNA, and small molecules) will also be presented.

WEDNESDAY, APRIL 19, 2023 - *continued*

11:10-11:30 **2D DNP MAS NMR of Newborn Coral Shows that Glucose Diet Leads to Modulation of Polysaccharide and Protein Levels**

Presenting Author: Gil Goobes

Saja Nasser (Dpt of Chemistry, Bar-Ilan University); Maayan Neder (Dpt. of Marine Biology, Haifa University); Boran Uluca (Institute of Complex Systems, Juelich Research Center); Umit Akbey (Institute of Complex Systems, Juelich Research Center); Henrike Heise (Institute of Complex Systems, Juelich Research Center); Tali Mass (Dpt. of Marine Biology, Haifa University); Gil Goobes (Bar-Ilan University)

Hard coral homeostasis and skeletal calcification has been compromised by ocean acidification and temperature rise, disrupting cross-symbiont interactions between corals and guest photosynthetic alga. Newborn corals transform from swimming planula to polyps adhering to ocean bottom and producing protective exoskeleton. Knowledge of their skeletogenesis and tissue production are crucial to survivability under detrimental seawater conditions. Scleractinian corals feed on predation of a variety of small organisms and obtain nutrients from symbiont algae and resident gut bacteria. Direct impact of food source on either tissue or skeletal growth is unavailable.

We utilized Dynamic nuclear polarization to indicate changes in polysaccharide/protein profiles in planula and polyps by feeding with either 13C carbonate/glycine or 13C6-glucose/glycine and a metabolite associated with bacterial activity.

11:30-11:50 **Measuring Conformational Transitions in Cellular Prion Protein in Condensed Phases through 1D 13C NMR**

Presenting Author: Marcus Tuttle

Marcus Tuttle (Yale University); Yangyi Liu (Yale University); Haote Li (Yale University); Mikhail A. Kostylev (Yale School of Medicine); Daniel J. Walsh (Geisel School of Medicine at Dartmouth); Stephen M. Strittmatter (Yale School of Medicine); Surachai Supattapone (Geisel School of Medicine at Dartmouth); Victor Batista (Yale University); Kurt W. Zilm (Yale University)

Cellular prion protein (PrPC) has a structured C-terminal domain and an intrinsically disordered N-terminus. However, PrPC can also adopt additional phases, including a meta-stable conformation that undergoes maturation. Understanding these states is important to understanding PrPC's role in diseases such as Alzheimer's (AD) and prion diseases. To study these states, we developed a gradient descent-based method to resolve secondary structural distributions from a single 1D 13C NMR spectrum of a protein. We call this Secondary Structure Distribution by NMR (SSD-NMR). We validated SSD-NMR with over 1000 simulated spectra and from experimental data. We then use SSD-NMR to develop a model wherein PrPC LLPS is coupled to an equilibrium between canonical PrPC and a minor conformation with a more structured N-terminus.

11:50-12:10 **Structural Dynamics of Fungal Cell Walls and Remodeling by Osmotic Stress Elucidated by Solid-State NMR**

Presenting Author: Liyanage Devthilini Fernando

Liyanage Fernando (Michigan State University); Malitha Widanage (Louisiana State University); Frederic Mentink-Vigier (National High Magnetic Field Laboratory, Florida State University); Sungsool Wi (National High Magnetic Field Laboratory/FSU); Andrew S. Lipton (Environmental Molecular Sciences Laboratory); Nancy Washton (Enviromental Molecular Sciences Laboratory); Jean-Paul Latge (Unit des Aspergillus, Dpartement de Mycologie, Institut Pasteur); Tuo Wang (Michigan State University)

Here we present the use of multidimensional solid-state NMR and dynamic nuclear polarization (DNP) techniques for characterizing the polysaccharides and proteins in pathogenic fungi including Aspergillus, Candida, and Rhizopus species. The story, comprised of five recent studies, summarized how fungal cell wall is structured in mycelia and conidia, and how the structure changes in response to external stresses, such as antifungal drug and salinity, and internal factors, such as carbohydrate deficiency and mutation. These studies yield essential information about carbohydrate structures of the cell walls and their adaptations at atomic levels that can serve as potential targets for discovering novel antifungal compounds with broad spectrums and improved efficacy.

12:10-12:30 **Solid State NMR Characterization of Low Complexity Protein Sequence Assembly Mechanisms**

Presenting Author: Dylan T. Murray

Dylan Murray (University of California, Davis); Upasana Sridharan (University of California, Davis); Yuuki Wittmer (University of California, Davis); Blake Fonda (University of California, Davis); Khaled Jami (University of California, Davis); Estely Carranza (University of California, Davis); Kayla Osumi (University of California, Davis); Daniel Farb (University of California, Davis)

Proteins harboring low complexity amino acid sequences form organized and condensed assemblies functionally and pathologically in living cells. These proteins are challenging to study using solid state NMR due to their degenerate amino acid sequences. We present our approach using an existing computational assignment algorithm that has successfully yielded unambiguous results for four such proteins. We then illustrate the power of these results in characterizing the molecular mechanisms for the macroscopic assembly of these fascinating protein domains.

WEDNESDAY, APRIL 19, 2023 - *continued*

WOF: Small Molecules: Polarization, Methods (parallel session)
10:45-12:30, Nautilus

10:45-11:10 **Applying hyperpolarization to break sensitivity barriers of NMR for analysis of complex mixtures**

Presenting Author: Mathilde Hauge Lerche

Mathilde Lerche (Technical University of Denmark)

With hardware development, smart acquisition design and in tandem with other methodologies, the sensitivity drawback of NMR has been circumvented for numerous but specific applications. The potential of quantitative NMR is, however, far greater than currently exploited.

Hyperpolarization by dissolution dynamic nuclear polarization (dDNP) has recently been applied to enhance the resolution and sensitivity of NMR to detect compounds in complex mixtures. We have developed a stable isotope tracer-based hyperpolarized NMR method aiming to quantitatively measure metabolic flux with high sensitivity and high contrast. With this method metabolic pathways and networks can be mapped.

In the presentation we consider selected studies, discuss advantages and disadvantages of the hyperpolarization method in context of the studies and give perspectives to further developments.

11:10-11:30 **NMR and DNP Tools to Decipher Whole-Cell Catalysis: The Effect of Substrate Mixtures**

Presenting Author: Francesca Sannelli

Francesca Sannelli (Technical University of Denmark); Pernille Rose Jensen (Technical University of Denmark, DTU Heath Tech); Sebastian Meier (Technical University of Denmark, Chemistry)

NMR spectroscopy has been widely explored to study biochemical function in cells and biofluids. Non-natural modulation of intracellular chemistry by effectors often remains elusive. This response is relevant for a deep understanding of biochemistry, for engineering and for industrial bio-production with non-natural reactions using catalysts of close-to-zero cost, especially when engineering is not possible. NMR spectroscopy is adaptable and minimally invasive. Using NMR and dDNP-NMR, we found that bio-sourced substrate mixtures, can massively reroute the central metabolism of Baker's yeast toward formation of C-C bonds on furfural. Carbon from glucose sequesters to 80% adducts and only 20% ethanol. Studies on the influx of glucose from fermentation to minor pathways producing amino acid and industrial precursors are ongoing.

11:30-11:50 **NMR-Based Fragment Screening of Biomolecular Targets from the SARS-CoV-2 genome**

Presenting Author: Maria Alexandra Wirtz Martin

Maria Wirtz Martin (Goethe Universitt); Hannes Berg (Goethe Universitt); Sridhar Sreeramulu (Goethe Universitt); Christian Richter (Goethe Universitt); Verena Linhard (Goethe Universitt); Anna Niesteruk (Goethe Universitt); Harald Schwalbe (Goethe Universitt)

The emergence of the SARS-CoV-2 virus resulted in a worldwide pandemic that not only changed our everyday day life, but also has given birth to several research initiatives focusing on viral research and drug development. Within the COVID19-NMR, we undertook a massive research initiative involving world-wide NMR groups contributing towards the production, characterization and screening of viral proteins and RNA.

Fragment based screening by NMR aims to find compounds which act as starting points for the development of drugs. We have screened more than 20 RNA and 25 proteins, thus obtaining high quality hits. 311 binders across the 25 SCoV-2 proteins were identified and 69 binders were found that interact with different structured RNA elements in the SCoV-2 RNA genome.

WEDNESDAY, APRIL 19, 2023 - *continued*

11:50-12:10 **Parahydrogen Hyperpolarization as a Tool for Sensitivity Enhanced NMR Metabolomics**

Presenting Author: Indrek Reile

Kerti Ausmees (National Institute of Chemical Physics and Biophysics); Nele Reimets (National Institute of Chemical Physics and Biophysics); Sirje Vija (National Institute of Chemical Physics and Biophysics); Merle Uudsemaa (National Institute of Chemical Physics and Biophysics); Aleksander Trummal (National Institute of Chemical Physics and Biophysics); Indrek Reile (National Institute of Chemical Physics and Biophysics)

Parahydrogen hyperpolarization offers a relatively accessible way of increasing NMR signals by approximately three orders of magnitude over what is offered by the regular means of NMR sensitivity. Applied to chemical analysis of mixtures, this manifests in a substantial lowering of the limit of detection (LoD), allowing to adopt NMR in studies that may not be feasible otherwise.

We will show that parahydrogen hyperpolarization can be applied to analysis of human biofluids, allowing to access metabolic information from mid-nanomolar concentration analytes. We will show proof of concept applications for targeted metabolite analysis, demonstrate the feasibility of hyperpolarized spectral libraries and give examples of metabolite classes that we have analyzed by parahydrogen hyperpolarization (e.g., nicotinamide derivatives, nucleosides, nucleotides, oligopeptides).

12:10-12:30 **Applications of long-lived states of Methylene Protons in Achiral Molecules**

Presenting Author: Anna Sonnefeld

Anna Sonnefeld (Department of Chemistry, Ecole Normale Superieure); Aiky Razanahoera (Department of Chemistry, Ecole Normale Superieure); Philippe Pelupessy (Department of Chemistry, Ecole Normale Superieure); Geoffrey Bodenhausen (ENS); Kirill Sheberstov (Ecole Normale Superieure Paris)

Long-lived states have lifetimes that can be much longer than the longitudinal relaxation time. Here we show that proton long-lived states can be excited in common molecules containing neighboring methylene groups without the need for a chiral center, including neurotransmitters like γ-aminobutyric acid, acetylcholine and dopamine, and bioactive compounds like β-alanine, taurine and homotaurine. We show that long-lived states can be excited simultaneously in several molecules in a mixture and be used to enhance contrast in MRI experiments. This contrast can either be achieved by selective excitation and readout schemes that only address one component of a sample, or by exploiting differences in long-lived state lifetimes.

ASILOMAR LODGER LUNCH for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees may purchase a lunch ticket bundle or dinner ticket online by April 7.
12:45-14:00, Crocker Dinning Hall

POSTER SESSION with Exhibit Booths
14:00-15:45, Fireside Pavilion
See Poster Listings at end of document.

Tutorial and Award Session
16:00-18:00, Merrill Hall

16:00-16:35 **Tutorial - Low-Field NMR and MRI- Opportunities,** Mark Conradi (*ABQMR, Emeritus Washington University St. Louis*)

16:35-17:10 **Tutorial - Hyperpolarization: An Overview of Principles, Methods, and Applications,** Boyd Goodson (*Southern Illinois University*)

17:10-17:35 **Varian Young Investigator Awardee**

17:35-18:00 **Award Presentations (Students/Postdoc & Corp Vendor Contest)**

ASILOMAR LODGER DINNER for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees may purchase a lunch ticket bundle or dinner ticket online by April 7.
18:00 – 19:00, Crocker Dinning Hall

VENDOR HOSPITALITY SUITES
From 18:30

THURSDAY, APRIL 20, 2023

Early Morning Lecture Series (for Students & ALL who wish to learn!)
07:00-07:50, Merrill Hall

07:00-07:50 **Understanding NMR Spectroscopy** Part 4 of 4

Presenter: James Keeler

Would you like to deepen or brush up your understanding of NMR theory? Join us for a morning lecture series by **James Keeler** (University of Cambridge), author of *Understanding NMR Spectroscopy*. These lectures assume only a modest prior knowledge of NMR theory and will cover some key topics of wide interest to budding NMR spectroscopists. Topics will include: Energy levels, Hamiltonians and operators; Introducing and using product operators; Relaxation; Coherence selection by phase cycling and field gradients

ASILOMAR LODGER BREAKFAST for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees cannot purchase breakfast tickets, but you can purchase lunch and dinner tickets by April 7.
07:30-09:00, Crocker Dining Hall

ThOA: BioSolution: Structures and Methods (parallel session)
8:45 - 10:10, Merrill Hall

08:45-09:10 **A great peak picker, and what you can do with it: bioMolecule assignment, methylNOESYs, and Universal Saturation Transfer Analysis**

Presenting Author: Andrew J Baldwin

Andrew Baldwin (University of Oxford)

Virtually all NMR analysis requires peak picking (to get peak locations/shape/intensity). We present UnidecNMR, a deconvolution algorithm that (semi) automatically peak picks in 1-4D data, whose results match those of experienced users. This enables streamlined backbone assignment, and analysis of high molecular weight complexes via 4D methyl NOE data. Applying this to the saturation transfer experiment (uSTA) of complex 1D ligand spectra provides accurate ligand poses. Wiring this up to a Bloch-McConnell simulation, we also obtain reliable Kon/Koff and KD values. We use this to explore a range of ligand/protein interactions, including sugars binding COV19 Spike proteins. We've embedded this all in nice software so you can all test it out.

09:10-09:30 **Tackling a tripartite glycan conundrum: Flexibility/Sparse structural data/Signal resolution**

Presenting Author: Darón I. Freedberg

Daron Freedberg (CBER/FDA)

Despite the vast diversity of glycans, we face common, and often interconnected, challenges in their NMR structural studies: 1) discrimination of distinct conformations amongst a conformation-rich landscape, 2) limited structural data and 3) spectral overlap. We've addressed these issues by improving sensitivity and resolution, though not simultaneously. We will show that labile ¹H signals help alleviate two of these challenges and thus increase the repertoire of structural data. Ultimately, these help to discriminate unique conformations and refine structural models. I will also present methods to extract thermodynamic and kinetic data for conformational equilibria in glycans in the fast exchange. Finally, I will discuss our ongoing efforts to enhance the resolution for the structural studies of larger oligosaccharides.

09:30-09:50 **Analyzing AlphaFold Structures with NMR data**

Presenting Author: Joseph Sachleben

Isabelle Gagnon (University of Chicago); Tobin Sosnick (University of Chicago); Jeffery Ellena (University of Virginia); Urszula Derewenda (University of Virginia); Zygmunt Derewenda (University of Virginia); Eric Jonas (University of Chicago); Joseph Sachleben (University of Chicago)

The recent introduction of AlphaFold has fundamentally changed our ability to predict the structure of proteins from their primary sequence. We describe some straightforward NMR and computational methods to test these predicted structures. NOESY spectra provide a potential contact map between residues. TALOS torsion angles from backbone chemical shift constrain the predicted structure's secondary structure. We compare NMR data to the predicted structure of three proteins. We find that scores derived NMR data scale with the rmsd difference between predicted and determined structures. Further understanding of the NMR derived scores will be made by mining the data available on on-line data bases. Insights from this work will lead to new faster methods of NMR structural refinement and assignment.

THURSDAY, APRIL 20, 2023 - *continued*

09:50-10:10 **Ligand-capped Co(II) Multiplies the Value of the Double-Histidine Motif for PCS NMR Studies**

Presenting Author: Angela M. Gronenborn

Wenkai Zhu (University of Pittsburgh School of Medicine); Darian T. Yang (University of Pittsburgh School of Medicine); Angela Gronenborn (University of Pittsburgh School of Medicine)

In structural studies by NMR, pseudocontact shifts (PCSs) provide both angular and distance information. For proteins, incorporation of a di-histidine (diHis) motif, coordinated to Co2+ has emerged as an important tool to measure PCS. Here, we show that using different Co(II)-chelating ligands, such as NTA and IDA, resolves the isosurface ambiguity of Co2+-diHis and yields orthogonal PCS datasets with different Delta-chi tensors for the same diHis bearing protein. In addition, the use of capping ligands effectively eliminates undesired intermolecular interactions, mediated by metal binding, which can be detrimental for PCS studies. Devising and employing ligand-capping strategies afford versatile and powerful means to obtain multiple orthogonal PCS datasets, significantly extending the use of the diHis motif for structural studies by NMR.

ThOB: Hyperpolarization (parallel session)
8:45 - 10:10, Chapel

08:45-09:10 **Patches and Pockets of Weird Water -- Exploring New Frontiers with ODNP**

Presenting Author: John M Franck

Alec Beaton (Syracuse University); Alexandria Guinness (Syracuse University); Alexandria Guinness (Syracuse University); John Franck (Syracuse University)

Overhauser Effect Dynamic Nuclear Polarization (ODNP) utilizes resonant electron spins to enhance and isolate the properties of small pockets or patches of water in the hydration layer of large macromolecules. Here, it explores the dynamics of water trapped inside small pockets by surfactants, and water along the interface of a transmembrane protein and at the surface of a globular protein. These studies share a common theme that while ODNP easily analyzes viscous and phase heterogeneous samples containing large macromolecules or macromolecular complexes, it suffers from some difficulties by requiring low fields and operation in conjunction with a functioning EPR resonator. In overcoming some of these difficulties, we develop new methods for visualizing and resolving NMR signal.

09:10-09:30 **Steady-state Hyperpolarization of 1H in Liquids by Overhauser Dynamic Nuclear Polarization with 13C–1H Polarization Transfer**

Presenting Author: Yu Rao

Yu Rao (EPFL); Amrit Venkatesh (EPFL); Pinelopi Moutzouri (EPFL); Lyndon Emsley (EPFL)

Dynamic nuclear polarization (DNP) is a method that can significantly improve the sensitivity of NMR. The only effective DNP mechanism for in situ hyperpolarization in solution is Overhauser DNP, which is usually inefficient for 1H at high magnetic fields. Here we demonstrate the feasibility of exploiting the efficient Overhauser DNP on 13C to obtain significant steady-state 1H hyperpolarization in solution at high magnetic field. Using a 400 MHz gyrotron-equipped 3.2 mm MAS DNP system, we obtain 1H DNP enhancement factors of 48, 8, and 6 for chloroform, tetrachloroethane, and phenylacetylene, respectively, at room temperature.

09:30-09:50 **Structural Description of CaCO3 Prenucleation Clusters through 13C MAS-DNP NMR**

Presenting Author: Thierry Azaïs

Thierry Azais (Sorbonne Universite); Tristan Georges (Sorbonne Universite); Vinavadini Ramnarain (IPCMS); Christel GERVAIS (LCMCP - Sorbonne Universite); Clment Sanchez (Collge de France); Ovidiu Ersen (IPCMS)

Calcium carbonate (CaCO3) is one of the most significant biominerals in Nature found in the skeletons of sea urchins, cuticles of crustaceans, mollusks shells or corals. It was recently shown that CaCO3 crystallization is occurring through a non-classical nucleation pathway for which various intermediate phases are involved. In this communication, we show that 13C MAS-DNP NMR allows the comprehension of the initial step of CaCO3 nucleation including the structural atomic description of CaCO3 prenucleation clusters. This method combines two advantages (i) the sensitivity enhancement induced by DNP, particularly useful for physiological concentration (1-4 mM), but also (ii) the low temperature that is quenching the nucleation process and allow the stabilization of such transient species.

THURSDAY, APRIL 20, 2023 - *continued*

09:50-10:10 **Study of Gadolinium Effects in the Hyperpolarization of [15N3]Metronidazole: an FDA-approved Antibiotic and Potential Hypoxia Probe**

Presenting Author: David O. Guarin Bedoya

David Guarin Bedoya (Athinoula A. Martinos Center for Biomedical Imaging, Department of Radiology, Massachusetts General Hospital); Sameer M. Joshi (Department of Chemistry, Integrative Biosciences (Ibio), Karmanos Cancer Institute (KCI), Wayne State University,); Anna Samoilenko (Department of Chemistry, Integrative Biosciences (Ibio), Karmanos Cancer Institute (KCI), Wayne State University); Mohammad S. H. Kabir (Department of Chemistry, Integrative Biosciences (Ibio), Karmanos Cancer Institute (KCI), Wayne State University); Erin E. Hardy (Department of Radiology, Athinoula A. Martinos Center for Biomedical Imaging, Massachusetts General Hospital, 149 13th St.); Atsushi M. Takahashi (Department of Brain and Cognitive Sciences, McGovern Institute for Brain Research, Massachusetts Institute of Technology); Jan H. Ardnkjaer-Larsen (Department of Health Technology, Technical University of Denmark, 348, rsteds Pl.,); Eduard Y. Chekmenev (Department of Chemistry, Integrative Biosciences (Ibio), Karmanos Cancer Institute (KCI), Wayne State University); Yi-Fen Yen (Athinoula A. Martinos Center for Biomedical Imaging, Department of Radiology, Massachusetts General Hospital)

In this work, we hyperpolarized [15N3]metronidazole(MNZ) with dynamic nuclear polarization(DNP) technique. Metronidazole is an FDA-approved antibiotic that can be potentially employed as a hypoxia-sensing probe. A good polarization (~6%) was achieved with very short polarization build-up time constants (~12min) and,T1 and T2 of 343 s and 20 s in liquid-state respectively. We used Electron Paramagnetic Resonance(EPR) spectroscopy to show that a sample of [15N3]MNZ + trityl AH111501 had narrower EPR linewidth and larger magnitude than AH111501 alone, indicating an efficient polarization transfer from the radical electrons to 15N and supporting our observations of fast DNP buildup. We also demonstrated that the addition of a gadolinium-based compound to the [15N3]MNZ+AH111501 sample broadened the EPR spectrum and prolonged DNP buildup as observed.

ThOC: Rethinking What We Know in MRI (parallel session)
8:45 - 10:10, Nautilus

08:45-09:10 **Hemodynamic brain mapping with high-field (14T) preclinical fMRI methods**

Presenting Author: Xin Yu

Xin Yu (MGH)

"Functional" MRI is developed to map neurovascular coupling-based hemodynamic changes, i.e. the CBV, CBF, and BOLD signals, as indirect measures of neuronal activity. Despite existing spatial specificity functional mapping studies, one intriguing question is "what can we detect when the spatial resolution is improved from the millimeter to the tens-of-micron scale?" Here, I will present two sets of high-resolution fMRI methods: line-scanning fMRI and single-vessel fMRI, using the 14T MRI scanner. The line-scanning fMRI allows laminar hemodynamic mapping with 50-micron resolution and 5-to-50ms sampling rates. The single-vessel fMRI enables the detection of arteriole and venule (20-70 micron)-specific hemodynamic responses across different brain regions in animals. Both methods enable the circuit-specific or vessel-specific hemodynamic mapping of awake transgenic mouse models.

09:10-09:30 **Assess Gas Flow field of the Lung using High Spatiotemporal Resolution Dynamic Hyperpolarized 129Xe MRI**

Presenting Author: Haidong Li

Hongchuang Li; Ming Zhang; Xiaoling Liu; Xiuchao Zhao, Yeqing Han; Chaohui Ye; Xin Zhou (National Center for Magnetic Resonance in Wuhan, Wuhan Institute of Physics and Mathematics, Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences, Wuhan, China)

Noninvasively imaging dynamic ventilation with high spatiotemporal resolution is of great demand for diagnosing lung diseases that related to obstructive ventilation. Unfortunately, limited by the short breathing cycle, it is difficult to obtain high temporal resolution and quantitatively analyze the airflow distribution. In this study, we propose a method for obtaining dynamic ventilation images with super-spatiotemporal-resolution via hyperpolarized 129Xe MRI, and obtain the flow field of hyperpolarized 129Xe gas in the lung by drawing lessons from optical flow methods. Our preliminary results demonstrated the feasibility of the optical flow methods for evaluating dynamic ventilation function regionally in vivo, which would be helpful for assessing the pathological changes of the lung caused by the diseases that related to dynamic ventilation abnormality.

THURSDAY, APRIL 20, 2023 - *continued*

09:30-09:50 **Development of a Compact NMR System to Measure pO2 in a Tissue-Engineered Graft**

Presenting Author: Efrain Torres

Efrain Torres (University of Minnesota); Saurin Kantesaria (University of Minnesota); Paul Wang (University of Minnesota); Parker Jenkins (University of Minnesota); Leah Steyn (The University of Arizona); Lance DelaBarre (University of Minnesota); Taylor Froelich (University of Minnesota); Daniel Pizetta (University of Sao Paulo); Dimitrios Sakellariou (KU Leuven); Alberto Tannus (University of Sao Paulo); Klearchos Papas (The University of Arizona); Michael Garwood (University of Minnesota)

A tissue-engineered graft (TEG) containing islet cells could serve as a bioartificial pancreas when implanted in the forearm of diabetic patients. The bioartificial pancreas requires supplemental oxygen delivery to ensure oxygen remains within physiological levels1. To monitor its oxygen levels, TEGs contain perfluorocarbons with oxygen-sensitive R1 values2. Here we present a tabletop oxygen scanner based on 19F NMR relaxometry. The system will be used in research and upcoming clinical trials evaluating a potential cell-based functional cure for type I diabetes.

09:50-10:10 **High Resolution MRI with Low Amplitude Gradient**

Presenting Author: Dan Xiao

Mark Armstrong (University of Windsor); Dan Xiao (University of Windsor)

The Pi Echo Planar Imaging (PEPI) sequence employs multiple refocusing RF pulses and unbalanced spatial encoding gradients. The phase accumulates throughout the entire echo train, so that the gradient duty cycle can be drastically reduced compared to fast spin echo (FSE). A shorter echo spacing may also be achieved. PEPI requires near ideal 180° refocusing pulses to eliminate the coherence pathway artifacts, which has limited its application to 3D imaging of small samples confined to the homogeneous region of the RF coil. Sufficient quality 2D PEPI could not be obtained due to the imperfect slice profile. In this work, an optimized phase cycling scheme is proposed to minimize coherence pathway artifacts with non-ideal refocusing pulses and enable 2D PEPI imaging.

COFFEE BREAK with Exhibit Booths and Posters, Fireside Pavilion
10:20-10:45

ThOD: Methodology and Applications of Inorganic Materials (parallel session)
10:45-12:35, Merrill Hall

10:45-11:10 **1H CSA: Friend or Foe?**

Presenting Author: Frederic A. Perras

Scott A. Southern (Ames National Laboratory); Takeshi Kobayashi (Ames National Laboratory); Alexander Paterson (University of Wisconsin-Madison); Yusuke Nishiyama (JEOL Ltd.); Frederic Perras (Ames National Laboratory)

Despite the high sensitivity, and recent resurgence, of 1H solid-state NMR, measurements of 1H chemical shift anisotropy (CSA) have remained rather niche. In many instances, we would even consider it a nuisance that leads to decoherence and t1 noise in 1H dipolar recoupling. This presentation will cover the development of highly stable dipolar recoupling methods that decouple the 1H CSA in addition to new 1H CSA recoupling schemes that enable the measurement of tensor skew, and small anisotropies. Lastly, the utility and limitations of 1H CSA for the measurement of dynamic information in low-sensitivity samples, such as heterogeneous catalysts, will be discussed.

11:10-11:30 **Solid-state NMR Characterizations of the Reorientational Dynamics of A-site Cations in 2D Organic-Inorganic Hybrid Perovskite**

Presenting Author: Tsyr-Yan Dharma Yu

Cheng-Chieh Lin (NTU-MST, National Taiwan University); Shing-Jong Huang (Instrumentation Center, National Taiwan University); Pei-Hao Wu (Institute of Atomic and Molecular Sciences, Academia Sinica); Vladimir M. Gelev (Department of Chemistry and Pharmacy, Sofia University); Chun-Wei Chen (Department of Materials Science and Engineering, National Taiwan University); Tsyr-Yan Yu (Institute of Atomic and Molecular Sciences, Academia Sinica)

Organic–inorganic hybrid perovskites (OIHPs) have attracted a significant amount of attention for photovoltaic applications since their power conversion efficiency has reached over 25%. Limited methods are available for investigating the reorientational dynamics of A-site cations in 2D OIHPs, which play a pivotal role in determining their physical properties. We characterized the dynamics of A-site cations using isotope labelling combined with ssNMR methods. While 2H NMR analysis reveals the existence of multiple modes of reorientational dynamics of methylammonium, REDOR NMR of 2D OIHPs incorporating (13C,15N)- methylammonium reflects the averaged dipolar coupling between the two nuclei undergoing different modes of motions. The interplay between the rigidity of the organic spacers and the A-site cations dynamics of 2D OIHPs is clearly revealed.

THURSDAY, APRIL 20, 2023 - *continued*

11:30-11:50 **Rapid dynamic nuclear polarization with conductive polymers**

Presenting Author: Quentin Stern

Quentin Stern (UCBL); Guillaume Verhaeghe (UCBL); Tho El-Dara (UCBL); Charlotte Bocquelet (UCBL); Sami Jannin (UCBL)

Dissolution dynamic nuclear polarization (dDNP) uses the high polarization of electron spins at low temperatures to polarize nuclear spins to near-unity levels on a broad variety of compounds. After dissolving the sample, this hyperpolarization translates into sensitivity gains for liquid-state NMR and MRI of up to five orders of magnitude. Here, we show that DNP is feasible on polyaniline polymers (PANI) at 1.6 K and 7 T and find a surprising variety of DNP mechanisms as a function of radical concentration. DNP on PANI opens the perspective of efficient DNP at moderate temperatures and hence, without the need for liquid helium since electrons in chiral PANI can be hyperpolarized by chirality-induced spin selectivity.

11:50-12:10 **Cross Polarization from Dipolar-Order under Magic Angle Spinning: The ADRF-CPMAS NMR Experiment**

Presenting Author: Tamar Wolf

Tamar Wolf (Weizmann Institute of Science); Lucio Frydman (Weizmann Institute of Science)

Techniques for enhancing low-gamma X-spin signals are crucial in solid-state NMR. The leading method to sensitize unreceptive X nuclei is Hartmann-Hahn cross polarization (HH-CP), often executed under MAS. Herein, we explore the possibility of utilizing 1H dipolar order created via adiabatic demagnetization in the rotating frame (ADRF), to enhance the X-spins under MAS. Somewhat unexpectedly, we find that an efficient polarization transfer via ADRF-CPMAS can be possible, exceeding in some instances that of an optimized HH-CPMAS. The experiment requires low-powers on both the 1H and the X channels, and displays unusual zero- and double-quantum matching conditions. These are analytically derived and numerically simulated, in predictions that compare well with experimental 13C and 15N results collected at different spinning speeds.

12:10-12:35 **Nuclear Spins as Probes of Electronic States in Semiconductors and the "Spin Bath" - What Can We Learn from Hyperpolarization via Optical Pumping**

Presenting Author: Sophia Hayes

Weijian Chen (Washington University); Michael West (Washington University)

Optically-pumped NMR in CdTe and GaAs is still yielding insights into both the electronic states in the semiconductors as well as the behavior of the "spin bath". A grand challenge in quantum technologies is the preservation of spin coherence lifetimes. Spin systems that coherently couple to light offer key capabilities for quantum technologies. Here we report on long nuclear spin coherence lifetimes of ^{113}Cd in CdTe that have been polarized through coupling to optically-oriented electrons.

ThOE: Techniques for Small Molecules (parallel session)
10:45-12:35, Chapel

10:45-11:10 **Novel and Robust Conformational Analysis to Advance 3D Structure Characterization of Cyclic Peptides**

Presenting Author: Qi Gao

Qi Gao (Merck); Xiao E. Wang (Merck); Ajay N. Jain (BioPharmics LLC); Edward N. Sherer (Merck); Mikhail Reibarkh (Merck)

The interest in macrocyclic peptides as new scaffolds in the development of novel drugs has significantly increased owing to their potential to interact with novel and challenging biological targets. Facile and precise elucidation of the 3D conformation of such molecules can provide insight into structure-activity relationships, which in turn illuminates molecular design toward improving pharmacological performance. We will present a newly developed approach featuring rapid determination of high-resolution 3D conformational ensembles of cyclic peptides and macrocycles in solution using a small number of NMR restraints using an advanced conformational sampling algorithm. The methodology developed can be applied to many therapeutic peptides and has the potential to contribute to developing novel medicines by enabling rapid access to high-resolution solution conformations.

THURSDAY, APRIL 20, 2023 - *continued*

11:10-11:30 **Operando Metabolomics of Healthy and Cholestatic Liver Tissue Slices By Microfluidic NMR**

Presenting Author: Marcel Utz

Bishnubrata Patra (University of Southampton); Manvendra Sharma (University of Southampton); Ruby Karsten (University of Groningen); Sabeth Verpoorte (University of Groningen); Jan G. Korvink (Karlsruhe Institute of Technology); Marcel Utz (University of Southampton)

A microfluidic platform is described that allows in-situ observation of metabolic processes in live murine liver tissue slices. The system is based on a transmission-line NMR microprobe, which is designed to accommodate a microfluidic chip that holds the tissue slice, and ensures nutrient and oxygen supply, as well as gas exchange and temperature stability.The system is capable of quantifying metabolic production/consumption rates of more than 20 different metabolites from a single tissue slice, with a time resolution of a few minutes.

11:30-11:50 **Integrated Approach of J-resolved STOCSY and INADEQUATE in 13C NMR Metabolomics**

Presenting Author: Mario Uchimiya

Mario Uchimiya (University of Georgia); Malin Olofsson (University of Georgia); McKenzie A. Powers (University of Georgia); Brian M. Hopkinson (University of Georgia); Mary Ann Moran (University of Georgia); Arthur S. Edison (University of Georgia)

Robust annotation of metabolites is a critical task in metabolomics. 13C-experiment INADEQUATE is an ultimate experiment that provides definitive structure based on carbon networks. Despite its utility, it is not always practical to collect INADEQUATE on every sample in a large study because of its relatively long experiment time. Here, we propose an alternative that integrates 13C homonuclear JRES, STOCSY, and INADEQUATE information. We tested this approach using the 13C-labeled endometabolome of a model marine diatom. This approach extracted both known and unknown diatom metabolites with structural information. The ability of this scheme was seen even in sugar regions, which are usually challenging due to severe peak overlap. This approach can maintain the quality of information but saves experiment time.

11:50-12:10 **Ultrafast diffusion NMR: an emerging tool for the analysis of mixtures**

Presenting Author: Rituraj Mishra

Rituraj Mishra (University of Nantes); Jonathan Yong (University of Oxford); Achille Marchand (University of Nantes); Corentin Jacquemmoz (Direction gnrale de l'Armement); Mohammadali Foroozandeh (University of Oxford); Jean-Nicolas Dumez (University of Nantes)

SPatially ENcoded Diffusion Ordered Spectroscopy (SPEN-DOSY) has emerged as a new time-efficient tool for the analysis of mixtures of small molecules in solution. Time efficiency is achieved using the concept of spatial parallelization of the effective gradient area and the data is processed by least-squares optimization to extract diffusion coefficients. The overlapping peaks, however cannot be separated and identified via such processing. We have implemented multivariate processing methods, DECRA, SCORE, OUTSCORE, and a recent univariate processing method, Matrix Pencil Method (MPM) for the separation of such overlap. However, as the number of components increases, it becomes difficult to separate them even with these methods. This limitation is addressed via using the PSYCHE sequence to record SPEN-DOSY NMR data.

12:10-12:35 **NMR as Tool in Photo- and Organocatalysis**

Presenting Author: Prof. Dr. Ruth Gschwind

Ruth Gschwind (University of Regensburg)

The detection and characterization of intermediates in catalytic reactions is crucial for the rational optimization of reaction conditions. However, in many rapidly expanding fields of asymmetric catalysis, mechanistic studies as well as structural investigations on intermediates or intermolecular interactions are scarce. In this talk I will present techniques and methods to extend the application of NMR in photocatalysis and ion pair catalysis and explain their impact on examples.

THURSDAY, APRIL 20, 2023 - *continued*

ThOF: Instrumentation II (parallel session)
10:45-12:30, Nautilus

10:45-11:10 **Frequency Modulation and DNP**

Presenting Author: Daphna Shimon

Daphna Shimon (The Hebrew University of Jerusalem)

In a standard dynamic nuclear polarization (DNP) experiment, we irradiate at a constant microwave (MW) frequency and observe the DNP enhancement of the nuclear signal. Recently, several groups have begun introducing frequency modulation (FM) of the MW irradiation. With FM, it is possible to affect more electron spins during the DNP experiment, often leading to an increase in the DNP enhancement. When several DNP mechanisms are active in the same sample, FM can also cause a change in the relative contribution of each mechanism. In this work, I will discuss performing FM and how it affects the various DNP mechanisms.

11:10-11:30 **Fabrication and Testing of Solenoids Optimized from System-Defined Constraints**

Presenting Author: Jessica I. Kelz

Jessica Kelz (UC Irvine); Jose L. Uribe (UC Irvine); Robert G. Marosi (UC Irvine); Filippo Capolino (UC Irvine); Rachel W. Martin (UC Irvine)

Solenoids are a common transceiver in NMR probes due to robust performance and relative ease of utilization, however published optimizations are limited. We developed a generalized approach that readily enables design and fabrication of solenoids optimized for homogeneity based on instrument-specific constraints using a Biot-Savart approximation. 3D-printed coil templates will be used to fabricate generated 3.2mm designs for a MAS probe including constant-pitch to maximize B1 and variable-pitch to maximize axial homogeneity. Field profiles will be benchtop tested using a high-resolution and automated ball-shift apparatus to assess any limitations prior to further experimental validation. This process is accessible to all instrumentation skill levels, time efficient, can improve performance, and has the potential to benefit more complicated designs in the future.

11:30-11:50 **A Low-Field Permanent Magnet Magnetic Resonance Instrument Designed for Multimodal Imaging: Application in the GAMMA-MRI Project**

Presenting Author: Dimitrios Sakellariou

Rodrigo de Oliveira-Silva (KU Leuven); Tomas Rodriguez (Inspiralia); Renaud Jolivet (Maastrich University); Luis Fraille (Universidad Complutense de Madrid); Magdalena Kowalska (CERN); Jean Noel Hyacinthe@hesge.ch (University of Applied Sciences of Western Switzerland); Julien Rivoire (RS2D); Stavroula Pallada (7HES-SO University of Applied Sciences of Western Switzerland); Dimitrios Sakellariou (KU Leuven)

Low-field magnetic resonance is witnessing renewed interest in the light of permanent-magnet based assemblies that offer transportability, low-cost in acquisition/maintenance, improved sustainability, as well as additional options for innovative customized design. This becomes appealing when MRI is combined with other imaging modalities, and/or even other detection/hyperpolarization methodologies. The GAMMA-MRI project is exploring the possibility to apply MR techniques to spatially encode the photons of hyperpolarized xenon isotopes emission and detecting using gamma detectors surrounding the imaged object. This mode of detection promises improving the limits of detection while maintaining the advantages of high-field MRI. A low-field MRI instrument will be presented which has been custom-designed and build complying with multiple constrains without impacting the quality of the MRI imaging.

11:50-12:10 **NMRduino: a modular, open-source platform for dc and low-field magnetic resonance**

Presenting Author: Michael Tayler

Sven Bodenstedt (ICFO - The Institute of Photonic Sciences); Michael Tayler (ICFO - The Institute of Photonic Sciences)

The "NMRduino" is a single-board magnetic resonance spectrometer based on Arduino that we have developed over recent years to study hyperpolarized NMR systems, fast-field-cycling NMR relaxation, high-resolution spectroscopy, and coherent control at low magnetic fields, as well as teach basic principles of magnetic resonance to student beginners. Having undergone several design iterations and extensive testing, it is ready for community release. In this presentation, we would like to discuss NMRduino's capabilities and show how you can obtain and use it. Main features and applications in latest research will be demonstrated.

THURSDAY, APRIL 20, 2023 - *continued*

12:10-12:30 **Simplifying in-situ photochemical analysis with NMRtorch**

Presenting Author: Wendy Nason-Palmer

Jack E. Bramham (University of Manchester); Wendy Nason-Palmer (Oxford Instruments Inc.); Robin J. Blagg (Oxford Instruments); James Sagar (Oxford Instruments); Alexander P. Golovanov (University of Manchester)

Much of chemistry is photo sensitive, whether that is biological reactions, catalysts, photo switches or degradation. NMR is an excellent tool for characterising the photosensitivity of chemicals or materials due to inherent richness of NMR information and the lack of reliance on optical detection. Here we present a new approach for In-situ photo NMR, NMRtorch. Thich enables convenient and portable in-situ sample illumination without optical fibres or probehead modifications, and can be easily moved between NMR systems, whether benchtop or high field. We will introduce the principles of this approach and demonstrate its performance in several typical photo-NMR applications including photo degradation, photoisomerization and photochemically induced dynamic nuclear polarisation (pCIDNP).

ASILOMAR LODGER LUNCH for attendees lodging at Asilomar. Asilomar lodgers will receive meal tickets at check-in. Off-site attendees may purchase a lunch ticket bundle or dinner ticket online by April 7.
12:45-14:00, Crocker Dinning Hall

POSTER SESSION
14:00-15:45
See Poster Listings at end of document.

ThOG: BioSolids IV (parallel session)
16:00 - 17:45, Merrill Hall

16:00-16:25 **High sensitivity NMR for structural determination of neurodegenerative disease-associated proteins inside cells**

Presenting Author: Kendra K Frederick

Kendra Frederick (UTSouthwestern)

The misfolded proteins associated with neurodegenerative disease can adopt a variety of different conformations, some of which are toxic. Because these proteins have identical amino acid sequences, the cellular environment clearly influences the final state, yet most structural studies do not include the cellular context and, perhaps because we are not studying the correct conformation, not a single therapeutic strategy for these diseases addresses the underlying protein misfolding pathology. Using new sensitivity-enhancement technology for solid state NMR spectroscopy, Dynamic Nuclear Polarization, we study protein structure in native environments - inside living cells - to reveal how both healthy and disease-relevant cellular environments influence protein structure.

16:25-16:45 **Structural insights into the Biofilm Forming Functional Amyloids**

Presenting Author: Umit Akbey

Umit Akbey (Structural Biology, University of Pittsburgh)

Aggregated proteins in the form of amyloid fibrils play a key role/function (functional amyloids) in maintaining the structural integrity of bacterial biofilms. Such functional amyloids strengthen biofilms and are a major threat to human health, since the (chronic) infections they cause are difficult to treat due to the biofilm structural integrity and insufficient penetration of drugs, thus promoting antibiotic resistance (antimicrobial resistance, AMR).

Here, I will present our recent work on NMR spectroscopy based structural characterization of several functional amyloids. Moreover, results from other strutural techniques and biophysical characterization will be presented.

16:45-17:05 **Intrinsic protein disorder in the solid state: a combined solid-state NMR and EPR approach**

Presenting Author: Ansgar Siemer

Sayuri Pacheco (USC); Silvia Cervantes (USC); Dhanya Reselammal (USC); Ansgar Siemer (USC)

When studying intrinsically disorders protein domains (IDDs), a strength of NMR spectroscopy is to provide local structural information via chemical shifts and dynamics via relaxation rates and residual dipolar couplings. A strength of (DEER) EPR, in this context, is to provide distance distributions with unambiguous assignments, making it very complementary to NMR spectroscopy. Here, we use both NMR and EPR in combination with MD simulations to define the conformational ensemble of IDDs that are often found on the surface of cross-β fibrils important in neurodegenerative diseases. In addition, we use a combination of solid-state and solution NMR techniques under MAS to determine the binding site of the co-chaperone DNAJB1 to these IDDs.

THURSDAY, APRIL 20, 2023 - *continued*

17:05-17:25 **Atomic-Resolution Magic-angle spinning NMR Structure of the Protein Encoded by Gene V of fd phage in Complex with its full-length Viral ssDNA**

Presenting Author: Amir Goldbourt

Amir Goldbourt (Tel Aviv University)

Single stranded filamentous bacteriophage viruses undergo an intermediate step where thousands of homodimers of a non-structural protein, gVp, bind newly synthesized strands of DNA, preventing further DNA replication and signaling assembly of new virions at the membrane. Past studies have only been able to model the ssDNA-bound conformation using X-ray and solution NMR structures of isolated dimers. We report here an atomic-resolution magic-angle spinning solid-state NMR structure of a monomer of gVp within the context of an 8233-nucleotide-long ssDNA in the nucleoprotein complex. The model presents significant conformational changes, having a backbone r.m.s.d. of 6.4Å with respect to the free form. We show how these modifications facilitate ssDNA binding mechanism and promote the reported cooperative binding generating the cellular assembly.

17:25-17:45 **Coherent DNP with Chirped Pulses**

Presenting Author: Robert G. Griffin

Yifan Quan (MIT); Manoj Subramanya (National High Magnetic Field Laboratory); Yifu Ouyang (MIT); Michael Mardini (Massachusetts Institute of Technology); Thierry Dubroca (National High Magnetic Field Laboratory); Stephen Hill (National High Magnetic Field Laboratory); Robert Griffin (Massachusetts Institute of Technology)

We present a study of coherent dynamic nuclear polarization (DNP) using frequency swept pulses at 94 GHz (W-band). Using chirped pulses, the polarization transfer efficiency can be optimized and an enhancement ε ~ 496 was observed using 10 mM trityl-OX063 as the polarizing agent in a standard d8-glycerol:D2O:H2O: 6:3:1 glassing matrix at 70K. The frequency swept pulses enhance the nuclear magnetic resonance (NMR) signal, and also reduce the recycle delay, accelerating the NMR signal acquisition.

ThOH: Quantum Calculations of NMR Parameters (parallel session)
16:00 - 17:50, Chapel

16:00-16:25 **The Importance of Nuclear Quantum Effects (Nuclear Delocalization) for Hydrogen Bonding and for Predictions of NMR Parameters**

Presenting Author: Martin Dracinsky

Martin Dracinsky (Institute of Organic Chemistry and Biochemistry)

Hydrogen atom is intrinsically quantum mechanical and nuclear quantum effects (NQEs), such as nuclear delocalization and tunneling are important for its properties. NMR spectroscopy provides a tool for the investigation of NQEs. Recent progress in combining experimental NMR with path-integral molecular dynamics (PIMD) simulations that include NQEs will be discussed. We used this combination of experiment and theory to investigate resonance stabilization of hydrogen bonds and for accurate predictions of isotope shifts. We have also investigated salt-to-cocrystal transformations of multicomponent pharmaceutical solids. A combination of solid-state NMR spectroscopy with DFT-PIMD simulations provides evidence of temperature-induced hydrogen-atom shift in cocrystals with short hydrogen bonds. The hydrogen atom can be significantly delocalized between the acid and the base, forming a hydrogen-bond continuum.

16:25-16:45 **A New Route to Chemical Shieldings and their Intrepretation from First Principles Computations**

Presenting Author: Josef W. Zwanziger

Josef Zwanziger (Dalhousie University)

We have developed a new formalism for calculating chemical shielding in solids and implemented it in the Abinit code. This new approach lets us determine the different contributions of the electronic bands to the shielding tensor, opening the way to detailed interpretation and chemical insight based on the individual orbital contributions. Our approach is based on a perturbative expansion of the total energy as a function of the magnetic field, making use of magnetic translation symmetry. This approach yields a band-by-band decomposition of the shielding and hence direct interpretation in terms of orbitals. We will discuss details of our implementation and examples and comparisons with other approaches.

THURSDAY, APRIL 20, 2023 - *continued*

16:45-17:05 **Relativistic DFT Calculations of NMR Parameters for the Platinum Group Elements**

Presenting Author: Sean Holmes

Sean Holmes (Florida State University); Jasmin Schnzart (Florida State University); Adam Philips (University at Buffalo); Jochen Autschbach (University at Buffalo); Robert Schurko (Florida State University)

The platinum group elements (PGEs), which include Rh, Ru, Pd, Os, Ir, and Pt, are widely used in catalysts, optical devices, sensors, alloys, and many other advanced materials, due to their unique covalent donation bonding. The combination of solid-state NMR (SSNMR) spectroscopy and density functional theory (DFT) calculations affords a unique opportunity to gain insights into this bonding. DFT calculations are invaluable for relating chemical shift (CS) and electric field gradient (EFG) tensors to molecular-level structure and bonding. Herein, we discuss the development and application of relativistic computational methods for calculating the NMR parameters for the PGEs, as well as CS tensors for light ligand atoms bonded to PGEs, and their interpretation in terms of electronic structure and bonding.

17:05-17:25 **High Precision Structures of Cellulose Polymorphs Obtained with an NMR Crystallography Approach**

Presenting Author: Darren Brouwer

Darren Brouwer (Redeemer University)

The detailed structural characterization of cellulose has presented numerous challenges due to its fibrous nature and multiplicity of crystalline forms and there remain outstanding questions, particularly concerning the hydrogen-bonding networks within and between cellulose chains. Fibre neutron and X-ray diffraction experiments have provided structures for the various forms of cellulose, however there are intrinsic limitations to the precision that can be achieved with fibre diffraction Here, it is shown that an "NMR crystallography" approach, in which SSNMR results and DFT calculations are combined, provides high precision structures of four of the polymorphs of cellulose.

17:25-17:50 **Speeding up CASE-3D with machine learning prediction of scalar couplings and chemical shielding tensors**

Presenting Author: Armando Navarro-Vázquez

Armando Navarro-Vazquez (Universidade Federal de Pernambuco); Alejandro Tanguma (Departamento de Qumica, Cinvestav); Higo de Araujo Oliveira (Departamento de Qumica Fundamental, Universidade Federal de Peranmbuco); Wildson Jose de Almeida Ramos (Departamento de Qumica Fundamental, Universidade Federal de Pernambuco); Armando Ariza-Castolo (Departamento de Qumica, Cinvestav)

The CASE-3D approach to the determination of stereochemical configuration and conformation may often require of DFT computations for prediction of chemical shielding tensors or scalar couplings. These computations may easily become a bottleneck on the computational process. We present here our machine-learning approaches to the prediction of scalar couplings, namely geminal 2JHH and 2JHC and vicinal 3JHH and 3JHC ones. Molecular representations combined purely geometry parameters with electronic parameters. The performance of our ML algorithms matched, if not improved, that of known empirical equations while having a much broader degree of applicability. Delta approaches based on cheap DFT computations for the prediction of 13C isotropic shieldings and chemical shielding anisotropies will be also presented.

ThOI: Inorganic, Organic and Hybrid Materials (parallel session)
16:00 - 17:50, Nautilus

16:00-16:25 **Cation Chaos in Photovoltaic Materials**

Presenting Author: Vladimir Michaelis

Vladimir Michaelis (University of Alberta)

A global decarbonization strategy is urgently needed in order to shift from our dependency of legacy fossil fuels to clean and reliable energy generation and storage alternatives. From a sustainability perspective the new materials will require a chemical design focused on highly abundant and inexpensive elements. Hence, to balance the chemical and optical properties with desired function requires an understanding of microscopic structure at the atomic scale. Solid-state nuclear magnetic resonance (NMR) spectroscopy is answering this call, paving the way to understanding ion substitution, doping, dynamics, and more. This contributed presentation will discuss our groups recent advances in mixed-ion photovoltaic materials containing exotic NMR-active nuclei.

THURSDAY, APRIL 20, 2023 - *continued*

16:25-16:45 **Charge Density Wave Order of the Kagome Superconductors AV3Sb5 (A=K, Rb, and Cs): Structural Studies using Single Crystal Angle-Dependent 51V NMR**

Presenting Author: Xiaoling Wang

Arneil P. Reyes (National High Magnetic Field Laboratory); Brenden Ortiz (University of California, Santa Barbara); Andrea Capa Salinas (University of California, Santa Barbara); Stephen Wilson (University of California, Santa Barbara); Xiaoling (Cocoa) Wang (California State University East Bay)

The newly discovered kagome metal family AV3Sb5 (A = K, Rb or Cs) have attracted widespread interest very recently in the field of condensed matter due to their rich physical phenomena including symmetry-breaking charge-density waves (CDWs) and superconductivity. The specific CDW order exhibits cation dependence, and the real component of CDW corresponds to a real space charge inhomogeneity and results in a superstructure deformation of the crystal lattice. For the first time, we applied 51V single crystal NMR experiments at cryogenic temperatures on the AV3Sb5 series with relative orientational dependence between the crystal coordinates and the magnetic fields, in order to investigate the structural evolution and patterns of structural deformations caused by CDW.

16:45-17:05 **Opportunities for Absolute Quantitative MAS NMR in chemical and pharmaceutical applications.**

Presenting Author: Eric Breynaert

Sambhu Radhakrishnan (NMRCoRe); Vinod C. Vinodchandran (NMRCoRe); Alysson Morais (NRMCoRe); Maarten Houlleberghs (NMRCoRe); Dirk Dom (NMRCoRe, KU Leuven); KArel Duericnkx (NRMCoRe); loes verheyden (COK-kat, KU Leuven); Eric Breynaert (NMRCoRe, KU Leuven)

Solid state NMR is largely used to characterize materials with respect to chemical composition and functionalities. NMR supersedes all other spectroscopies in determining relative amounts of individual components in complex mixtures, but absolute quantification has long remained challenge. This contribution demonstrates how standard addition combined with MASNMR enables determination of water or residual solvents in porous materials used in catalysis, adsorption or controlled release. This information is essential for catalytic or adsorption applications, to optimize production or to fulfil quality control requirements set by health and safety regulations. The method is further extended to absolute quantification of solvents in adsorption applications and to the determination of crystalline silica impurities in amorphous silica in the frame of REACH legislation.

17:05-17:25 **One- and Two- Dimensional Pure Isotropic Proton NMR Spectra in Solids using Deep Learning**

Presenting Author: Pinelopi Moutzouri

Pinelopi Moutzouri (EPFL); Manuel Cordova (EPFL); Bruno Simes De Almeida (EPFL); Daria Torodii (EPFL); Lyndon Emsley (EPFL)

We have recently suggested new approaches, relying on the combination of fast MAS and 2D correlations, to tackle the problem of resolution in 1H NMR of solids. These approaches yield pure isotropic proton (PIP) spectra that contain only isotropic shifts and provide the highest 1H NMR resolution available today in rigid solids. Here, we extend the PIP approach to a second dimension and obtain ultra-high resolution 1H-1H double-quantum / single-quantum (DQ/SQ) dipolar correlation spectra for samples of L-tyrosine hydrochloride and ampicillin. We obtain two-dimensional DQ/SQ spectra with significantly higher resolution as compared to DQ/SQ spectra acquired at 100 kHz MAS allowing the identification of resolved isotropic correlation peaks that were previously overlapped.

17:25-17:50 **Reaction mechanism of syngas conversion on bifunctional catalysts revealed by solid-state NMR spectroscopy**

Presenting Author: Guangjin Hou

Guangjin Hou (Dalian Institute of Chemical Physics)

Syngas (H2/CO) is one of the most important C1-chemistry platforms for the utilization of non-petroleum carbon resources such as natural gas, coal or shale gas. Recently, an increasing number of studies have demonstrated that the bifunctional catalyst concept of physically mixing metal oxides and zeolites (OXZEO) provides a promising alternative to go beyond the ASF limitation and tackle the selectivity challenge, but the active sites and underlying mechanism are not yet explored. Herein, we performed solid-state NMR spectroscopy to investigate the mechanism of the syngas conversion over these bifunctional catalysts, including the activation of syngas on the oxide surface, site-selective adsorption, the reaction intermediates, the first C-C bond formation, and the reaction routes in zeolite, etc.

Monterey Bay Aquarium Social Event
18:30-22:30
Adv. Purchase Ticket is Required! Ticket sales close Mon. April 17 at noon OR when sold-out. Don't be disappointed, please purchase your ticket ASAP!

POSTER TOPICS-OVERVIEW

POSTER 001
Molecular Mechanisms of Survival at Low Temperatures and Drastic Dehydration for Antarctic Lichen
Presenting Author: Aleksandra Andrzejowska

Complete Author List:
Aleksandra Andrzejowska (Jagiellonian University); Karol Kubat (Jagiellonian University); Kazimierz Strzaka (Jagiellonian University); Angelica Casanova-Katny (Catholic University of Temuco); Hubert Haraczyk (Jagiellonian University)

Lichens can survive severe desiccation and extreme cold. Therefore, they are excellent models for studying resistance to these. This study attempts to determine changes in molecular behaviors of bound water in Antarctic endemic lichen, Umbilicaria antarctica, when exposed to stresses.
The dependence of low-temperature water behavior on hydration level was investigated by 1H-NMR temperature measurements for thalli at three different hydration levels. Liquid water was observed in the thalli at temperatures as low as -17˚C. These findings were confirmed by DSC scans. Moreover, we recorded photosynthetic activity even at -12˚C.
Additionally, non-cooperative immobilization of water was discovered, suggesting the presence of supercooled water. Thanks to the BPP theory the average distance between relaxing proton pairs and activation energies were determined.

POSTER 002
SASSY NMR: SimultAneous Solid and Solution spectroscopY
Presenting Author: Rajshree Ghosh Biswas

Complete Author List:
Rajshree Ghosh Biswas (University of Toronto); Ronald Soong (University of Toronto); Amy Jenne (University of Toronto); Monica Bastawrous (University of Toronto); Myrna Simpson (University of Toronto); Andre Simpson (University of Toronto)

Traditional NMR spectroscopy has evolved as separate fields of research (solid or solution/gel) each with specialised probes, experiments, and sample preparation techniques. However, it is the synergism between all phases (solutions, gels, solids) that gives rise to environmental and biological reactivity. Traditional monitoring methods involve sequential experiments which miss information in the non-observed phase. Here, a simple, easy-to-use technique known as SASSY is introduced to simultaneously observe all phases using standard, solid-state equipment. SASSY uses alternating in-phase (IP) and anti-phase (AP) slices with a stepped decoupling regime to isolate all fractions, ranging from crystalline solids to gels and dissolved material, with each scan. This study introduces SASSY to monitor processes and observe complex samples such as a living freshwater shrimp.

POSTER 003
13C-13C J-Coupling Measurements in the Sup35p Segment GNNQQNY Using Solid State NMR
Presenting Author: Edward P. Saliba

Complete Author List:
Edward Saliba (MIT); Robert G. Griffin (MIT)

We present 13C-13C J-coupling measurements in the Sup35p segment GNNQQNY using the in-phase anti-phase (IPAP) pulse sequence developed by Cadars, et al. The doubly selective nature of IPAP allows nearly all of GNNQQNY's J-couplings to be measured in pseudo-2D experiments. The knowledge of accurate homonuclear J-couplings is necessary when fitting TEDOR buildup data, as evolution of the carbon magnetization takes place under both the dipolar couplings to the 15N spins, and the homonuclear J-couplings to other 13C spins. We use this information to fit representative TEDOR buildup curves in GNNQQNY. Furthermore, we use this information to illustrate the ability of a frequency selective J-based double quantum filter to produce high resolution 1D spectra of peptide samples.

POSTER 004
Studying cell interfaces with solid-state NMR
Presenting Author: Thomas Kress

Complete Author List:
Thomas Kress (University of Cambridge); Astrid Berge (University of Cambridge); Marie Juramy (University of Cambridge); Melinda Duer (University of Cambridge)

In this work, we present improved Goldman-Shen experiments to study cell interfaces between bovine vascular smooth muscle cells (VSMCs) and their surrounding 13C-enriched extra-cellular matrix. These experiments rely on relaxation filters and proton spin diffusion that can transport proton magnetisation up to 10 nm away from cell membranes which enabled us to record interface-edited CP (1D) and PDSD (2D) spectra.

POSTER 005
Drug Binding and Oligomeric Structure of the SARS-CoV-2 Envelope Protein Studied by Solid-State NMR
Presenting Author: Noah H Somberg

Complete Author List:
Noah Somberg (MIT); Joao Medeiros-Silva (MIT); Westley W. Wu (MIT); Hyunil Jo (University of California); Jun Wang (University of Arizona); William F. DeGrado (University of California); Mei Hong (MIT)

As of January 2023, COVID-19 has caused over six million deaths worldwide. SARS-CoV-2, the causative virus of the global pandemic, has four structural proteins. Among these, the envelope protein E is responsible for the acute respiratory symptoms of the disease. E is therefore a potential antiviral drug target. Structural information about E remains scarce, and no approved drugs targeting E currently exist. Using the 19F CODEX technique, we determine that E forms a homopentamer, and under certain conditions these pentamers cluster in the membrane. The drug binding of the channel blocker hexamethelene amiloride (HMA) is probed using 19F spin diffusion, 1H{19F} and 13C{19F} REDOR experiments, which together reveal complex dynamics of HMA in the lipid membrane.

POSTER 006
Solid-state NMR Exchange Spectroscopy: Characterizing Site-Specific Water Exchange in Biological Systems
Presenting Author: Riqiang Fu

Complete Author List:
Riqiang Fu (National High Magnetic Field Lab); Rongfu Zhang (Florida State University); Timothy A. Cross (National High Magnetic Field Lab); Shenlin Wang (Peking University)

Understanding water dynamics and structure is an important topic in biological systems, as many essential biological processes take place with the aid of water. In this presentation, we extend our previously reported one-dimensional (1D) method for water-protein 1H chemical exchange measurements via indirect detection into a two-dimensional (2D) scheme, allowing for the probing of site-specific water exchangeable sites in the biological systems. Since those sites that are not in exchange with water are largely suppressed in the resulting 2D spectra, the characterization of the site-specific water exchange dynamics becomes possible in such much simplified spectra. Here we demonstrate the feasibility of using this method for studying water exchange dynamics in biological systems (e.g., dynamics of RNA base pairs).

POSTER 007
Elucidating the hydration effect on structure and dynamics of hyaluronic acid in extracellular matrix hydrogels studied by solid state NMR
Presenting Author: Pushpa

Complete Author List:
Pushpa Rampratap (University of Groningen); Alessia Lasorsa (University of Groningen); Marthe Walvoort (University of Groningen); Patrick Van der Wel (University of Groningen)

Hyaluronic acid (HA) is a highly abundant natural polysaccharide and a fundamental component of the extracellular matrix (ECM). The macromolecular size of the HA polymer regulates tissues macro- and micro-environments, and its up-regulation is a hallmark feature of certain tumors. Probing the molecular structure and dynamics of high molecular weight (HMW) polysaccharides in a physiological-like environment is crucial but also technically challenging. Thus, isotopically enriched HMWHA was produced enabling systematic investigations by multidimensional ssNMR spectroscopy. We identify different conformations and dynamics in HA polymer, as a function of hydration level and site-specific changes when HA is interacting with ECM. The developed methods apply similarly to further studies of HA-based hydrogels and biomaterials, in wide use in pharmaceutical and cosmetic industries.

POSTER 008
Chirp Mixing Schemes for Broadband Homonuclear Correlations
Presenting Author: Sungsool Wi

Complete Author List:
Neeraj Sinha (Centre of Biomedical Research, SGPGIMS Campus); Tuo Wang (Michigan State University); Frederic Mentink-Vigier (National High Magnetic Field Laboratory, Florida State University); Sungsool Wi (National High Magnetic Field Laboratory/FSU); Lucio Frydman (Weizmann Institute of Science)

A chirp pulse mixing scheme, Adiabatic Linearly FREquency Swept reCOupling (AL FRESCO) method, was employed to establish two-dimensional (2D) homonuclear dipolar 13C-13C, 15N-15N, and 1H-1H correlations. This scheme uses a remarkably low rf power even under a ultrafast magic-angle spinning (MAS) rate by employing a single or a series of weak frequency-chirped pluses on the nuclei that constitute heteronuclear dipolar couplings with those homonuclear dipolar pairs under consideration for recoupling. Key considerations required for optimizing these mixing schemes are discussed and experimental results were demonstrated on uniformly 13C,15N-labeled protein samples. Also discussed were 2D homonuclear 13C-13C correlations demonstrated on natural abundant 13C samples by incorporating a double-quantum filter while utilizing the signal enhancement effect from the dynamic nuclear polarization (DNP).

POSTER 009
Simultaneous Recoupling of Chemical Shift Tensors of Two Nuclei by R-Symmetry Sequences
Presenting Author: Gal Porat-Dahlerbruch

Complete Author List:
Gal Porat-Dahlerbruch (University of Delaware); Tatyana Polenova (University of Delaware)

Chemical shift tensors (CSTs) are sensitive probes of structure and dynamics. R-symmetry pulse sequences (RNCSA) can efficiently recouple CSTs in MAS NMR experiments, for a broad range of conditions and MAS frequencies. We introduce dual-channel R-symmetry (DORNE-CSA) pulse sequences for simultaneously recording CSTs of two different nuclei, in a single experiment. We demonstrate DORNE-CSA performance for simultaneous measurement of 13C and 15N CSTs, on a U-13C,15N-labeled microcrystalline L-histidine. We show that the DORNE-CSA method is robust, provides accurate CST parameters, and takes only half of the measurement time compared to a pair of RNCSA experiments otherwise required for recording the CSTs of individual nuclei. DORNE-CSA approach is broadly applicable to a wide range of biological and inorganic systems.

POSTER 010
Characterization of TDP43 C-terminal Disease-Associated Aggregates with MAS Solid-state NMR
Presenting Author: Blake Fonda

Complete Author List:
Blake Fonda (UC Davis); Dylan T. Murray (UC Davis)

TDP43 is a human transcription factor protein whose pathological aggregation is associated with neurodegenerative disorders, including ALS. Previous characterization by other researchers on ex-vivo ALS-patient derived fibrils with cryo-electron microscopy yielded a characterization of the fibril rigid core region. To better understand TDP43 fibril structural variants as well as the behavior of the rest of the pathologically associated TDP43 C-terminal domain, in-vitro formed C-terminal fibrils are generated and studied with ssNMR. CP and INEPT based MAS ssNMR allows for a structural comparison versus the previously identified ex-vivo ALS TDP43 fibrils. A major goal of this work is to better understand TDP43 structural variant selection as a result of formation conditions such as phoshomimetic substitution.

POSTER 012
Monitoring Lignification in Model Plant Arabidopsis thaliana by Solid-State NMR
Presenting Author: Wancheng Zhao

Complete Author List:
Wancheng Zhao (Michigan State University); Fabien Deligey (Michigan State University); Sarah A. Pfaff (Pennsylvania State University); Daniel J. Cosgrove (Pennsylvania State University); Tuo Wang (Michigan State University)

Plant cell walls formed by complex carbohydrates and lignin constitute the majority of lignocellulosic biomass and serve as an inexhaustible resource of biomaterials and biofuel. Lignin is a polyphenolic biopolymer deposited during secondary cell wall formation, which provides strength, assists water transporting, and resists microbial attacks. The mechanism of lignification is only partly understood. Here, we employ solid-state NMR to monitor the lignification process in the model plant Arabidopsis thaliana. Our results showed that lignin deposition is fully completed for the mature stems above 20 cm height, and the lignification process relates to the position of the stem. Molecular insight is obtained on the formation of lignin-carbohydrate interface. These findings are beneficial to the rational design of efficient biomass-conversion pathways.

POSTER 013
Characterization of Kindlin-2 Binding to Phosphatidylinositol Phosphates
Presenting Author: Andrew Nieuwkoop

Complete Author List:
Tom Osborn Popp (Rutgers University); Ashley Bernstein (Rutgers University); Robert D. Palmere (Rutgers University); Insha Chhabra (Rutgers University); Zainab O. Mustapha (Rutgers University); Andrew Nieuwkoop (Rutgers University)

Kindlin-2 (K2) is a peripheral membrane protein regulated in part through its binding to phosphatidylinositol phosphates (PIPs). The Nieuwkoop lab works to understand the PIP binding sites of K2 PIP binding domains and characterize the effects of PIP binding on the structure and dynamics of K2. We use solution and solid-state NMR to investigate the binding sites of the F0 and PH PIP binding domains of K2. 1H detected solid-state NMR experiments at 100+ kHz MAS pair with solution NMR titrations with IP4 and PIP liposomes and solid-state experiments on PIPs in lipid bilayers. 1H, 13C, 15N and 31P-detected experiments acquired at static, 15, and 40 kHz MAS are also used to fully characterize the K2-PIP binding event.

POSTER 014
Comparing the Cell Wall of Aspergillus Species and Investigating Their Responses to Antifungals
Presenting Author: Isha Gautam

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Isha Gautam (Michigan State University); Malitha Widanage (Louisiana State University); Frederic Mentink-Vigier (National High Magnetic Field Laboratory, Florida State University); Ping Wang (Louisiana State University); Tuo Wang (Michigan State University)

The fungal cell wall contains polysaccharides that are absent in humans, making it a promising target for antifungal agents. Here we compare the cell wall of two opportunistic pathogens Aspergillus fumigatus and Aspergillus nidulans using solid-state NMR and Dynamic Nuclear Polarization (DNP). Both fungal species showed similar composition in the rigid portion of the cell wall but A. nidulans have a higher percentage of galactosaminogalactan in the mobile shell. When treated with the antifungal drug caspofungin, A. fumigatus exhibited higher hydrophobicity with increased chitin content to compensate for the loss of β-glucan caused by drug inhibition. These findings will provide a foundation for discovering promising antifungal drugs.

POSTER 015
Unique Structural Features of Covid-19-Associated Rhizopus Species Revealed by Solid-State NMR
Presenting Author: Qinghui Cheng

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Recently the covid-19-associated mucormycosis caused by fungi such as Rhizopus species has emerged globally as a challenge to our health system, and it leads to a severe increase in mortality rate during the pandemic. Here we employed 2D 13C-13C/15N correlation solid-state NMR to characterize the dynamics and structure of cell-wall polysaccharides in the Rhizopus delemar. Data on the dynamics, water-contact, and intermolecular contacts suggest a framework formed by rigid chitin and semi-rigid chitosan. However, the content of mobile β-1,3-glucan, the major target of almost all wall-targeting antifungal drugs, became very minor. This unique structural feature requires and assists the development of novel antifungal agents targeting other carbohydrate components in the cell wall to combat against these fungal infections.

POSTER 016
Cryo-stopped flow methodology for the investigation of prenucleation species: first steps towards time-resolved solid-state NMR
Presenting Author: Ieva Goldberga

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In this work, we show the potential of the cryo-stop flow methodology to follow the formation of calcium phosphate ' prenucleation species' (PNS) at a different time in a biomineralization context. This cryo-stop flow methodology enables the addition and mixing of independent Ca2+ and phosphate solutions at controlled concentrations, volumes and rates. Then The subsequent freezing at defined time points enables to stop the precipitation reaction and analyze transient PNS by low-temperature solid-state NMR in order to follow their formation step-by-step. Here, we describe the set-up and condition optimization to study these PNS at the early stages of hydroxyapatite (Ca10(PO4)6(OH)2) formation, the main component of bones and teeth.

POSTER 017
Combined Effects of Calcium and Phosphatidylserine on the Dynamics of PIP3-Containing Lipid Bilayers
Presenting Author: Ashley D Bernstein

Complete Author List:
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Phosphatidylinositol phosphates (PIPs) play a large role in cellular processes by binding with extreme specificity to partner proteins. Regions of the cytosolic side of the human cellular membrane have elevated levels of phosphatidylinositol triphosphate (PIP3) and phosphatidylserine (PS), both of which are anionic. MAS ssNMR was used to study calcium titrations of PIP3-containing liposomes with and without PS. The presence of calcium has a significant effect on the 31P and 1H chemical shifts, T1 and T2 of the PIP3-containing lipid bilayers. We attribute these differences in relaxation to changes in the dynamics and observe distinct results for each phosphate species present. The effect of a certain concentration of calcium on these phosphates is altered in the presence of PS.

POSTER 018
19F DNP-Enhanced Fast (25-40 kHz) MAS NMR of Protein/RNA Complexes
Presenting Author: Kumar Tekwani Movellan

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We present the first demonstration of 19F DNP combined with fast (25-40 kHz) MAS frequencies for studies of protein/RNA assemblies. We report remarkably high, up to 128-fold signal enhancements in DNP MAS NMR spectra of nucleocapsid protein (NP) assemblies with RNA fragments, both containing fluorine labels. We discuss the resolution gains and the overall benefits of high MAS frequencies. Our work establishes the power of 19F DNP-enhanced fast MAS NMR spectroscopy for structural characterization of biological assemblies.

POSTER 019
DNP NMR Assisted 'In-Cell' Structural Characterization of Mitochondria localized α-Synuclein Toxic Conformers
Presenting Author: Swapna Bera

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Swapna Bera (Postdoctoral researcher)

The role of α-Synuclein, a major hallmark of Parkinson's disease (PD), in modulating mitochondrial function in both physiological and pathological conditions has long been a topic of intense debate. Multiple studies reported on mitochondrial dysfunction and oxidative stress by α-Syn's aggregation. Unfortunately, the dynamically interchanging heterogenous conformations of α-Syn make the in-cell structural studies very inflexible. With our novel development of a cellular systems platform for in-vivo structural biology (JACS.2021,143,44,18454–18466) I am investigating mitochondria localized α-Syn structure inside mammalian cells and reveal how cellular environments influence protein's amyloid core structure. Collectively, this structural information in live cells will directly link cellular toxicity to protein conformation and will transform our mechanistic understanding of protein misfolding and mitochondrial impairment in PD etiology.

POSTER 020
Solid-state NMR with a CPMAS CryoProbe enables structural studies of human blood protein vitronectin bound to hydroxyapatite
Presenting Author: Tata Gopinath

Complete Author List:
Tata Gopinath (Medical College of Wisconsin)

The low sensitivity of solid-state NMR is a major bottle neck for studying protein samples under dilute conditions, such as, membrane proteins and protein-ligand complexes. Cryogenically cooled probe technology overcomes sensitivity limitations enabling solid-state NMR applications to challenging biomolecular systems. Here we present solid-state NMR data acquired with a CP-MAS CryoProbe for the human blood protein vitronectin bound to hydroxyapatite. Vitronectin is a major blood protein that regulates many different physiological and pathological processes. The high sensitivity of the CP-MAS CryoProbe enabled us to acquire three-dimensional solid-state NMR spectra for sequential assignment of vitronectin bound to hydroxyapatite. We also demonstrate residue specific water-protein interactions that provide structural insights into water coordination in the protein complex.

POSTER 021
Measuring the Fluidity of Deep Sea Hydrothermal Vent Bacterial Membranes at High Pressure with Solid State NMR
Presenting Author: Thomas Osborn Popp

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Thermopiezophiles are microorganisms that grow at high pressures and temperatures (10 - 100 MPa, 50-100 °C), and are typically isolated from deep-sea hydrothermal vents. Currently, little is known about the chemical and biophysical properties of these organisms, due to the technological challenges associated with observing them under conditions that mimic their natural habitat. Here we employ solid state NMR under both static and magic angle spinning (MAS) conditions to study thermopiezophile phospholipid membranes as a function of both pressure and temperature. Our initial results suggest that these organisms modify the lipid composition of their membranes in response to growth pressure in order to maintain a well-defined membrane fluidity.

POSTER 022
Solid-State NMR Investigation of Molecular Attributes in Frozen Solution
Presenting Author: Yong Du

Complete Author List:
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Investigating microenvironmental properties and the underlying physiochemical mechanism in frozen solutions is a critical but underexplored task. For example, the physical environment of biologics changes dramatically during freezing which may lead to severe stability issues. However, it is technically challenging to probe the local molecular properties (e.g., phase separation, pH, ionic strength, protein structure and dynamics) in an in situ, high-resolution, and quantitative manner. Our studies aim to explore these molecular attributes in frozen solutions using solid-state NMR spectroscopy.

POSTER 023
Combined use of Solid-State and Solution NMR to Understand Allosteric Transitions in a Ligand-Activated Oligomeric Protein
Presenting Author: Rodrigo Muzquiz

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TRAP is an allosterically regulated protein that is activated by tryptophan (Trp) to bind RNA and terminate tryptophan biosynthesis in Bacilli. We performed solution and solid-state NMR experiments on TRAP to understand how binding of Trp to its 11 identical sites modulates its RNA binding function. We performed methyl CPMG relaxation dispersion experiments in solution on Thr/Ile methyl labeled TRAP in the absence and presence of Trp. These experiments showed strong dispersions indicative of µs-ms time scale exchange in apo-TRAP that are diminished in holo-TRAP. Complementary solid-state INEPT experiments identified residues invisible in solution and CP experiments. These experiments allow us to characterize the structural and dynamic landscape of apo-TRAP and new insights into mechanisms of its regulation by Trp.

POSTER 024
Exploring binding site of MODAG-005 on α-Synuclein aggregates as a novel PET tracer
Presenting Author: Myeongkyu Kim

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Early detection of a-synuclein (αSYN) aggregates and observing the pathological process has been a challenge. Positron emission tomography (PET) is a non-invasive in vivo imaging technique useful for early diagnosis of aggregates in the human brain. However, a target-specific tracer to detect pathological aggregates of αSYN is missing. Here, we report the development of anle170322 (PET tracer) based on anle138b, a compound shown to have therapeutic activity in animal models of neurodegenerative diseases. The structure of the fibril was determined by cryo-EM and the binding sites (internal and external) of anle170322 were identified through solid-state NMR spectroscopy and dynamic nuclear polarization (DNP). Interestingly, two binding sites are found depending on the preparation protocol.

POSTER 025
CPMAS NMR Platform for Direct Compositional Analysis of Mycobacterial Cell Wall Complexes and Whole Cells
Presenting Author: Xinyu Liu

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Mycobacteria cause chronic incurable infections and are alarmingly resistant to currently available antibiotics that target cell-wall biosynthesis. Resistance is attributed to assumed differences in cell-wall composition across species, which is challenging to analyze with conventional biochemical methods. We introduce an approach to directly observe chemical composition of mycobacterial cell walls using solid-state NMR. By obtaining 13C CPMAS spectra of cell-wall components, we uncovered a higher arabinogalactan-to-peptidoglycan ratio in M. abscessus, which is noted for its antibiotic resistance, relative to a basic model strain M. smegmatis. Differentiating influences of cell-wall targeting antibiotics were observed in spectra of treated whole cells. Our platform will be valuable in evaluating cell-wall composition and antibiotic activity among different mycobacteria and guiding effective combination treatment regimens.

POSTER 026
Analyzing Biopolymer Motion via Rotating Frame Relaxation in MAS NMR of Large Anisotropy 13C Sites
Presenting Author: Eric G. Keeler

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The study of biomolecular motions by magic angle spinning NMR rotating frame relaxation measurements has been demonstrated to yield fruitful results. Focusing on systems where modulation of the chemical shift interaction is the driving force for the mechanism of relaxation we demonstrate a strategic approach to extract motional time scales, activation energies, and the order parameters from rotating frame relaxation data. We use both model free analysis and numerical simulations, including treatment of the relaxation data in the time domain, to demonstrate the optimal conditions for obtaining precise and accurate time scales of motions. Furthermore, we extend this exercise to the model ion channel KcsA to provide an example.

POSTER 027
Discovery and Characterization of Natural Modified Polysaccharides in Bacteria Biofilms Using SSNMR.
Presenting Author: Alexandre Poulhazan

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Cellulose is the most abundant biopolymer on Earth and solid-state NMR was uniquely enabling in the discovery of a modified cellulose produced by E. coli– phosphoethanolamine cellulose. Together with curli amyloid fibers, these polymers form remarkable basket-like structures surrounding E. coli. In exploring the production of celluloses in E. coli strains, we discovered another polysaccharide produced in high abundance. This discovery was enabled by our use of genetic modifications to suppress the co-production of curli and enable isolation and characterization of the polysaccharide. I will present our characterization by solid-state and solution-state NMR of this potentially as-of-yet unidentified but with potential relevance in infection polysaccharide. We are expanding our discovery platform to seek out new chemistry and alternately modified polysaccharides.

POSTER 028
Structural Analysis of Membrane-mediated Amyloid Aggregates of human islet amyloid polypeptide (hIAPP)
Presenting Author: Venus Singh Mithu

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Evidence suggests that the interaction between hIAPP and phospholipid membrane is pivotal in causing β cell failure, a hallmark of Type II diabetes mellitus (T2DM) pathology. Thus, structural characterization of this interaction is essential for designing therapeutic interventions. We have investigated the structural fold of full-length recombinant hIAPP fibrils grown in the presence of small unilamellar vesicles (SUVs) composed of zwitterionic POPC and negatively charged POPS phospholipids. Sequence-specific assignment of hIAPP was achieved using 1H-detected correlation spectroscopy at 55 kHz magic-angle-spinning (MAS) and 1H-mediated 13C-13C through-space correlation spectroscopy under slow MAS conditions. Chemical shift-based dihedral angle predictions and long-range inter-residue contacts will be used in conjunction with Cryo-EM-based investigations to obtain the atomic structure of membrane-mediated fibrils.

POSTER 029
New Insights into the Influence of β-lactam Antibiotics against S. aureus by Solid-State NMR Spectroscopy
Presenting Author: Till Kallem

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Till Kallem (Stanford University)

The mechanism of killing by cell-wall-targeting β-lactam antibiotics against the Gram-positive pathogen S. aureus remains poorly understood. This is partially due to shortcomings in conventional biochemical methods for studying the highly crosslinked S. aureus cell wall. Furthermore, β-lactams can induce an unexplained paradoxical killing trend against S. aureus such that lower doses outperform higher doses (Eagle Effect). We leveraged 13C and 15N CPMAS solid-state NMR analysis of whole cell and cell wall samples to reveal different compositional changes under low and high doses of meropenem against a methicillin susceptible strain, S. aureus 29213. Our results indicate that meropenem induces two distinct lethal pathways, depending on dosage, that correlate with differential killing kinetics and underpin the Eagle Effect in this system.

POSTER 030
NMR Crystallography of Toho-1 β-Lactamase Enabled by Nearly Complete Backbone and Sidechain Assignments
Presenting Author: Christopher Williams

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Nearly complete backbone and sidechain chemical shifts are reported for a microcrystalline sample of the 28 kDa Toho-1 β-Lactamase: using only two 2D and three 3D experiments at 900 MHz, 96% of the backbone and 80% of the nonaromatic sidechains can be assigned, including the mechanistically important active site sidechain residues. These chemical shifts allow preliminary refinement of the structure and protonation states of the active site using NMR crystallography – the integrative combination of solid-state NMR with diffraction and first principles computational chemistry.

POSTER 031
Study of phosphorus clusters in tau fragment fibrils via multiple quantum solid-state NMR under DNP
Presenting Author: Lokeswara Rao Potnuru

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Multiple quantum spin counting (MQ-SC) is a technique that can identify the formation of clusters by measuring the multi-quantum coherence orders between the coupled spins. MQ-SC has been applied to various glass and solid materials at varying magic angle spinning (MAS) rates. In the present study, we show the utility of MQ-SC on the vitrified solution samples of phosphate-containing species of amorphous calcium phosphate (ACP), and crystalline hydroxyapatite (HAp) and tau fragment fibrils at 100 K under DNP conditions. 31P MQ-SC experiments were carried out by using the SR218 pulse sequence to create multiple even and odd MQCOs at 10 kHz MAS frequency to find out the cluster sizes and clustering of phosphate species.

POSTER 032
Solid-state NMR analyses of Glycera worm jaws and biomimetic analogs
Presenting Author: Arun Kumar Patel

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Glycera (bloodworm) jaws are comprised almost entirely of organic matter; though exhibit robust mechanical properties that are similar to those of inorganic solids. Glycera jaws are unique in that they are composed predominantly of melanin (40–50 wt%), which is typically a soft material and not associated with load-bearing properties. Our objective is to understand the atomic-level origins of this unusual natural material and compare its composition and structure with that of synthetic analogs. Here, we report solid-state two-dimensional (2D) 13C{1H} correlation NMR, 2D 1H{1H} single quantum-double quantum (SQ-DQ), and dynamic-nuclear-polarization (DNP)-enhanced 15N NMR analyses that enable complicated compositional similarities and differences to be established between Glycera worm jaws and polydopamine and which correlate with their respective mechanical properties.

BIOMOLECULAR SOLUTION NMR (Posters 033 – 113)

POSTER 033
NMR Structural Studies of an Insect Cytokine: Manduca sexta Stress Responsive Peptide-3
Presenting Author: Andy Su

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Similar to innate immunity in vertebrates, insects rely on both humoral and cellular responses to defend them from pathogen invasion. Recently, a family of peptides has been identified in many insects including mosquitoes, which may function as insect cytokines to regulate immune responses. Our previous structural studies suggest that these peptides adopt a fold similar to C-terminal sub-domain of EGF, and they may interact with EGFR-like molecules. Manduca sexta stress responsive peptide-3 (SRP3) is predicted to be a 27-residue peptide (FLIKSSGCPKGYVKRGTFCFPDEDYDY), stabilized with a disulfide bond. We have initiated structural/functional and dynamics studies on SRP-3 using homo and hetero-nuclear multi-dimensional NMR spectroscopy to obtain experimental data that will aid in the development of potential EGFR inhibitors useful in cancer treatment.

POSTER 034
Structure Elucidation of an Invisible Excited State with Allosteric Relevance in a KRAS Oncogenic Mutant Using RDCs
Presenting Author: Gabriel Cornilescu

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Localized dynamics of RAS, including regions distal to the nucleotide binding site modulates the interaction with effectors and regulators. Among several oncogenic mutants, methyl relaxation dispersion experiments revealed highly synchronized conformational dynamics in the active KRAS-G13D, indicative of exchange between two conformational states in solution. Methyl and 31P NMR spectra of active KRAS-G13D in solution also indicate a two-state ensemble, with peaks corresponding to the State 1 conformation and to an intermediate state, different from the known State 2 conformation recognized by RAS effectors. We used residual dipolar couplings to solve and cross-validate the structure of the intermediate state, which shows regions of structural fluctuations with functional relevance and potential for drug discovery.

POSTER 035
Structure of Calmodulin Bound to Two Different Functional Sites in the Retinal Cyclic Nucleotide-Gated Channel Revealed by NMR Spectroscopy
Presenting Author: Aritra Bej

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Retinal cyclic nucleotide-gated (CNG) channels (composed of three CNGA1 and one CNGB1 subunits) exhibit a Ca2+-dependent inactivation mediated by calmodulin (CaM). Defects in the Ca2+-dependent regulation of CNG channels may be linked to retinitis pigmentosa and color blindness. Here, we reported the binding analysis and NMR structures of CaM bound to two distinct cytosolic sites within rod CNGB1 called CaM1 (residues 565-587) and CaM2 (residues 1120-1147). The binding studies revealed that CaM1 prefers binding to Ca2+-bound CaM N-lobe (residues 1-79) whereas CaM2 binds to Ca2+-bound CaM C-lobe (residues 80-149) with higher affinity. We solved separate NMR structures of Ca2+-saturated CaM bound to CaM1 and CaM2, identified the key intermolecular contacts, and proposed a Ca2+-dependent conformational switch in the CNG channel.

POSTER 036
NMR study on the interaction of human ZBP1 with its target Z-DNA
Presenting Author: Youyeon Go

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youyeon Go (Gyeongsang national university); Joon-Hwa Lee (Gyeongsang national university)

ZBP1 functions as a central regulator of cell death and inflammatory responses. ZBP1 is capable of sensing Z-form RNAs produced during IAV infection, cumulating in a form of caspase-independent, inflammatory cell death. ZBP1 interaction with DNA, as well as viral and endogenous RNA via its N-terminal Z-DNA-binding domains of ZBP1, has been reported. The Zα1ZBP1 and Zα2ZBP1 structure are similar to other Z-DNA binding proteins, although it demonstrates an unusual Z-DNA recognition. In this study, we performed HSQC experiments on complexes of hZα1ZBP1 and hZα2ZBP1 with d(CG)3 duplex r(CG)3 duplex at various DNA-to-protein molar ratios. The results from previous studies can produce valuable insights into the distinct molecular mechanism of the DNA duplex B–Z transition induced by hZα1ZBP1 and hZα2ZBP1.

POSTER 037
Structure Characterization of the Lipid Nanoparticle Surface in an mRNA Vaccine using High Field NMR Spectroscopy
Presenting Author: Maple Wang

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Lipid nanoparticles (LNPs) have been successfully used as a carrier for messenger RNA (mRNA) vaccines. The surface properties of LNPs are important to the stability and function of mRNA vaccines. Polyethylene glycol (PEG) is a functional lipid at the surface of LNPs that improves colloidal stability, increases circulation time, and inhibits cellular uptake. We explore the lipid composition at the surface of the LNPs using high-field nuclear magnetic resonance (NMR) spectroscopy. Our results demonstrate that NMR can detect and resolve PEG chains on the surface of intact LNP and provide quantification of PEG and other lipid components. Comparative NMR analysis of different vaccine preparations and stability samples provides a global view of the mRNA-LNP surface structure for enhanced product understanding.

POSTER 038
Intermediate-state-trapped Mutants Unravel a Sequential Conformational Allostery of G Protein-coupled Receptor
Presenting Author: Libin Ye

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Please see the attachment.

POSTER 039
A 13C Direct-Detect Nuclear Magnetic Resonance Method to Investigate Lysine Acetylation
Presenting Author: Olivia Fraser

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Lysine N-acetylation is a ubiquitous post translational modification (PTM) that affects proteins involved in a wide range of cellular processes. Despite this, the molecular mechanisms by which acetyl marks are installed, affect protein function, and are removed, are not well characterized in comparison with other PTMs. This is in part due to limitations of current methods, which can require exogenous tags or acetyllysine mimics. We demonstrate the ease and utility of a novel 13C direct-detect method to observe acetyllysine using histone H3 tail acetylation as a model. This method does not require chemical modifications that could alter the modified lysine's properties or interfere with downstream biochemical processes, making it suitable for use in systems where changes beyond acetylation are unacceptable.

POSTER 040
Ranking mAb-excipient Interactions in Biologics Formulations by NMR Spectroscopy and Computational Approaches
Presenting Author: Chunting Zhang

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Excipients are added to biopharmaceutical formulations for enhancing protein stability and developing robust formulations, but the mechanism by which they confer stability remains not fully understood. Our study aimed at using STD-NMR to provide direct experimental evidence to excipient-mAb binding affinity, and therefore ranking a series of excipients with respect to their dissociation constant and non-specific binding constants. Molecular dynamic and Monte Carlo simulations were done in parallel, to rank the excipient proximity to the proteins and thereby corroborating the ranking. Finally, the excipient ranking was correlated to mAb conformational and colloidal stability, therefore aiding with excipient selection in biologic formulations, by providing insights into mAb-excipient affinities before conducting a conventional excipient screening study which is time-consuming.

POSTER 041
Calcium dissociation and functional unfolding: A discovery by NMR for a 1.1 mDa component of the C. difficile binary toxin
Presenting Author: Spiridon Sevdalis

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Clostridium difficile infection (CDI) is challenging because treatment options are limited, and high recurrence rates occur in hypervirulent strains. The C. difficile toxin (CDT) has a toxic enzymatic component, termed CDTa, and a pore-forming or delivery subunit termed CDTb. A novel mechanism for CDT delivery is unlike that observed for any other members of the AB toxin family (i.e., anthrax). Specifically, CDTb was shown to be regulated by a Ca2+-dependent unfolding event that is consistent with CDT entry into endosomes. Thus, lowering of free Ca2+ concentration upon entry induces conformational exchange in its receptor binding domain1 (RBD1) to "trigger" protein dynamic features throughout CDTb that allows for delivery of toxic CDTa into the host cell cytoplasm.

POSTER 042
New cross-polarization schemes for heteronuclear transfers involving labile protons in biomolecular NMR
Presenting Author: Jihyun Kim

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INEPT-based experiments are widely used for $^{1}H\rightarrow^{15}N$ transfers but often fail when involving labile protons due to solvent exchanges. J-based cross-polarization (CP) offers a more efficient alternative to perform such transfers, particularly when leveraging the additional $H^{water}\leftrightarrow H^{N}$ exchange process to boost the $^{1}H\rightarrow^{15}N$ transfer. This demands spin-locking both $^{1}H^{water}$ and $^{1}H^{N}$ by a strong ^{1}H RF field, while fulfilling the $\gamma_H B_{1,H}=\gamma_N B_{1,N}$ Hartmann-Hahn condition. Given the low value of γ_N/γ_H, these demands cannot be simultaneously achieved by the power-limited cryogenic probes used in high-field contemporary NMR. Here we introduce two CP alternatives that can alleviate this limitation and demonstrate their performance on double and triple resonance transfer experiments on amino acids and intrinsically disordered proteins, that confirm theoretical expectations.

POSTER 043
Watson-Crick-like Tautomeric and Anionic G•T/U Conformational States in RNA-DNA Hybrids: Context Dependence and Implications for Transcriptional Errors
Presenting Author: Or Szekely

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G-T/U wobble mismatches in B-DNA and A-RNA can form low-abundance Watson-Crick-like conformations through tautomerization or ionization, having implications in replication and translation errors. Little is known about propensities to form WC-like conformations in RNA-DNA hybrids and their roles in transcriptional errors. Using NMR R1ρ relaxation dispersion we show that hybrid dG-rU and dT-rG wobble mismatches exist in equilibrium with low-populated short-lived tautomeric and anionic WC-like conformations. Tautomeric exchange-kinetics were very similar to those reported previously, while anionic population and forward rate-constant were uncharacteristically ten-fold higher for dG-rU versus rG-dT. Reexamination of prior measurements revealed large variations in anionic apparent pKa, with a strong preference to ionize in cytosine-rich sequence contexts. Our study indicates plausible roles for WC-like conformations in transcriptional errors and indicates that mutagenic dynamics strongly depend on nucleic acid sequence and structural contexts.

POSTER 044
Quantitative analysis of sterol modulated monomer-dimer equilibrium of β1-adrenergic receptor by DEER spectroscopy
Presenting Author: Nina Kubatova

Complete Author List:
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GCPRs play a vital role in intracellular signaling pathways and control various physiological processes in eukaryotes. The oligomerization properties of GPCRs, and hence their cellular functions, may be modulated by various components within the cell membrane. Using DEER spectroscopy, we demonstrate different effects of soluble cholesterol analog CHS and cholesterol derivative bile salt sodium cholate on the oligomerization propensities of β1-adrenergic receptor (β1AR) in DDM micelles. Global fitting of DEER echo curves for spin-labeled β1AR upon titration with sodium cholate and CHS demonstrates that saturation of micelles with the former induces receptor dimerization, while specific binding of the latter to β1AR inhibits dimerization and stabilizes the monomeric form.

POSTER 045
Structural Study of the NS2B Membrane Protein from Zika Virus in SDS Micelles by Solution NMR
Presenting Author: Beatriz Rosa Penna

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Zika virus (ZIKV) emerged as a global public health concern due to its relationship with severe neurological disorders. NS2B is a viral membrane protein responsible for regulating viral protease activity and critical for virus replication, making it an attractive antiviral drug target. This work aims to elucidate the structure and study the dynamics of the full-length ZIKV NS2B in SDS micelles through solution NMR. Despite the challenge, we propose a structure of ZIKV NS2B based on NMR experimental data, which was consistent with the molecular dynamics data and with the described in the literature. Our work will be important to improve understanding of the role of NS2B in viral replication and for prospection of inhibitors against ZIKV.

POSTER 047
Structural Dynamics Study of Thermophilic Proteins by NMR - TTHA0849 of Thermus thermophilus
Presenting Author: Karen Stephanie dos Santos

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TTHA0849 presents a PR-10 fold that forms a hydrophobic cavity. This work aims to characterize the structural dynamics of TTHA0849 in its free and bound state and compare it with the mesophilic counterparts (Bet v 1, among others) that shares the same fold and the ability to bind hydrophobic compounds. We used intrinsic fluorescence, circular dichroism, and NMR to evaluate the structure, dynamics, and interaction with hydrophobic compounds. TTHA0849 recombinant protein was obtained and purified by chromatography, and our data showed it has high thermal stability and chemical resistance. Furthermore, we have evidence of complex formation with compounds. The backbone assignment is almost complete (93 %), and relaxation parameters (R1, R2, and hetNOE) were collected in different temperatures and are being analyzed.

POSTER 048
Visualizing Proteins in Mammalian Cells by 19F NMR Spectroscopy
Presenting Author: Wenkai Zhu

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In-cell NMR spectroscopy is a powerful tool to investigate the behavior of biologically important molecules in physiologically relevant environments. We studied proteins delivered into mammalian cells by electroporation and showed that interactions with cellular components frequently broaden resonances in 1H-15N HSQC spectra beyond detection. This contrasts findings from 19F spectroscopy, where resonances for selectively fluorinated proteins are readily observed. In addition, we show that 19F paramagnetic relaxation enhancements (19F PREs) can provide valuable distance information for structure characterization in physiological contexts. The in-cell 19F PRE-derived distances are in good agreement with in-cell 19F ENDOR measurements on the same paramagnetic proteins, thus providing an effective means to obtain accurate distances in the cellular milieu.

POSTER 049
Hsp104 Chaperone Induces "Off-pathway" Oligomerization of Aβ-42 Monomers
Presenting Author: Shreya Ghosh

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Hsp104 is a unique disaggregase chaperone found in yeast, yet has been shown to function synergistically with mammalian chaperones, without displaying any overt toxicity and in turn, conferring increased stress tolerance. Additionally, Hsp104 is also the sole known chaperone to disaggregate mature amyloid fibrils. While the ability of Hsp104 to prevent and disaggregate fibrils is common knowledge, yet the pathway, mechanism, and kinetics associated with the activity still remains unanswered. To this end, I have used a combination of NMR along with imaging techniques of EM and AFM and showed that Hsp104 aids in the formation of "off-pathway" Aβ-42 oligomers that cannot materialize further into fibrils.

POSTER 050
Drug-like Small Molecules That Inhibit Expression of the Oncogenic MicroRNA-21
Presenting Author: Bhawna Chaubey

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The discovery of drug-like small molecules which bind specifically to the precursor of the oncogenic and pro-inflammatory microRNA-21 with mid-nanomolar affinity is reported. The small molecules target a local structure at the Dicer cleavage site and induce distinctive

structural changes in the RNA which correlate with specific inhibition of miRNA processing. The most potent one reduces cellular proliferation and miR-21 levels in cancer cell lines without inhibiting kinases or classical receptors. Structurally conservative single nucleotide substitutions eliminate the conformational change induced by the small molecules, which is not observed in other miRNA precursors. These molecules are highly ligand-efficient (MW330) displaying specific biochemical and cellular activity, thereby providing an avenue towards therapeutic development in multiple diseases where miR-21 is abnormally expressed.

POSTER 051
Partial Assignments of Tunicate βγ-crystallin Structure
Presenting Author: Mina Mozafari

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Tunicate Ciona intestinalis (Ci-βγ-crystallin) is a model to study evolution of vertebrates as it has an evolutionary position between the microbial crystallins and the vertebrate lens proteins. The single-domain βγ-crystallin of the tunicate Ci-βγ-crystallin represents the single-domain ancestor of the vertebrate βγ-crystallin. It has been shown that the tunicate Ci-βγ-crystallin has a high affinity to bind to Ca2+ and that this binding greatly stabilizes the protein both thermally and chemically. However, human γS-crystallin doesn't bind to Ca2+ but it stays stable. NMR spectroscopy is used to study the structure and properties of Ci-βγ-crystallin to better understand the stability and evolutionary progression from ancestral to human lens βγ-crystallins.

POSTER 052
Probing Biodegradation Mechanism and Kinetics of Stimuli-Responsive Microcapsules Utilizing 1H NMR Spectroscopy
Presenting Author: Uyen Thi Do

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In this study, the stimuli-responsive microcapsules were fabricated using gelatin, alginate, and hyaluronic acid as wall materials; additionally, enzyme-induced decomposition mechanisms were proposed by observing spectral changes in 1H NMR analyses. The synthesized microcapsules are spherical, tunable in size, with high encapsulation efficiency, and neutral pH-induced cutaneous release of hydrophobic core from microcapsules was recorded. Examination of the short-term and long-term degradation kinetics reveals that the determination of the degradation rate constant of the major components in the capsule is feasible and suggests two types of 4-stage degradation mechanisms that are enzyme-specific. These findings suggest that capsule decomposition can be thoroughly investigated using 1H NMR spectroscopy to provide a practical strategy for monitoring degradation properties in developing new biodegradable materials.

POSTER 053
An Investigation on Microbial Degradation of Biodegradable Microcapsules Using 1H-NMR for Innovative Eco-friendly Material Development
Presenting Author: Yeeun Park

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Biodegradable microcapsule based on natural polymer is considered a replacing material of microplastics without causing environmental pollution. However, in the case of measuring biodegradability of these particles, there is disadvantage that it takes quite a long time. In this study, we discuss the investigation of pre-screening that confirms the degradation of natural polymer-based microcapsules by microorganisms using 1H-NMR. Experiments confirmed that the spectrum of a sample containing microcapsules and microbes became sharp than that of only microcapsules. Also, the new peak that didn't appear in the 1H-NMR spectrum of only microcapsules and microorganisms was also observed. Through this, the possibility of biodegradation pre-screening technology that can be executed on an NMR-based lab-scale was conformed.

POSTER 054
NMR chemical shift assignments of the SCoV-2-delta element 3_s2m utilizing multidimensional NMR
Presenting Author: Tobias Matzel

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The SCoV-2 genome consist of a 30.000 nt (+) strand RNA which not only encodes for the viral proteins but also shows highly structured 5' and 3' untranslated regions. One of these regulatory RNA elements is the stem loop II motive 3_s2m, which is highly conserved between distantly related viruses although its function is still unclear.
Utilizing a 4D-HMQC-NOESY-HMQC experiment and different 13C filtered NOESY spectra chemical shifts were assigned for over 90% of the relevant NMR resonances. The 4D was used to assign NOEs between aromatic (H6/H8) and sugar H1' protons, which enabled us to assign 98% of the C6, C8 and C1' resonances. We then assigned C2' C5' sugar resonances using selective labelling in combination with 13C-filtered NOESYs.

POSTER 055
Structural and Dynamical Investigation of Small Proteins
Presenting Author: Dennis J Pyper

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Small proteins have been ignored in the past, because detection and identification of the coding DNA posed significant problems. Modern transcriptomics technologies have made it possible to identify increasingly more sORFs. They were found in all domains of life and it was shown that they have a variety of purposes. We apply NMR to obtain the structures and dynamics of small proteins in their apo state as well as with ligands. Furthermore, NMR enables us to identify the folding state of small proteins, which include folded, partially folded, molten globule or an unstructured state. As part of an academic collaboration we investigated 37 small proteins (14 - 78 amino acids). For three small proteins we calculated high-resolved solution NMR structures.

POSTER 056
Insightful 1H-NMR Studies of Physical Properties and Solubilization in Polymeric composites for Advanced Industrial Applications
Presenting Author: Jiwon Kim

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NMR spectroscopy can provide vital information on the design and formulation improvements of polymeric composites, which can be effectively used in cosmetics and medical applications. Here, industrially practical NMR approaches are introduced that analyze the fundamental reason of phospholipid solubilization by specific surfactants, and the comparison of physicochemical properties in nanoliposome according to the packing structure. As a first project, we explored that 1,2-hexandiol prevents crystallization of phospholipids by performing a 1H-NMR-based titration experiment to investigate the phenomenon that phospholipids are selectively solubilized only on 1,2-hexandiol. Subsequently, the pliability of nanoliposome with hexagonal (C16) and orthorhombic (C22) packing structures was compared by measuring T2 relaxation according to the temperature, and hexagonal packing samples were found to be more flexible.

POSTER 058
Analysis of Sidechain Dynamics using Slow-Relaxing Methyl Quadruple-Quantum Coherences
Presenting Author: Christopher A. Waudby

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We present an experiment for to measure the relaxation rates of two quadruple-quantum transitions in $^{13}CH_3$-labelled methyl groups. These coherences are protected against relaxation by intra-methyl dipolar interactions and so have unexpectedly long lifetimes within perdeuterated biomacromolecules. These coherences have high sensitivity to chemical exchange and therefore provide ideal probes of dynamic processes. We show analysis of magnetic field-dependent zero-, double- and quadruple-quantum Hahn echo relaxation rates provides a robust indication of chemical exchange and can determine relative magnitudes of proton and carbon chemical shift differences. We also report new CPMG relaxation dispersion experiments that exploit quadruple-quantum coherences to provide increased sensitivity and improved precision in parameter estimates, particularly for ^{1}H chemical shift differences.

POSTER 059
NMR Characterization of the C-terminal Domain of the Streptococcus Mutans Adhesin P1
Presenting Author: Emily-Qingqing Peng

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Streptococcus mutans is the virulent bacterium primarily responsible for dental cavities. Prior work indicates that the naturally occurring C-terminal truncation product (C123, 51kDa) of the cell surface-localized adhesin P1 plays an important role in the formation of functional amyloids. We are characterizing C123 in its soluble and amyloid forms by solution NMR and DNP-enhanced solid-state NMR, respectively. Using AlphaFold, we developed constructs for both C123 and the individual C3 domain (17.5 kD) that exhibit superior NMR resolution compared to earlier constructs used for microbiology and X-ray diffraction experiments. This enabled us to improve solution NMR assignments for comparison to our ssNMR studies of the structural transition to amyloid.

POSTER 060
Development of an NMR Method for Selective Detection of Protein Acetylation
Presenting Author: Kyungryun Lee

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Acetylation regulates protein functions involved in cellular processes including gene expression, protein-protein interactions, and enzymatic reactions. Therefore, determination of protein acetylation is important to predict protein behaviors in cells. In this work, we developed an NMR pulse sequence, Ac-FIND (Acetylation-FIltered and aNd eDited), based on the isotope filtering and editing technique to selectively detect protein acetylation. Ac-FIND was able to detect signals arising from acetylated moieties of both chemically acetylated α-synuclein and ubiquitin. Furthermore, a single signal corresponding to intracellular N-terminal acetylation was

observed by Ac-FIND when non-acetylated $^{13}C/^{15}N$ labeled α-synuclein was introduced into live HEK293 cells. The results presented here demonstrate the usefulness of NMR method for detecting specific protein modifications in vitro as well as in live cells.

POSTER 061
A Study of the PopZ Binding Partners Mechanism
Presenting Author: Logan M Brown

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Intrinsically disordered proteins (IDPs) are found in all life forms, serving essential functions throughout the cell cycle. Despite the importance of this class of protein, surprisingly little is known about how they function. One example of these IDPs is PopZ which plays an important role in the cell cycle of Caulobacter crescentus, where it is responsible for gathering essential proteins to the cell poles during cell division. Currently, there are 10 known binding partners for PopZ each with vastly different structures and functions. We aim to elucidate the mechanism for this protein-protein interaction with two of its binding partners that will enhance our understanding of how the shared hub-binding site is so promiscuous yet maintain specificity to protein binding targets.

POSTER 062
Statistical Approaches for Robust Analyses of 15N Spin Relaxation Measurements
Presenting Author: Bruce Johnson

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Inferences about the dynamics of biological macromolecules requires specification of appropriate theoretical models for NMR spin relaxation in solution. Here we describe two approaches to model selection and their integration into a user-friendly GUI based software application. We illustrate the two approaches with an application to nitrogen-15 relaxation measurements on the bZip transcription factor domain of the Saccharomyces cerevisiae protein GCN4. Nitrogen-15 relaxation data at four static magnetic fields allowed bootstrap aggregation. The first approach utilizes
bootstrap aggregation, or bagging, to mitigate the effects of model-selection error in fitting variants of the model-free formalism. The second approach utilizes regularization to select model parameters that minimize overfitting. These two approaches are integrated into our RING NMR Dynamics application.

POSTER 063
Structural characterization of an Antifreeze Protein, ApAFP752
Presenting Author: Krisztina Varga

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Antifreeze proteins are found in a wide range of cold adapted organisms, and they contribute to their freeze resistance. Antifreeze proteins adsorb to the ice surface and inhibit the growth of ice crystals. The goal of this project is to investigate the mechanism by which antifreeze proteins protect against the damage typically inflicted by the cold, including the underlying molecular mechanism of ice-binding. Here we are presenting structural and functional characterization of an antifreeze protein, ApAFP752 from the desert beetle Anatolica polita utilizing nuclear magnetic resonance (NMR) spectroscopy and other biophysical methods. Current work focuses on the application of this protein in cryopreservation protocols, and on engineering a version of the antifreeze protein with enhanced activity.

POSTER 064
Surface Accessibility of Intrinsically Disordered Protein Residues Probed by 2D Time-Resolved Photo-CIDNP Experiments
Presenting Author: Jonghyuk Im

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Surface accessibility data can provide crucial information in determining the correct conformational ensemble of intrinsically disordered proteins. Photochemically induced dynamic nuclear polarization (photo-CIDNP) takes place upon a transient interaction between a photo-excited dye and a surface-exposed aromatic residue. Herein, we made technical advancements to investigate the surface accessibility of alpha-synuclein (α-Syn) using time-resolved photo-CIDNP (TR-CIDNP). High-energy laser pulses were transferred to NMR samples via an optical fiber by implementing a pulse stretcher. 2D TR-CIDNP spectra were acquired at ultrahigh resolution while maintaining sample integrity under multiple laser irradiations. Simultaneous application of laser and rf pulses enabled quantitative analysis of TR-CIDNP. Surface accessibility of four tyrosine residues and conformational change of α-Syn induced by divalent cations was accurately investigated by photo-CIDNP.

POSTER 065
Probing the Dynamics of Cataract-Related Human γS-crystallin Deamidation Variants
Presenting Author: Megan Rocha

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The human eye lens is a transparent tissue despite being filled at an impressive 400 mg/mL with a class of extremely-long lived proteins called crystallins. As crystallins age, they accumulate solubility damaging post-translation modifications, such as deamidation, until a tipping point is reached and cataract-related light scattering aggregates are formed. Our lab has shown that progressive deamidation of human γS-crystallin (HγS) increases the protein's susceptibility to oxidation with minimal structural perturbation. I hypothesize that distorted protein dynamics are the underlying cause of oxidation susceptibility in HγS deamidation variants. The fast dynamics of four HγS deamidation variants were determined by NMR spectroscopy. My data reveals that residues critical for HγS stability have significantly altered protein dynamics.

POSTER 066
Dynamic Basis for dA-dGTP and dA-d8OGTP Misincorporation via Hoogsteen Base Pairs
Presenting Author: Stephanie Gu

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Replicative errors contribute to genetic diversity for evolution but can lead to genomic instability. Here, we show DNA dynamics determines the frequency of misincorporating the A-G mismatch and altered dynamics explains the frequency of 8-oxoguanine misincorporation. NMR measurements revealed Aanti-Ganti (pop. >91%) forms sparsely-populated and short-lived Aanti+-Gsyn (pop. ~ 2%, kex ~ 137 s-1) and Asyn-Ganti (pop. ~ 6%, kex ~ 2200 s-1) Hoogsteen conformations. 8OG redistributed the ensemble rendering Aanti-8OGsyn dominant. A kinetic model where Aanti+-Gsyn is misincorporated quantitatively predicted dA-dGTP misincorporation by

human polymerase β, its pH dependence, and 8OG impact. Thus, 8OG increases replicative errors relative to G because guanine oxidation redistributes the ensemble in favor of Aanti-8OGsyn which exists transiently and in low-abundance in A-G.

POSTER 067
Identifying and Overcoming Artifacts in 1H-based Saturation Transfer NOE NMR Experiments
Presenting Author: Lucio Frydman

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Magnetization transfer experiments are versatile NMR tools providing site-specific information about organic and bio-molecules, as driven by polarization being transmitted by chemical exchanges or by cross-relaxation processes. We have recently discussed how saturation magnetization transfer (SMT) experiments could leverage repeated repolarizations arising from exchanges between labile and water protons, to enhance connectivities revealed via the nuclear Overhauser effect (NOE). Repeated experience with SMT has shown that a number of artifacts may arise in these experiments, which may confound the information being sought, in particular when seeking small NOEs among closely spaced resonances. Herein these phenomena are experimentally demonstrated, theoretically analyzed, and resolved with a number of proposals.

POSTER 068
Interaction studies of DNA G-quadruplex and Zuo1 protein complexes by NMR spectroscopy
Presenting Author: Ines Burkhart

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We investigate here the protein Zuo1, an eukaryote-specific J-protein from S. cerevisiae. Zuo1 was identified as a novel G4 binding protein in vitro and vivo and is able to influence the conformational equilibrium of G4s. Our findings suggest that the C-terminal domain is mainly responsible for G4 interactions. We investigated the complex formation of 15N-labelled Zuo1 with human and yeast derived G4-DNAs. Besides NMR spectroscopy we use CD spectroscopy and ITC to unravel the binding properties of the complex. Our results show that Zuo1 binds semi-stabilized G4 structures with stronger affinity than highly stabilized forms. We investigated two different sequences with various ionic conditions. Our data support the idea that Zuo1 discriminates between G4s with different thermodynamic stabilities.

POSTER 069
Dissecting the Conformation-Dependent Substrate Specificity of Vitronectin
Presenting Author: Kyungsoo Shin

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Vitronectin is a major serum protein capable of binding to many groups of molecules to modulate physiological and pathological processes. Vitronectin is often recruited to insoluble deposits associated with age-related diseases, but the biophysical mechanism behind this remains elusive. Previously, we determined the structure of vitronectin's largest domain (i.e., hemopexin-like domain). This four-bladed β-propeller domain is topologically complex with electrostatic protrusions and hydrophobic grooves for many potential molecular interactions. Using NMR, we identify the binding sites of various molecules observed in disease-related insoluble deposits. We demonstrate structural plasticity and how it regulates substrate specificity using biophysical and biochemical approaches, providing insight into how vitronectin conformational flexibility may be utilized to control its molecular interplay for disease progression.

POSTER 070
Cost-Efficient Stable Isotope Labeling of Proteins Overexpressed in Mammalian Cells
Presenting Author: Ravinder Elupula

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We have developed culture media containing stable isotope-labeled amino acids (15N and 13C15N) for mammalian HEK293T and insect Sf9 cells. The culture media are based on stable isotope-labeled protein hydrolysates from fermented Cupriavidus necator. The media were optimized to achieve high isotope incorporation and protein yields. For this purpose, the cells were adapted to low serum culture media. In addition, labeled biomass-derived hydrolysates, labeled yeast autolysates and lipid extracts were explored as media ingredients. Overexpressed eGFP yields in the new media were comparable with cells cultured in standard DMEM/F12 media. Based on pH, osmolality, glucose concentration and turbidity the media are stable for at least 8 weeks when stored at 4°C.

POSTER 071
Histone H4 Tail Conformation in Nucleosomes Studied by Paramagnetic NMR
Presenting Author: Wenjun Sun

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The packaging of DNA into chromatin plays a critical role in genome function. The fundamental repeat unit of chromatin, the nucleosome, consists of ~147 bp DNA and a histone protein octamer containing two copies each of histones H2A. H2B, H3 and H4. Dynamically disordered H4 N-terminal tails are key components in chromatin regulation. Here we investigate the conformational ensemble of these H4 tail domains in nucleosomes by paramagnetic relaxation enhancement (PRE) solution NMR spectroscopy and recombinant nucleosomes reconstituted with 15N-enriched H4 and labeled with paramagnetic tags at multiple histone H3 sites located on the nucleosome surface. The experimental PRE data are interpreted in conjunction with MD simulations, which indicate that H4 tails engage in a fuzzy interaction with nucleosomal DNA.

POSTER 072
Dynamic properties and functional roles of sizable intrinsically disordered linkers in villin/dematin family of cytoskeleton regulators in plants and humans.
Presenting Author: Serge L. Smirnov

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Dedicated actin-regulating proteins incessantly remodel non-covalent actin filaments of the cytoskeleton according to the changing environmental conditions. Villin, dematin and other related actin regulators have folded and sizable intrinsically disordered regions (IDRs) in their polypeptides. Plant villins have IDRs of 150-190 residues which connect two folded segments. Our data indicate that some of these IDRs are capable of specific F-actin binding thus providing novel actin binding sites. Dematin has a massive IDR (315 amino acids) which is capable of regulatory binding with the only folded domain in the protein. The poster presents NMR applications for deciphering dynamics and binding interfaces in the IDRs in plant villin and human dematin as well as NMR characterization of functional features in these proteins.

POSTER 073
Chemical shift assignment of 43x2kDa TrpB from Pyrococcus furiosus
Presenting Author: Hanna Kavaleuskaya

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The tryptophan synthase complex is an allosteric enzyme catalyzing the last two steps in the biosynthesis of the L-tryptophan in bacteria, plants, and fungi. The two reactions are kept in phase by allosteric interactions between the two subunits. Due to the size limit for solution-state NMR, the beta subunit TrpB has remained poorly characterized. To study the system of TrpB, we used combination of solution and solid-state NMR of a wild type PfTrpBwt and standalone PfTrpB2B9 from Pyrococcus furiosus. Backbone walk experiments, as well as 4D-SOFAST-HMQC-NOESY-HMQC were recorded for TrpB2B9. We were able to obtain 50% of assignment. This approach will allow us to analyze the mechanisms of allosteric communication in multi-subunit proteins on a milli- to micro-second timescale.

POSTER 074
Structure Elucidation of RNA Aptamers Bound to Derivative Hoechst Dyes
Presenting Author: Natasha Evans

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RNA aptamers are oligonucleotides that can specifically bind to small target molecules with high affinity and are typically selected through a process known as Systematic Evolution of Ligands by Exponential Enrichment. A 2008 study by Sando et al. identified an RNA aptamer that binds to a Hoechst dye derivative with tert-butyl (tBu) substituents. The goal of this research is to elucidate the structure of the tBu Hoechst-RNA complex using a combination of nuclear magnetic spectroscopy and computational modelling experiments. Preliminary results suggest that the RNA aptamer forms a stem-loop structure, with a central bulge region to accommodate for the bulky tBu groups. Determining the structure of this complex will allow for its potential use as an aptamer-based biosensor building block.

POSTER 075
Tackling a tripartite glycan conundrum: Flexibility/Sparse structural data/Signal resolution
Presenting Author: Marcos

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We face common, and often interconnected, challenges in the NMR structural studies of glycans, namely: 1) Discrimination of distinct conformations amongst a conformation-rich landscape; 2) Limited structural data for more reliable molecular modeling; 3) Spectral overlap, especially for homopolymers. We are tackling these issues by improving sensitivity and spectral resolution, though both cannot be simultaneously achieved. We show that labile 1H signals can help alleviate two of these challenges, increase structural data and provide a path to discriminate potentially "biologically active" conformations; therefore, improving the quality of both, the acquired data and the derived structural models. We also present our ongoing efforts to enhance the spectral resolution to enable the structural studies of larger homo- and hetero-oligosaccharides.

POSTER 076
Structural and Dynamical Investigation of the Histidine Triad in GMCSF
Presenting Author: Jennifer Cui

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GMCSF is a cytokine that displays promiscuous binding with ligands dependent on pH. A cluster of three histidine residues appear to be central to changes in structure and subsequent binding interactions. In order to understand the structure, stability, and motions contributed by each of the histidine residues composing the histidine triad, we have mutated each one to either Arg, Tyr or Asn. We have determined changes in structure and dynamics which allow us to rank importance of position and chemical properties of each histidine to the overall behaviour of GMCSF.

POSTER 077
Structural Characterization of Phosphoethanolamine Methyltransferase from P. falciparum Using Solution NMR Spectroscopy and MD Simulations
Presenting Author: Alexandra Pozhidaeva

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Recent emergence of drug-resistant malarial Plasmodium emphasizes the need for development of new treatments. Phosphatidylcholine, a major phospholipid of P. falciparum membranes, is synthesized through a pathway in which phosphoethanolamine methyltransferase (PfPMT) converts phosphoethanolamine to phosphocholine. This pathway is absent in mammals making the enzyme an attractive therapeutic target. Crystal structures of the PfPMT single S-adenosylmethionione-dependent catalytic domain with its substrates/inhibitors have been determined. Yet, the structure of the apo protein has never been crystalized suggesting conformational dynamics. Here, we characterize apo PfPMT and gain insight into the mechanism of its inhibition by amodiaquine using solution NMR methods in combination with molecular dynamics simulations. This work provides a basis for future efforts to develop new potent anti-malarial compounds.

POSTER 078
Determination of the Structural and Dynamical Properties of the crucial periplasmic chaperone SurA
Presenting Author: Filippo Castegnaro

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Survival protein A (SurA) is a crucial ATP-independent holdase chaperone residing in the periplasmic space of Gram negative bacteria and is essential for the cell viability in stationary phase. It plays a central role in the biogenesis of different outer membrane proteins (OMPs), transporting them from the inner to the outer membrane avoiding their misfolding and aggregation. Despite the general knowledge about SurA-function, the detailed mechanisms of how SurA transports its Omp cargo as well as how it transfers the unfolded OMPs to the BamA downstream remains elusive. To gain more knowledge about this important periplasmic chaperone and its role in OMPs biogenesis, we are studying the structure and dynamic properties of SurA at atomic level using solution NMR techniques.

POSTER 079
NMR Investigation of Cyclic-di-GMP Riboswitch Folding Pathway
Presenting Author: Ji-Yeon Shin

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Riboswitch is a structural RNA motif located at the 5'- end of bacterial mRNA, consisting of aptamer domain and expression platform. The aptamer domain binds a ligand, and the expression platform is responsible for gene expression by switching its conformation. Among the numerous types of riboswitches, this study investigates the folding pathway of the c-di-GMP riboswitch through NMR experiments. The conformational changes of its 3 states (free, apo and holo) under different conditions of Mg^{2+}, salt and c-di-GMP at the level of secondary and tertiary structure were determined through analysis of NMR spectra. Further NMR and ITC experiments with site-specific RNA mutations confirm the importance of tertiary interactions for the structural stability of RNA.

POSTER 080
A Study on the Target DNA Recognition of the Human Transcription Factor Meis1
Presenting Author: Seo-Ree Choi

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Myeloid ecotropic viral integration site-1 (Meis1) is a human transcription factor and plays an important role in myeloid leukemia. Meis1 contains a three amino acids loop extension (TALE) homeobox domain (HD) and specifically binds to a common DNA sequence, 5' – TGACA - 3'. In this study, we performed NMR experiments on the complexes of TALE-HD of human Meis1 (Meis1-HD) with a decamer DNA duplex containing its consensus sequence. We prepared the mutant DNA duplexes containing a single base-pair substitution as well as double or triple substitution mutants and compared the Meis1-HD-DNA interactions using HSQC titrations. This study provides key structural features of the Meis-HD-DNA complex and the information about the molecular mechanism of target DNA recognition of the MEIS1.

POSTER 081
Structural Investigation of Human U6 snRNA Recognition by The Spliceosome Recycling Factor SART3
Presenting Author: Kyeong-Mi Bang

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Human SART3 is a multifunctional protein involved in the pre-mRNA splicing, including assembly and recycling of the U4/U6 snRNP. SART3 contains two RRMs at the C-terminus, whereas the homologous yeast Prp24 employs four RRMs for specific U6 snRNA recognition. We investigated the tertiary interaction between RRMs and U6 snRNA using biochemical assays and NMR methods. We report monomeric SART3 binds tightly to the asymmetric bulge of U6 snRNA, demonstrating that two RRMs sufficient to bind the U6 snRNA. SART3 RRMs adopt a tandem βαββαβ motif, and they bind to the bulge region of U6 snRNA via a conserved electropositive surface and aromatic residues. Also, we confirm that 5'-end regions of bulge of U6 snRNA interact with SART3 RRMs.

POSTER 082
NMR Hydrogen Exchange study of wild type and mutant MIR390a
Presenting Author: Ho-seong, Jin

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In plants, primary transcripts with miRNA foldbacks are processed by the RNase-III like enzyme DICER-LIKE1 complexed with HYPONASTIC LEAVES1 and SERRATE to generate miR/miR* duplex. The levels of mature miR390 influence the leaf number prior to flowering in the life cycle of plants. The G-to-A point mutation that was calculated to stabilize a relatively nonpaired region near the base of the miR390a foldback, resulting in misprocessing of the miR390/miR390* duplex. To understand the molecular mechanism of biogenesis of primary miR390a to mature miR390, an NMR hydrogen exchange study was performed using model RNAs mimicking the cleavage site of wild-type (WT) and bulge-stabilizing mutant pri-miR390a constructs.

POSTER 083
Towards Understanding Protein Quality Control of Integral Membrane Proteins by the Bacterial Metalloprotease FtsH
Presenting Author: Hannah Fremlen

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FtsH is an essential zinc-dependent integral membrane protease residing in the Escherichia coli (E. coli) inner membrane responsible for degradation of unfolded or aggregated proteins as a part of the protein quality control machinery. As a member of the AAA+ protein family, FtsH requires several cycles of ATP to unfold and translocate substrates for subsequent cleavage. Both the ATPase domain and the protease domain reside on a single polypeptide chain in the cytosolic region and assembles into a homohexamer with a ring-like structure. Despite its vital role in E. coli, still very little is known about this membrane protein. To gain further insight into the detailed structure and dynamic properties of FtsH, solution NMR is used as the main method.

POSTER 084
Structural Elucidation of the PH Domain of Akt-like Kinase in Trypanosoma cruzi: A New Target for Treatment of Chagas Disease
Presenting Author: Karina A. Stadler

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Chagas disease is caused by the protozoan parasite Trypanosoma cruzi. The disease is endemic to Latin America but current treatment options are inefficient and cause severe side effects. Although Akt-like kinase (TcAkt) represents a promising drug target, its structure and mechanism of action are still not resolved. Human Akt is stated to be activated by phosphoinositide (PIP) binding, enabling the pleckstrin homology (PH) domain to dislodge from the kinase domain. The structure and binding studies of the N-terminal TcAkt PH domain reveal PIP-induced structural changes, highly associated with kinase activation. Our findings reveal unique insights into the structure and functionality of the so far scarcely understood TcAkt, thereby forming the basis for the development of efficient drugs against Chagas disease.

POSTER 085
How to find needles in a haystack: an STTD on-cell NMR method to boost interaction studies
Presenting Author: Tamás Milán Nagy

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Saturation Transfer Difference (STD) NMR experiments have been used routinely to characterize protein-ligand interactions. However, care has to be taken when studying complex systems with on-cell STD methods; interference should be avoided with any non-specific interactions of small or high molecular weight components present in cells.
The triple difference STD (STTD) strategy proposed here can eliminate the contribution of any undesired specific and non-specific interactions, and so the resulting spectrum reveals the direct binding between the protein and ligand of interest. Experimental findings are demonstrated on different biological samples. First, the binding between rapamycin and the TRPM8 menthol receptor is investigated in atomic detail, then interactions between endomorphin-2 and mu-opioid receptor (MOR) mutants are presented.

POSTER 086
Structural Basis of Substrate Recognition and Allosteric Activation of the Pro-apoptotic Mitochondrial HtrA2 Protease
Presenting Author: Emelie Aspholm

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HtrA2 is a mitochondrial serine protease of the HtrA family found in all kingdoms of life. Residing in the inner mitochondrial membrane it exerts a role in protein quality control and acts as a proapoptotic factor when released into the cytosol, cleaving inhibitor of apoptosis (IAP) proteins such as XIAP. HtrA2 has been implicated in Parkinson's and Alzheimer's disease and in several different cancer types, making it an important target of study. We have used advanced solution NMR spectroscopy methods together with biophysical and biochemical characterization to show how HtrA2 is allosterically activated via its PDZ domain. Further, we show that divalent metal ions can modulate the activity of HtrA2, refining the model of HtrA2 regulation in the apoptotic pathway.

POSTER 087
Structural studies of AgA by solution NMR
Presenting Author: Maria Luiza Caldas Nogueira

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Dental caries is one of the most prevalent infectious diseases. It has a high impact on an individual's quality of life and its healthcare-associated costs. S. mutans is the primary pathogen that causes dental caries resulting from biofilm formation. Biofilms are composed of polysaccharides, proteins, and eDNAs. S. mutans has four known amyloidogenic proteins: SMU_63c, Adhesin P1, WapA, and cnm. WapA is cleaved by sortase Aga. WapA binds collagen I, and fibronectin, and promotes dendritic cell maturation. It is a vaccine candidate against S. mutans caused teeth caries. Despite its importance, the AgA structure is still not available. Here we use solution NMR to perform structural studies of the AgA.

POSTER 088
Using NMR to enable the discovery of small molecules stabilizing the interaction between 14-3-3 and Estrogen receptor α
Presenting Author: Adam Lewis

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The 14-3-3 adapter protein binds an array of client proteins. The biological consequence of 14-3-3 binding depends on the client in question but can affect cellular localization, activity, stability, trafficking or complex formation. It was demonstrated that phosphorylation of Estrogen receptor α (ERα) at T594 favors binding of 14-3-3, thereby inhibiting receptor dimerization and transcriptional activity. We initiated an effort to identify small molecules that enhance the 14-3-3:ERα interaction. Primary screening hits were validated using two NMR-based assays: a 19F-CPMG-1D reporter assay and a 1H,15N-2D-TROSY-HSQC assay. The 19F reporter assay confirmed complex stabilization while 2D NMR confirmed binding. These efforts allowed prioritization of hits for X-ray confirmation and medicinal chemistry optimization.

POSTER 089
Human Znf706: A Tiny G-quadruplex-Binding Protein
Presenting Author: BIKASH R. SAHOO

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The cellular function and structure of a large percentage of proteins containing disorder remains enigmatic. We identified that a tiny human protein Znf706 that is characterized by a high degree of charge and partial disorder binds to G-quadruplexes, an emerging cellular component whose biological roles need to be better defined. NMR studies combined with other biophysical, biochemical, and cellular approach revealed that Znf706 preferentially binds to G-quadruplex with sub-micromolar to nanomolar affinity. 19F NMR revealed Znf706's preference to bind parallel G-quadruplexes, and NMR backbone assignments of Znf706 enabled us to identify G-quadruplexes binding to its dynamic and disordered N-terminal domain. Znf706 and well-characterized G-quadruplexes serve as biophysically amenable models to help in the understanding of protein and G-quadruplex interactions.

POSTER 090
Elucidating Biomacromolecular Interactions with DIrect Saturation COmpensated (DISCO) NMR
Presenting Author: Darcy C. Burns

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Interactions between protein and macromolecules are imperative to biomaterials development. When exogenous materials are introduced to biological fluids they are rapidly bound by native protein. The nature of these binding interactions determines the fate of the candidate materials. Despite their importance, the causal link between polymeric biomaterials composition and their protein binding mechanisms are poorly understood. This is partly driven by the lack of tools to investigate the underlying solution-state binding interactions at atomic resolution. We developed Direct Saturation Compensated NMR to help address this shortfall. DISCO is a refinement of the saturation transfer difference (STD) and double difference (STDD) techniques that correct for R.F. irradiation spillover artifacts and was used to probe binding interactions in different mucin-polymer bioconjugates.

POSTER 091
Ligands and Fast Dynamics in the Neurotensin GPCR: Extracting Parameters from High Noise Triple Quantum Relaxation Data.
Presenting Author: Scott Anthony Robson

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G protein-coupled receptors (GPCRs) convey ligand-based signals from outside to inside the cell through changes within the GPCR itself. The signaling information that can be passed is multifaceted and dynamic changes within the receptor seem to play a role in how signals are transmitted. We have used 3Q/SQ methyl relaxation experiments to monitor fast dynamics of methionine sidechains in the presence of various ligands for the Neurotensin receptor. These experiments are insensitive and low solubility of GPCR/micelle samples means data collection and analysis is challenging. We have adopted Bayesian parameter estimation to extract order parameters by simultaneously fitting models for 3Q and SQ relaxation rather than the traditional 3Q/SQ ratio method. This approach is more robust given high noise.

POSTER 092
Pulsed Saturation in Hyper-CEST NMR for Hosts with Different Exchange Kinetics
Presenting Author: David Hernandez

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HyperCEST NMR with exchanging hyperpolarized 129Xe and molecular hosts reveals the presence of such molecules at picomolar detection limits. The combination of applied RF saturation and the Xe exchange kinetics is critical to achieve optimum signal contrast while avoiding unwanted RF heating. Here, we study the HyperCEST responses of two types of Xe hosts for BP and dSNOB saturation. Depolarization of Xe with a short residence time in hosts with desirable faster exchange benefits from the shaped pulses. However, the full potential of such agents can only be realized when strong saturation is applied where the dSNOB pulses are slightly outperformed by the BP scheme. With a growing family of HyperCEST reporters, the optimum saturation scheme should be chosen carefully.

POSTER 093
Probing Glycan-Galectin Interactions with State-Of-The-Art Multinuclear NMR and Computational Methods
Presenting Author: István Timári

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The inhibition of glycan-galectin interactions represents a promising perspective towards developing therapeutics controlling cancer development. We have investigated the binding of multiple human Galectin-3 (hGal-3) inhibitors, specifically that of di(β-D-galactopyranosyl)selenide (SeDG), and di(β-D-galactopyranosyl)diselenide (DSeDG) analog. The binding affinities of these derivatives to hGal-3 were determined by 1H-15N HSQC and competition STD experiments. We have demonstrated that the enhanced detection sensitivity inherent in our original 1H-77Se CPMG-HSQMBC method got a further significant boost by using 77Se-enriched ligands,

such as [77Se]DG. Our work opens perspectives for utilizing isotopically enriched selenoglycosides for rapid monitoring of lectin-binding of selenated as well as non-selenated ligands. The synthesis, NMR and computational study of novel glycomimetics targeting of hGal-3 protein are on the way in our group.

POSTER 094
Using Intermolecular Paramagnetic Relaxation Enhancements to Constrain Dynein/Dynactin Interactions
Presenting Author: Nikolaus M. Loening

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Cytoplasmic dyneins are multiprotein complexes that carry out retrograde transport in cells. In mammalian dynein, processive motion is only observed when dynein interacts with another protein complex, dynactin. The main site of interaction between dynein and dynactin is the N-terminal portion of dynein intermediate chain (N-IC) and the coiled-coil 1B (CC1B) region of the p150Glued subunit of dynactin. Despite evidence for this interaction from binding studies, the exact location of where these proteins bind remains elusive due to the dynamic nature of the interaction and the presence of intrinsically-disordered regions in IC. By using intermolecular paramagnetic relaxation enhancements (PREs) we have been able to constrain the location of IC binding on p150Glued.

POSTER 095
Utilizing SAR by NMR and CADD-based Screening Methods to Develop Novel CDTb-RBD2 Drug Inhibitors
Presenting Author: Mary Cook

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C. diff is an oft-reoccurring and hard-to-treat gut infection, especially in strains containing binary toxin (CDT), which possesses an ART toxin component (CDTa) and a pore-forming/delivery subunit (CDTb). CDTb assembles into symmetric and asymmetric di-heptameric states, both of which possess surface-accessible receptor binding domains (RBD1 & RBD2). RBD2 has been shown to be critical for CDT toxicity, making it the target for NMR-based drug discovery studies. Utilizing "SAR by NMR", 2D [1H, 15N]-HSQCs were collected from a small molecule fragment screen, looking for perturbations that indicated binding effects - and therefore possible binding sites. These sites were confirmed with SILCS, which was then used (with other CADD methods) to generate a compound list for the next round of NMR-based screening.

POSTER 096
Specificity-enhancing Mutations Remodel Dynamic Allostery in CRISPR-Cas9
Presenting Author: Erin Skeens

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CRISPR-Cas9 is an innovative genome editing tool with broad applications in bioengineering. However, the occurrence of off-target effects has limited its use as a precision therapeutic for human disease. Efforts to engineer variants of Streptococcus pyogenes Cas9 for increased specificity have revealed that Rec3, a subdomain of the recognition lobe, is an important functional handle for specificity, as many variants contain the majority of their specificity-conferring mutations within Rec3. The mechanisms by which mutations in Rec3 contribute to the specificity of Cas9, especially considering their distance from the catalytic sites, is not well understood. We employed solution NMR spectroscopy and MD simulations to characterize the structural and dynamic effects of high-specificity mutations on Rec3 and more broadly, Cas9

POSTER 097
Development of a high-resolution NMR technique to measure diffusion of proteins at near-physiological conditions
Presenting Author: Jongchan Lee

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Measuring the translational diffusion of proteins can provide useful information about their size, shape, and surrounding environments. NMR technique, called diffusion NMR or diffusion-ordered spectroscopy (DOSY), is widely used to study the diffusion of molecules. In this study, we have proposed a method that separates the nuclei used for diffusion measurement (alpha protons, 1Hα) and those used for detection (1H/15N and 13C'/15N correlations). This effort improved the resolution of diffusion measurements on polypeptides in a mixture of biomolecules, thereby permitting the investigation of coexisting species under near-physiological conditions.

POSTER 098
A Potassium Ion-Dependent Structural Switch in the G-rich Region of a Long-Noncoding RNA
Presenting Author: Jasna Brcic

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Long-noncoding RNA REG1CP was shown to promote cancer cell proliferation and tumorigenicity by activating the REG3A gene in colorectal cancer. The mechanism involves REG1CP binding and recruiting helicase FANCJ to the REG3A locus. A guanine-rich (G-rich) sequence within REG1CP was proposed to fold into a non-canonical structure called G-quadruplex (G4) which is recognized by FANCJ. We show by NMR that a G-rich oligoribonucleotide from REG1CP forms two structures, a hairpin (HP) and a G4, that coexist in slow exchange in K+ solution and provide insight into their folding topologies, interconversion, and how they interact with FANCJ-peptides. Our structural study suggests that conformational switching between G4 and HP structures within REG1CP could modulate REG1CP and FANCJ interaction implicated in colon cancer.

POSTER 099
NMRFAM User Program
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The NMRFAM user program provides access to 10 NMR spectrometers (500 MHz – 900 MHz) equipped for a variety of solution and solid-state NMR experiments, with three more spectrometers scheduled for installation this year (including 1.1 GHz). Our scientists provide advice and assistance in experimental design, data acquisition and processing for studies of molecular structure, dynamics and interactions. We have experience with a range of sample types, including soluble and membrane proteins, fibrils, RNA, small molecules, and metabolomics. We offer training in experimental design, data acquisition, data processing and analysis for solution and solid-state NMR of proteins and RNA.

POSTER 100
Backbone Conformational Equilibrium Correlates with Enzyme Activity in Mismatched DNA
Presenting Author: Gary Meints

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A mystery remains regarding how repair enzymes such as thymine DNA glycosylase (TDG) identify a canonical DNA base in the incorrect pairing context. We have previously used 31P NMR to investigate the energetics of DNA backbone BI-BII interconversion and the effect of a mismatch or lesion compared to canonical DNA. We found perturbations to the free energy (~1 kcal/mol) and enthalpy (2-5 kcal/mol) of activation for the BI-BII interconversion localized to the phosphates flanking the mismatch. We found correlations of our DNA phosphate backbone equilibrium (Keq) to enzyme kinetics or binding parameters of several enzymes, suggesting the backbone equilibrium may play a role in mismatch recognition and/or conformational rearrangement and energetics during nucleotide flipping or other aspects of enzyme interrogation.

POSTER 101
Structural and functional characterization of SARS-CoV-2 Nucleocapsid linker reveal RNA binding and regions of self-association
Presenting Author: Hannah Stuwe

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The nucleocapsid protein (N) of SARS-CoV-2 is essential for virus replication, genome packaging, and maturation. N is comprised of N-terminal and C-terminal folded domains that are separated by a disordered, Ser,Arg-rich, linker and flanked by disordered tails. We have analyzed the linker region using NMR spectroscopy and other biophysical techniques. Our results show that the linker region binds viral RNA and that this binding is modulated by phosphorylation and naturally occurring mutations. Further, we have assigned an alpha-helical region in the linker and shown that this region undergoes concentration dependent self-association. These data show that the linker region can contribute to functions normally associated with the folded domains and suggests it could regulate protein-protein interactions and RNA-protein interactions.

POSTER 102
Structural determinants of the versatile functions of RNA complexes in the SARS-CoV-2 Nucleocapsid
Presenting Author: Patrick N. Reardon

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The nucleocapsid (N) protein of the SARS-CoV-2 coronavirus binds to viral RNA, condensing it inside the virion, facilitating viral transcription in infected cells, and phase separating with RNA to form liquid condensates. However, the structural determinants of these interactions are not clear. To identify the role of RNA structure in mediating these interactions, we characterize the binding between the folded domains of N and short model RNA segments. We demonstrate that the N-terminal domain (NTD) binds preferentially to single-stranded RNA. We identify a second, weak RNA-binding face on the NTD and another on the C-terminal domain that are critical for phase separation. We propose a model of how variations in N-RNA binding can promote binding, phase separation, or virion formation.

POSTER 103
Targeting Receptor Binding Domain 1 (RBD1) From Clostridioides difficile Binary Toxin (CDT) With Drug-Like Fragments
Presenting Author: Spiridon E Sevdalis

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Hypervirulent cases of Clostridioides difficile (C. difficile) infection express C. difficile binary toxin (CDT). CDT is comprised of a catalytic A subunit (CDTa) and seven cell binding/translocase B subunits (CDTb). CDTb's receptor binding domains (RBDs) were revealed to be important for CDT toxicity. A novel calcium-binding site was discovered in RBD1, facilitating the formation of a stable conformation in the presence of calcium. As RBD1's fold facilitates CDT activity, development of inhibitors specific to RBD1 was initiated. Screening of RBD1 in the presence of 1000 chemical fragments was monitored by tracking residue CSPs from 2D [1H, 15N]-HSQC spectra. These NMR data informed Site Identification by Ligand Competitive Saturation (SILCS) simulations to generate larger, drug-like compounds specific for RBD1.

POSTER 104
Biophysical Characterization of Novel Plexin–GTPase Interactions
Presenting Author: Chinmayi Prasanna

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Plexin receptors play a crucial role in neuronal development, particularly in axon guidance, cytoskeletal re-arrangement, and signal transduction. In earlier work, Buck lab discovered a unique Rho GTPase binding domain (RBD) in plexins. The Rho-family regulatory GTPases interact with RBD carrying out plexin regulation in the intracellular environment. In addition to this region, another part of the plexin intracellular region appears to interact tightly with certain Rho GTPases. The structural mechanism of the effects of Rho GTPases on plexin signaling still remains elusive. In this study, we use various plexin constructs to probe interaction with Rho GTPases using–microscale thermophoresis (MST), nuclear magnetic resonance (NMR) spectroscopy, and AlphaFold 2 (AF2) based predictions and obtain structural and sequence-specific insights.

POSTER 106
Thermodynamic and kinetic characterization of non-conventional hydrogen bonding in Lewis antigens
Presenting Author: Jeahoo Kwon

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The Lewis antigens are a well-known family, whose structures were thought to be conformationally inflexible, until recently. Herein, we provide evidence for conformational flexibility between hydrogen bonded conformations and non-hydrogen bonded for 10 Lewis antigens and two of their rhamnose analogs. We also characterize the thermodynamics and kinetics of the H-bonds in these molecules, using an alternative method to simultaneously fit a series of temperature-dependent fast-exchange NMR spectra. We determined that the H-bonded conformation is favored by approximately 1 kcal/mol over the non-H-bonded conformation. Additionally, comparison of temperature-dependent 13C linewidths in various Lewis antigens and the two rhamnose analogs, reveals H-bonds between the carbonyl oxygen of the N-Acetyl groups of N-Acetylglucosamine and the OH2 group of galactose/fucose.

POSTER 107
Progress Towards Characterizing a Solution Phase NMR Oligomer of Peptides Derived from Aβ
Presenting Author: Jason Zhu

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The Aβ peptide assembles into oligomers and fibrils that are central to the pathology of Alzheimer's disease (AD). Despite mounting evidence suggesting that oligomeric species are neurotoxic, there are few high-resolution models of Aβ oligomer assembly in aqueous solution. To address the need for models of cytotoxic oligomers, I synthesized peptides derived from the amyloidogenic fragments from the central and C-terminal regions of Aβ and evaluated their assembly using 1H NMR, TOCSY, NOESY, DOSY, and other NMR techniques. The amyloidogenic peptides adopt β-hairpin conformations, are soluble at millimolar concentrations, and show concentration dependent oligomerization. These studies provide insight into how Aβ oligomerizes in aqueous solution.

POSTER 108
Investigating the Structural Change of Ci-$\beta\gamma$-Crystallin at Various Calcium Concentrations
Presenting Author: Matthew Jimenez

Complete Author List:
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Tunicate (Ci-$\beta\gamma$-crystallin) is from a vertebrate $\beta\gamma$-crystallin lineage before the evolution of the lens. The $\beta\gamma$-crystallin superfamily contains both the vertebrate eye lens and the microbial calcium binding proteins. The vertebrate β- and γ- crystallins are structural proteins that make up the refractive tissue of the lens while the microbial $\beta\gamma$-crystallin proteins bind to divalent cations. Tunicate contains both properties. Studying the calcium binding affinity on tunicate through various concentrations will describe the evolution process of $\beta\gamma$-crystallins and the role of calcium binding stability. This is done through titrating different protein to calcium concentration ratios on tunicate then taking HSQC's to determine the calcium binding sites and the structural changes that tunicate undergoes through various ratios of calcium.

POSTER 109
Structural Insights into Selective Complexation of Rare Earth Elements by Peptide Surfactants Revealed by NMR Spectroscopy
Presenting Author: Surabh KT

Complete Author List:
Surabh KT (City College of New York); Denize C. Favaro (Advanced Science Research Center); Luis Ortuno (City College of New York); Charles Maldarelli (City College of New York); Robert J. Messinger (City College of New York)

Rare earth elements (REEs) are crucial components in numerous modern technologies. However, current REE separation processes are challenging, energy-intensive, and detrimental to the environment. Recently, we developed an eco-friendly REE separation process at the air-water interface using a peptide surfactant (LBT1LLA) derived from Ca^{2+} binding loops in calmodulin. The peptide surfactants bind trivalent REE^{3+} cations with high affinity and show selective complexation along the lanthanide series, enabling subsequent recovery and isolation. Here, we elucidate the 3D molecular structures of bound and unbound LBT1LLA using multidimensional solution-state biomolecular NMR to better understand ion coordination in the binding loop. In addition, we identify specific residues that perturb REE^{3+} selectivity, yielding insights into design principles for targeted REE complexation.

POSTER 110
Structure and Chaperone Activity of Human Heat Shock Protein B8
Presenting Author: Daniel C. Farb

Complete Author List:
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Aggregation of certain globular proteins has been implicated with neurodegenerative diseases such as ALS and dementia. HspB8 (Heat shock protein B8), an ATP-independent chaperone protein, binds misfolded proteins and thereby prevents their pathological aggregation. To fully understand the chaperone activity of HspB8, a high-resolution structure of the chaperone is needed. Size-exclusion chromatography and MALDI-TOF mass spectrometry have confirmed that HspB8 occurs in a monomeric and dimeric state in solution, therefore solution NMR is a suitable tool for this. Solution NMR relaxation measurements and chemical shift perturbations will report on the structural transformation in the head and tail domains of HspB8 upon interaction with misfolded proteins. Here, we present our initial characterization of HspB8 and assess sample conditions for NMR measurements.

POSTER 111
HIV-1 p17 Interactions with Heparan Sulfate
Presenting Author: Kari Pederson

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Glycosaminoglycans (GAGs) are expressed ubiquitously on mammalian cell surfaces and interact with a wide variety of biological molecules to modulate processes, including immune response, regulation of cell growth and blood-stream clotting. Disruption of GAG-binding has the potential to prevent infection by viruses, such as HIV-1, and to reduce inflammation caused by autoimmune disorders. Within HIV-1, three proteins are known to bind to the sugars present on the surfaces of human cells. One of these, p17, is the focus of this study. Here we investigate the sequence specificity of the heparan sulfate-p17 interaction using microarray screening and NMR titration. HIV-1 p17 demonstrates a preference for binding to 2-O-sulfated heparan sulfate, which may present a target for future drug development.

POSTER 112
Phosphopeptide Binding Modulates Arrestin 2 Isoleucine Conformational Dynamics
Presenting Author: Tucker Shriver

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Arrestins underpin critical signaling pathways in the body, yet the structural mechanism of their activation remains unclear. Crystal structure data of bound and unbound structures of Arrestin 2 reveal the presence of a 20 degree interdomain twist between portions of the protein upon binding. Previous work by Shiraishi et al (2021) determined that multiple methyl NMR peaks occur for single isoleucine residues, suggesting the presence of slow conformational exchange. This work shows that this exchange is sensitive to the presence of the fully phosphorylated Vasopressin-2 receptor c-terminal peptide (V2Rpp) and that the Ile residues experience similar global thermodynamic and conformational changes upon V2Rpp binding. Such behavior suggests that these isoleucine methyls may be sensitive probes of Arrestin 2 activation.

POSTER 113
Pre-Homonuclear Decoupling: NMR Measurements of Intrinsically Disordered Proteins at Ultrahigh Resolution
Presenting Author: Sohyun Jung

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Intrinsically disordered proteins (IDPs) carry out important functions in cells and are related to the pathogenesis of many neurodegenerative diseases. To analyze IDPs, it is necessary to resolve different NMR signals generating from IDP residues. In this work, we present a new homonuclear decoupling scheme called Pre-Homonuclear Decoupling (PHD). The PHD method does not apply radiofrequency pulses and pulsed field gradients during the FID period, but is rather based on the in-phase/antiphase (IPAP) principle. We applied PHD to HSQC/TROSY and observed 3-fold narrower 1HN line widths compared with the control experiments. The PHD scheme provided narrow 1HN line widths with minimal artifacts for high-resolution analysis of IDPs.

ECLECTICA IN MAGNETIC RESONANCE (Posters 114 – 121)

POSTER 114
Electric-field-induced orientation of molecules in solution
Presenting Author: Ulrich Scheler

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In-situ application of a strong electric field induces orientation of molecules bearing an electric dipole moment in aqueous solution. In proton-only experiments the resulting residual dipolar coupling is detected in a J-resolved experiment. For a small molecule ß-alanine a weak order parameter is observed. A peptide of 10 residues exhibiting a dipole moment of 200 D shows a residual dipolar coupling that can be described by a Gaussian broadening of the dipolar lineshape and is scaling with the applied electric field. The electric field is applied synchronously to the NMR experiment and can thus be applied selectively for certain periods of the experiment.

POSTER 115
Artificial Intelligence (AI)-based Lanthanide Sensing Utilizing ^{19}F-Paramagnetic Guest Exchange Saturation Transfer (^{19}F-ParaGEST) Fingerprinting
Presenting Author: Elad Goren

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Highly-selective non-fluorescent analytical tools are required to monitor complex media lanthanide traces. Here, we combine our lanthanide-based ^{19}F paramagnetic guest exchange saturation transfer (**^{19}F-paraGEST**) approach with magnetic resonance fingerprinting (**MRF**) to show an **AI-driven NMR-based lanthanide sensing platform**.
For this, a Bloch-McConnell-equations-based library of ^{19}F-paraGEST effects was generated for Ln-α-CD (host) - fluorinated guest pairs and trained an AI-based system to reveal lanthanides' identity and concentration in an unknown solution. Using an optimized MRF acquisition protocol, the platform was examined on combinations of Ln-α-CDs, where the AI-MRF system recognized up to three lanthanides and their concentrations.
We envision **^{19}F-paraGEST MRF**, currently developed for nine lanthanides, will allow fast and selective sensing of lanthanides in wastes for future green chemistry applications.

POSTER 116
On the Effects of Quadrupolar Relaxation in Earth's Field NMR Spectra
Presenting Author: Adam Robert Altenhof

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NMR conducted at earth's field is not sensitive to the chemical shift interactions and yields only J-coupled spectra (JCS). These experiments offer several benefits over those at high field; however, several aspects of JCS have not been explored, such as the effects of relaxation from quadrupolar heteronuclei. Coupling from 14N to 19F or 1H is common in small organic compounds and can have a significant impact on the JCS depending on the magnitude of T1(14N) and T2(14N). Herein, I will describe a study on fluoropyridine samples with unique fluorine substitution that demonstrates the effects of 14N relaxation on JCS. SPINACH simulations are used to model the magnitudes and signs of all J-couplings for fluoropyridines and to determine T1(14N) and T2(14N).

POSTER 117
Stereochemical Effects on Chemical Exchange Measured with Homonuclear J-coupling Spectroscopy
Presenting Author: Stephen Devience

Complete Author List:
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We demonstrate stereochemical effects on proton exchange between a chiral solvent and chiral solute. We used homonuclear J-coupling spectroscopy with the Synchronized Echo pulse sequence at 6.5 mT to measure proton exchange between the chiral solvent ethyl L-lactate and both achiral and chiral solutes. Spectra of ethyl L-lactate mixed with water show that proton exchange modulates the intensity of spectral dips. Ethyl L-lactate spectra in the presence of either L- or D-mandelic acid showed no difference. However, spectra of ethyl L-lactate mixed with the ester methyl L- or D-mandelate showed that the D enantiomer exhibited faster exchange. We hypothesize that differences in the geometry of collisions and/or hydrogen bonding with L vs. D entantiomers strongly affect the rate of exchange.

POSTER 118
Is CEST Possible at Ultralow Fields? 1H-14N Scalar Relaxation as an Alternative to Saturation Pulses
Presenting Author: David E. Korenchan

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Ultralow-field MRI would benefit from access to molecular information, but the absence of measurable chemical shifts poses a major challenge. We propose to sensitize proton signal to amide and/or amine chemical exchange by inducing the depolarization of proton spins via scalar coupling to nitrogen-14, producing an effect analogous to proton radiofrequency saturation in chemical exchange saturation transfer (CEST) MRI. We demonstrate with high-field NMR spectroscopy of a highly concentrated urea sample that we can induce amide proton scalar relaxation by Hartmann-Hahn matching, and we show that the resulting signal depletion decreases the water proton pool via chemical exchange. Future work will adapt this technique to ultralow-field MRI scanners to provide chemical exchange contrast on low-cost MRI scanners.

POSTER 119
Boosting 19F-NMR Sensitivity via Spectral Focusing for Target Environmental Analysis
Presenting Author: Flavio Vinicius Crizostomo Kock

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In this work, a new NMR approach that uses single and multiple focusing to select discrete signals and/or chemical shift windows is introduced. In summary, the target 19F signals are first isolated by a gradient spin echo, and then focused by a train of shaped pulses during acquisition, with real time compensation for off-resonance phase decoherence. The user can choose whether to target one species (for example a multiplet from a single compound) or a window (for example all (CF2)n groups, to gauge long chain content in a mixture) depending on the goal of the study. Detection limits are improved by one order of magnitude for simple well dissolved systems and approaching 2 orders of magnitude for complex polymeric systems.

POSTER 120
Solid State NMR Analysis of Chemical Agent Degradation on Composite Beads
Presenting Author: David J. McGarvey, Ph.D.

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Using High-Resolution Magic Angle Spinning (HR-MAS) techniques, the reactions of chemical agents on composite beads were investigated. The composite beads consist of an active component that is integrated into a polymer matrix. The non-destructive nature of the NMR experiment allowed for the determination of the kinetics of the degradation, and a half-life of the agent on the matrix was determined. Both structural and quantitative information can be obtained simultaneously, so the identity of the breakdown products could be determined, as well as the rate of formation of each over time.
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POSTER 121
Water-Proton T2 Relaxation in Tree Leaves Using Nuclear Magnetic Resonance at Hypogeomagnetic Fields
Presenting Author: Anne Fabricant

Complete Author List:
Anne Fabricant (Helmholtz Institute, University of Mainz); Dmitry Budker (Helmholtz Institute, JGU Mainz and UC Berkeley); Danila Barskiy (Johannes Gutenberg University Mainz)

In our labs, we are developing a portfolio of experiments which employ magnetic-sensing techniques from physics and chemistry to investigate vital processes in living plants. Here we report on a relaxometry setup within the framework of zero-to-ultralow-field nuclear magnetic resonance (ZULF NMR) applied to the study of water dynamics in intact ex vivo green leaves.

HYPERPOLARIZATION METHODOLOGIES (Posters 122 – 168)

POSTER 122
Towards Tabletop Recyclable Hyperpolarization with a Compact Freeze, Melt, and Flow DNP Polarizer
Presenting Author: Charlotte Bocquelet

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Hyperpolarization methods provide a way to greatly improve the inherently low sensitivity of NMR. Dissolution Dynamic Nuclear Polarization (d-DNP) was introduced more than twenty years ago, and now provides a 10'000-fold gain in sensitivity on a routine basis. However, the experiment is in a single-shot manner, therefore mostly incompatible with NMR spectroscopy, ruling out for example phase cycling and multidimensional sequences. Our ambition is to turn dissolution DNP into a new version that will be widely compatible with NMR spectroscopy by replenishing hyperpolarization with a compact closed-loop freeze, melt, and flow system. Here I will present the design and performances of the polarizer, with some preliminary results regarding the flow.

POSTER 123
13C Radio Amplification By Stimulated Emission of Radiation (RASER) of Hyperpolarized Pyruvate
Presenting Author: Shiraz Nantogma

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We demonstrate 13C RASER from tautomeric mixture of HP allyl [1-13C]pyruvate (ketal and hemiketal forms) prepared by hydrogenation of propargyl [1-13C]pyruvate with parahydrogen. 13C RASER pyruvate signals are produced from sample concentrations as low as 125 mM using commercial detector with Q of 32. We also show that although the substrate undergoes fast exchange (compared to the time scale of the experiment) between its ketal and hemiketal forms, the 13C RASER signal from one species does not "bleed" to less concentrated species that cannot enter RASER emission on its own. This work paves the way for 13C molecular imaging of HP pyruvate on conventional MRI scanners that lack 13C excitation electronics.

POSTER 124
Hyperpolarized 13C NMR of Biofluid Samples at Natural Abundance by Dissolution Dynamic Nuclear Polarization
Presenting Author: Victor Ribay

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Dissolution dynamic nuclear polarization (d-DNP) has recently shown promises for applications to 13C NMR metabolomics at natural abundance, however, previous studies have been restricted to metabolite extracts. Here we report, for the first time, the suitability of 13C d-DNP to provide rich information on a biofluid (urine). Single-scan 13C spectra recorded after d-DNP exhibit dozens of metabolite signals at natural abundance with a high resolution and repeatability. Moreover, accurate absolute concentrations can be retrieved relying on a standard addition workflow. These preliminary results showcase the ability of d-DNP to provide highly resolved and hyperpolarized spectra of biofluids, thus opening promising application perspectives for both untargeted and targeted metabolomics.

POSTER 125
Across cities dDNP: design and performance of a compact He bath cryostat with NMR capability to transport hyperpolarized samples
Presenting Author: Andrea Capozzi

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Hyperpolarization via dissolution Dynamic Nuclear Polarization (dDNP) has the potential to revolutionize diagnostic radiology. Nevertheless, the methodology struggles to enter everyday clinical practice. One of the reasons why broad consensus among clinicians is still missing lies in the technical complexity that characterizes hyperpolarization via dDNP. Differently from PET, hyperpolarized (HP) MR contrast agents cannot be transported and have to be prepared on-site.
We developed a robust methodology to change this paradigm. We herein present our latest updates on transportable hyperpolarization technology. Combining non-persistend UV-induced radicals and purpose build hardware, we demonstrated the first "across-cities-dDNP" experiments. We hyperpolarized glucose in Copenhagen, transported it for 320 km, and performed HP-MRI at Aarhus University Hospital in a rat model.

POSTER 126
Overhauser Effect or Thermal Mixing or Something New
Presenting Author: Yifan Quan

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We propose a new mechanism for DNP that is different from OE, SE, CE and TM. We denote the mechanism as Resonant Mixing (RM). We believe that this mechanism is responsible for the observed dispersive shaped DNP field profile for trityl samples near the EPR center. This new effect is purely due to the mixing of states by the microwave field together with the hyperfine coupling. The theory is furthermore applied to treat the OE, providing an analytical solution. When the ZQ and DQ cross relaxations are different we obtain an absorptive shaped DNP field profile near the EPR center, i.e. OE, while when they are the same, we obtain a dispersive shaped DNP field profile, i.e. RM.

POSTER 127
Magic Angle Spinning EPR at 14 T
Presenting Author: Ilia Kaminker

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Dynamic Nuclear Polarization (DNP) is revolutionizing solid-state NMR spectroscopy by allowing for over a hundredfold signal enhancements. Most contemporary DNP experiments are performed under magic angle spinning (MAS), and considerable experimental and theoretical effort has been dedicated to describing electron-nuclear polarization transfer, being the most crucial part of DNP, under these conditions. While this polarization transfer has been extensively studied using EPR under static-DNP conditions, yielding valuable mechanistic insights, this process was never experimentally investigated using EPR under MAS. Here we describe the first EPR experiments performed on spinning samples at 14 T and 390 GHz. This development paves the way for experimental observation of polarization transfer under the relevant conditions, serving to better understand DNP and facilitate further developments.

POSTER 128
Efficient Polarization Re-distribution in Hyperpolarized Propane Produced Via Pairwise Parahydrogen Addition
Presenting Author: Nuwandi M. Ariyasingha

Complete Author List:
Nuwandi Ariyasingha (Wayne State University); Anna Samoilenko (Wayne State University); Shiraz Nantogma (Wayne State University); Oleg G. Salnikov (International Tomography Center SB RAS); Nikita V. Chukanov (International Tomography Center SB RAS); Igor V. Koptyug (International Tomography Center SB RAS); Eduard Chekmenev (Wayne State University)

Polarization redistribution between nascent parahydrogen-derived protons with other protons is studied in hyperpolarized propane using Parahydrogen Induced Polarization. We have synthesized site-selective isotopically labeled 3-D-propylene for our studies. The deuterium presence in HP propane breaks the magnetic equivalence of methyl protons, which resonate at different frequencies. Pairwise parahydrogen addition to 3-D-propylene leads to 1,2-addition of parahydrogen, which we confirm by detecting corresponding PASADENA spectra, when synthesis is performed in the weakly-coupled regime at 14 T. Next, we employ ALTADENA (strongly-coupled) regime for pairwise addition to confirm that parahydrogen-derived HP protons can spontaneous polarize the –CDH2 protons, which did not originate from parahydrogen. These findings improve our understanding about hyperpolarized symmetric small hydrocarbons.

POSTER 129
Relaxation Dynamics of Nuclear Long-Lived Spin States in Parahydrogen Hyperpolarized Butanes
Presenting Author: Anna Samoilenko

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We present a systematic relaxation dynamics study of hyperpolarized butane as a potential candidate for inhalable hyperpolarized contrast agent in human lung imaging. HP butane was prepared via pairwise parahydrogen addition using 1-butene and 2-butene and detected at high-filed and low-field for comparison. Heterogeneous pairwise parahydrogen addition to butene provides a simple and robust approach for creating HP butane in the gas phase with high signal enhancements (PH~1% at 1.4 T) in comparison with widely studied HP propane using Rh/TiO2 catalyst. Moreover, the lifetimes of HP butane in both high-field (3.5±0.1s at 3.7 atm) and low-field (5.8±0.1s at 3.7 atm) are greater compared to that of HP propane making HP butane an excellent candidate for human pulmonary imaging and beyond.

POSTER 130
Reversible NMR Hyperpolarization of 15N in Unmodified Amino Acids Unraveled at High Magnetic Field
Presenting Author: Ewoud Vaneeckhaute

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Amino acids (AA's) and ammonia are unique metabolic markers, exerting essential roles in the nitrogen metabolism and cell regulation across plants and humans. Parahydrogen, the singlet spin isomer of molecular hydrogen, stands out as an exceptionally competitive hyperpolarization agent to boost their signal intensity in NMR. Production of hyperpolarization fueled by p-H₂ relies on transition metal catalysis, rather than on physical principles, thus inducing chemically selectivity towards specific molecular targets, traditionally excluding unmodified AA's from its repertoire. Here, we present an elegant all-high-field approach to reversibly hyperpolarize ¹⁵N heteronuclei in free and catalyst-bound pristine alanine and ammonia while at the same time providing swift straightforward access to the molecular structure of the active hyperpolarization catalyst complexes.

POSTER 131
Multi-dimensional Mapping of SABRE-SHEATH 13C Hyperpolarization of [1-13C]pyruvate
Presenting Author: Isaiah Adelabu

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Isaiah Adelabu (Wayne State University); Shiraz Nantogma (Wayne State University); Thomas Theis (North Carolina State University); Boyd Goodson (Southern Illinois University); Eduard Chekmenev (Wayne State University)

NMR hyperpolarization boosts sensitivity of MRI by 4-6 orders of magnitude, therefore, enabling real-time metabolic imaging. HP [1-13C]pyruvate is the leading 13C HP contrast agent because of its central role in metabolic activities with elevated uptake in cancers and other diseases. Rapid hyperpolarization of [1-13C]pyruvate via SABRE technique has enabled polarization values of up to 15%. Here, we report on simultaneous pH and temperature mapping of SABRE hyperpolarization process of [1-13C]pyruvate demonstrating complex trends that are readily understood through 2D mapping. We find that temperature and pH modulate C-13 polarization as well as the exchange rates and chemistry of SABRE process, therefore, enabling new approaches to improve the efficiency of SABRE-SHEATH polarization process of [1-13C]pyruvate.

POSTER 132
Benchtop N-15 NMR Spectroscopy (1 T) of in situ Hyperpolarized Molecules with Natural Isotopic Abundance
Presenting Author: Danila Barskiy

Complete Author List:
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Analytical applications of benchtop NMR spectrometers could be expanded if robust hyperpolarization techniques were available for generating high degrees of
proton and heteronuclear spin polarization at natural isotopic abundance and without the need for sample shuttling. By using SABRE (Signal Amplification By Reversible Exchange) and optimized pulse sequences, we demonstrate NMR signals from biomolecules at mM concentrations in situ at 1 tesla (Figure 1). Since nuclear polarizations achieved in this way can exceed ten percent and measurements can be repeated multiple times, the sensitivity of benchtop-NMR detection is sufficient for measuring molecules at natural abundance of 15N nuclei. The presented methodology may find utility for the analysis of low-concentration chemicals using benchtop NMR spectroscopy with the aid of affordable hyperpolarization.

POSTER 133
Biomolecular Applications of Low-Field NMR using SABRE Hyperpolarization
Presenting Author: Christian Hilty

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Nuclear spin hyperpolarization changes the sensitivity equation in NMR by uncoupling spin polarization from the magnetic field. Thus, low-field detection under biologically relevant conditions becomes possible. Here, we demonstrate the use of SABRE hyperpolarization for the detection of biomolecular interactions, whereby polarization is generated and signals are detected at milli-Tesla magnetic fields and below. This mode of detection not only presents an opportunity for simplification and cost savings in routine NMR experiments such as for ligand screening, but also significantly expands the magnetic field range accessible for the measurement of molecular dynamics and related parameters.

POSTER 134
The Virtues of Stopping Dynamic Nuclear Polarization
Presenting Author: Sami Jannin

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In dissolution dynamic nuclear polarization experiments, the near unity polarization of unpaired electron spins is transferred to surrounding nuclear spins via microwave irradiation. By intermittently stopping DNP by simply gating the microwaves, one can restore the near-unity electron spin polarization within a fraction of a second. As the electron polarization gets back towards unity, the electron flip-flop probability vanishes, which has a dramatic effect on transverse nuclear relaxation and on nuclear spin diffusion. I will present here the virtues of intermittently stopping DNP by simple microwave gating, which enables us to perform more efficient cross-polarization, detect EPR line shapes without EPR instrumentation, measure electron spin-lattice relaxation, and microwave saturation time constants, and study nuclear spin diffusion of invisible nuclear spins.

POSTER 135
Hyperpolarization of Dopamine and its Application using Home-Built Parahydrogen Instrument
Presenting Author: Quy Son Luu

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Dopamine is not only associated with Parkinson's, Alzheimer's, and schizophrenia diseases but also a precursor to polydopamine (PDA) formation. The PDA is a well-known mussel-inspired adhesive for multifunctional surface modification. Here, we built the bubbling system with high pressure (3 - 5 atm) and controlled the bubbling time via infrared (IR) remote control with an Arduino. In addition, we study the tracking of dopamine and the mechanism of PDA formation through the 1H and 13C nuclear magnetic resonance (NMR)-based signal amplification by reversible exchange (SABRE) and SABRE in shield enables alignment transfer to heteronuclei (SABRE-SHEATH) technique.

POSTER 136
Hyperpolarized 29Si Magnetic Resonance Spectroscopy of Selectively Radical-Embedded Silica and α-Quartz Nanoparticles
Presenting Author: Thi Quynh Nguyen

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Silica nanoparticles exhibit favorable characteristics for development as 29Si MRI probe. To mitigate the inherently low sensitivity of 29Si MRI, Dynamic Nuclear Polarization (DNP) technique can be applied to greatly amplify the NMR signals. Here, the 29Si DNP hyperpolarization of silica nanoparticles with selectively radical embedding in core, shell, and entire particles, are discussed. These particles can self-polarize without external radical addition and owing to the distribution of radicals homogeneously inside the particles, significant enhanced 29Si hyperpolarization signal is achieved. However, the signal lifetime is relatively short due to the paramagnetic effect of embedded radicals, thus amorphous silica is converted to crystalline α-quartz structure to extend the T1 relaxation time and open the opportunity for in vivo 29Si MRI application.

POSTER 137
NMR at microTesla Fields for Hyperpolarization Applications
Presenting Author: Laurynas Dagys

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The obstacle of low sensitivity in NMR may be alleviated by enhancing typically weak signals by orders of magnitude with use of hyperpolarization techniques. In this work we present Para-Hydrogen Induced Polarization (PHIP) as an efficient and convenient method for versatile production of hyperpolarized targets. The method can be performed at different magnetic fields each providing different benefits, but we direct the focus to microTesla fields as a regime that provides design freedom in aspects such as scalability and robustness. We discuss how relaxation influences polarization transfer techniques and demonstrate that variety of methods can be conveniently applied at microTesla fields. These developments display the potential for widespread application of hyperpolarization using PHIP on many target molecules.

POSTER 138
Hyperpolarized solution-state NMR spectroscopy via intermolecular NOE with parahydrogen-polarized source molecules
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In the past year we have published and presented work showing significant solution-state NMR enhancement by using overwhelming source magnetization to cross relax with small target molecules in solution. This work was originally demonstrated using optically-polarized naphthalene 1H spins and shown to give enhancements up to 2600 (over 60 MHz), 50 (400 MHz), and 40 (600 MHz). In the current work we show our efforts to translate this idea to a parahydrogen-polarized source molecule to enable a more accessible, high throughput method. Primarily, we have observed that conversion of singlet-state polarization to usable magnetization under normal experimental conditions is physically limited and is linked to the dipolar field effect. We will discuss such obstacles and recent advancements in this presentation.

POSTER 139
Utilizing N@C60 as a Polarizing Agent: Electron-Decoupled MAS DNP
Presenting Author: Nicholas Alaniva

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Nitrogen endofullerene (N@C60) is a well-protected unpaired electron source that features extremely narrow electron resonances and long relaxation times. Here, frequency-chirped microwaves are used to decouple electron- and 13C-spins in magic-angle spinning (160 parts-per-million) N@C60:C60 powder, improving DNP-enhanced 13C NMR signal intensity by 12% for 7 s polarization, and 5% for 30 s polarization. This extension of electron decoupled MAS DNP beyond previously employed trityl radicals is a step toward utilizing N@C60 as a controllable electron-spin source for magic-angle spinning magnetic resonance experiments.

POSTER 140
Proton-only Detection of Hyperpolarized 13C2-pyruvate by S2M and R pulses
Presenting Author: Iuliia Mandzhieva

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Hyperpolarized NMR has been developed to create high degrees of nuclear spin polarization approaching order unity compared to a polarization of ∼10−5 at thermal equilibrium.
Pyruvate is the most promising HP MRI substrate agent because it plays a central role in vital metabolic pathways and could be used as a biomarker for various diseases. However, HP MRI detection requires specialized 13C capabilities, including installing expensive coils and the entire RF hardware chain of amplifiers for pulses and TR switches and spectrometers.
The combination of SABRE-SHEATH and S2M/S2M composite/R pulses create an opportunity for proton-only HP MRI with existing MRI scanners and may be particularly attractively when used with low-field MRI machines establishing an affordable molecular imaging platform.

POSTER 141
Catalyst and Methanol Free Injection of SABRE Hyperpolarized [1-13C]Pyruvate Detected In Vivo
Presenting Author: Keilian John MacCulloch

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First catalyst and methanol free solution of hyperpolarized pyruvate detected in vivo employing the Para-Hydrogen Induced Polarization (PHIP) modality known as Signal Amplification By Reversible Exchange (SABRE). SABRE is a simple, inexpensive, and fast hyperpolarization modality that directly hyperpolarizes pyruvate near room temperature. In this work, highly polarized [1-13C]pyruvate was generated in an ethanol/water mixture and then filtered through a C18 cartridge for catalyst removal prior to injection into a healthy Wistar rat. The hyperpolarized pyruvate signal was monitored in vivo with a dynamic pulse sequence in a 1.5 T MRI.

POSTER 142
Enabling the Broad Class of Alpha-Keto Acids as SABRE Hyperpolarization Targets and Exploring Their Polarization Dynamics
Presenting Author: Stephen McBride

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In this work, we expand the scope of SABRE substrates by employing SABRE-SHEATH and temperature cycling to hyperpolarize six different α-keto acids: [1-13C]pyruvate (PYV), α-ketoglutarate (AKG), oxaloacetic acid (OAA), phenylglyoxylic acid (PGA), phenylpyruvate (PPYV), and 2-oxobutyrate (2-OB). Additionally, we obtained T1 relaxation and polarization buildup measurements to develop a theoretical model to characterize SABRE polarization relaxation and buildup dynamics. In conclusion, several new α-keto acids were added to the SABRE substrate scope, and a mathematical model for the SABRE dynamics was refined to explain the hyperpolarization build-up and decay dynamics.

POSTER 143
Improving SABRE Polarization Through Three-Dimensional Magnetic Field Manipulation
Presenting Author: Shannon L Eriksson

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X SABRE is a rapidly developing heteronuclear hyperpolarization modality where μT magnetic fields along the leading axis are commonly used to facilitate population transfer between spin states of interest. Because the necessary fields are small, it is trivial to rapidly change the magnetic field in three dimensions, a degree of freedom that has yet to be explored. Here, we introduce pulse sequences developed using conventional magnetic resonance techniques like decoupling, as well as an arbitrary shaped pulse optimized using an evolutionary strategy. These pulse sequences protect singlet spin order in existing pulsed SABRE SHEATH techniques, facilitate direct measurement of the initial coherent dynamics in this complex system, and improve magnetization yields up to 7-fold and singlet yields up to 4-fold.

POSTER 144
Optimizing Parahydrogen-Enhanced 1H MRI Using a Clinical "Point-of-Care" Low-Field Scanner.
Presenting Author: Nadiya Iqbal

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When combined with hyperpolarization, low-field (LF) MRI has the potential to address many limitations presented by conventional MRI. We report on our continuing efforts to explore the potential for integrating a clinical low-field (64 mT) point-of-care MRI scanner with parahydrogen-based hyperpolarization. To increase the scanning speed, we have investigated imaging of SABRE-hyperpolarized substrates using a batch-mode approach where SABRE is performed in the fringe field, followed by rapid sample transfer to the scanner's head coil, boosting the signal to allow high-resolution images to be obtained in under 15 s. We are currently working on building a continuous flow system to enable uninterrupted SABRE 1H MRI to better enable optimization, as well as efforts to perform low-field MRI with HP propane gas.

POSTER 145
Data-constrained Determination of Applied Flip Angles to Improve Hyperpolarized 13C MR Kinetic Modeling in the Presence of Large B1 Variations Encountered in Abdominal Imaging
Presenting Author: Tanner Nickles

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A major challenge in the clinical translation of [1-13C]pyruvate hyperpolarized (HP) 13C MRI in human abdominal studies is B1 inhomogeneities across the large FOV that could confound metabolite-specific imaging methods. Given variations in B1, the exact applied flip angle in all voxels across the abdomen is often unknown, leading to errors in the quantification of metabolite conversion-rates. To overcome this limitation in abdominal studies, kinetic modeling within a numerical simulation regime was successfully used to determine the actual flip angle in the presence of variations in B1 without explicitly knowing it. In this study, the estimated flip angle approximated ground truth and <7% kPL bias was determined at both high and low metabolite conversion-rates.

POSTER 146
DNP Study of Crude Oil Suggests Several Competing DNP Mechanisms
Presenting Author: Timothy Keller

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The radical content in crude oil gives rise to several DNP mechanisms, most notably the Overhauser effect and solid effect. In this work, we provide evidence for a 3rd DNP mechanism in crude oil, the cross effect.
In recent work, we have performed high resolution DNP at low fields in liquids. This resolution has allowed us to distinguish the aliphatic and aromatic protons in the 1H NMR spectrum of crude oil.
We find that aromatic protons of crude oil exhibit larger enhancements than the aliphatic protons. At high microwave powers, we observe a decrease in the enhancement for many samples. We attribute this to an "over-saturation" effect which provides evidence for the cross effect DNP mechanism.

POSTER 147
Advances in SABRE hyperpolarization, including RASER detection, and first in-vivo demonstrations
Presenting Author: Thomas Theis

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Parahydrogen induced polarization (PHIP) is cherished for its relative simplicity and ease. Here we report on the most recent progress with hydrogenative and non-hydrogenative PHIP (aka SABRE). We report on three major advances, which include (a) SABRE-SHEATH (SABRE in Shield Enables Alignment Transfer to Heteronuclei) hyperpolarization of carbon-13 in many different alpha-keto acids, thereby significantly broadening the substrate scope (b) the first carbon-13 Radiofrequency Amplification By Stimulated Emission of Radiation (RASER) measurements exhibiting striking non-linear "quantum" detection thresholds and (c) the first in-vivo molecular imaging after SABRE-SHEATH hyperpolarization of [1-13C]pyruvate showing the detection of metabolic conversion of pyruvate to lactate, carbonate pyruvate-hydrate and alanine.

POSTER 148
Improved TinyPol Radicals for High-Field and Fast MAS DNP NMR
Presenting Author: Moreno Lelli

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Here we present the development of new water-soluble di-nitroxides biradicals, analogous to TinyPol, but with optimized molecular geometry, promoting high electron-electron (e-e) magnetic interaction, and incorporating also recent concepts such as the stereo-controlled conformation and efficient radical-solvent interaction.
These new systems show excellent performances, especially at high magnetic fields and fast MAS.
In particular, M-TinyPol(OH)4 shows enhancements up to about 200 even at 65 kHz of MAS frequency and 18.8 T. These systems provide high overall sensitivity that places them at the highest values among the polarizing agents developed in aqueous media at 18.8 T. The role of the structural improvements will be discussed, also with the aid of simulations.

POSTER 149
Investigation of LC-Photo-CIDNP Magnetic-Field Dependence of Molecules bearing a Quasi-Isolated Spin Pair via a Rapid Shuttle Field-Cycling Device
Presenting Author: Siyu Li

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Low-concentration photo-chemically induced dynamic nuclear polarization (LC-photo-CIDNP) is an emerging optically enhanced technique that leads to significant NMR sensitivity enhancements in solution. LC-photo-CIDNP requires solvent-exposed aromatic moieties, e.g., tryptophan and tyrosine. This study investigates the magnetic-field dependence of the 13C LC-photo-CIDNP of a Trp isotopolog (Trp-alpha-13C-beta,beta,2,4,5,6,7-d7) bearing a quasi-isolated 1Halpha-13Calpha spin pair. We employed a new rapid-shuttle field cycling device with in-situ side illumination and fast field-transition times. This setup enables rapidly shuttling NMR samples and maintaining excellent resonance lineshapes. Analysis of the magnetic-field dependence of LC-photo-CIDNP of Trp-alpha-13C-beta,beta,2,4,5,6,7-d7) led to identifying remarkable enhancement factors of ca. 1,000 at low illumination fields (ca. 50 MHz, 1.18T). This approach bears promise for the future detection of larger biomolecules (e.g., proteins).

POSTER 150
PHIP 13C Radiofrequency Amplification by Stimulated Emission of Radiation (RASER)
Presenting Author: Christopher Nelson

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Hyperpolarized Magnetic Resonance Imaging (HP MRI) is hailed as a next-generation molecular imaging modality. Because 13C resonate at ~4 times lower frequencies compared to protons, excitation of 13C spins is hardly possible with conventional clinical MRI scanners. In this work, we demonstrate a 13C RASER, detected without an excitation pulse, in HP ethyl [1-13C] acetate prepared via pairwise addition of parahydrogen to vinyl [1-13C] acetate and polarization transfer from the protons to the carbon-13 via magnetic field cycling. RASER signals were detected using a non-cryogenic 1.4T NMR spectrometer. RASER signals were observed for several minutes from a single sample, achieving 21mHz NMR linewidths. Our work demonstrates the feasibility of 13C RASER creation using a bolus of HP ethyl-[1-13C] acetate.

POSTER 151
15N SABRE-SHEATH and NMR/MS/DFT Characterization of Amino-Metronidazole, a Metabolic Product of the Antibiotic and Prospective Hypoxia Contrast Agent Metronidazole
Presenting Author: Ishani Senanayake

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The antibiotic metronidazole (MNZ) has gained interest as a potential MRI contrast agent for imaging hypoxia. SABRE-SHEATH can provide efficient hyperpolarization of 15N3-MNZ, but the envisioned MRI approach requires that MNZ will rapidly undergo structural changes in hypoxic environments with significant 15N frequency differences manifested in its downstream metabolic products. We have conducted computational (DFT) studies to predict 15N chemical shifts of different relevant species, as well as performed NMR studies of amino-MNZ (despite anticipated stability concerns). Direct hyperpolarization of naturally abundant 15N spins in amino-MNZ via SABRE-SHEATH (enhancement ~18,000), along with long-duration 1H-decoupled 15N NMR experiments, allowed comparison with both 15N3-MNZ and naturally abundant MNZ, showing significant 15N shift differences that showed good agreement with DFT predictions.

POSTER 152
Dissolution DNP on hydrophobic molecules using organic solvents opens new perspectives for the study of complex organic mixtures
Presenting Author: Chloé Gioiosa

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Dissolution DNP has proven to be a very powerful hyperpolarization technique enabling exceptional increase in sensitivity in liquid state NMR. Over the last two decades, numerous applications of dDNP have emerged in fields of research such as metabolomics, biochemistry or imaging on rather aqueous matrices. It is however yet to be widely developed in fields such as the energy industry where hydrophobic matrices need to be probed. Here, we present our first d-DNP results on 13C labelled benzaldehyde prepared in a toluene glassing matrix, hyperpolarized through cross-polarization on a Bruker prototype DNP polarizer, and rapidly dissolved with isopropanol and injected in a 14T spectrometer. These first experiments allowed us to reach signal enhancement over 70 000 with 11% 13C polarization.

POSTER 153
SABRE Hyperpolarization of Cytosine and other Nucleobases
Presenting Author: Max Gemeinhardt

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A number of DNA nucleobases were polarized by an efficient and comparatively low-cost methodology, signal amplification by reversible exchange (SABRE) and its heteronuclear extension, SABRE in Shield Enables Alignment Transfer to Heteronuclei (SABRE-SHEATH). For example, SABRE hyperpolarization of natural abundance 3-methyladenine yielded 15N signal enhancement of ~3,300 at 9.4 T. The same methodology afforded 15N and 13C signal enhancements of doubly labeled cytosine of up to ~240-fold and ~50-fold, respectively. The hyperpolarization-enhanced spectra of the different nucleobases can provide insight into the presence of different tautomers and their respective ability to support SABRE processes. In turn, this information can potentially help to select and successfully hyperpolarize novel biologically relevant molecular targets for various envisioned applications.

POSTER 154
Fast Nanomolar Detection and Fragment Screening on a Benchtop NMR Spectrometer Boosted by Photo-Induced Hyperpolarization
Presenting Author: Gabriela Stadler

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The sensitivity limitation of NMR spectroscopy can be overcome by photo-chemically induced dynamic nuclear polarization (CIDNP). The expansion of the method's chemical space is shown with the design of a 212-compound fragment library and its screening against PIN1 at 600 MHz. With single-scan experiments of a few seconds using low micromolar concentrations, we reach an unprecedented screening rate of 1500 samples per day with our new flowthrough system. Moreover, photo-CIDNP spectra are acquired within 3 minutes on a cryogen-free 80 MHz benchtop spectrometer, which is demonstrated by a miniscreen with 28 fragments. We reached a detection limit of 100 nM in only 3 minutes on the benchtop spectrometer, while without hyperpolarization no signal was detected after days of continuous measurement.

POSTER 155
The Emergence of Efficient DNP in Trityl-Based Multiradicals at Sub-Nanometer Electron-Electron Distances
Presenting Author: Raj Chaklashiya

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Multi-Electron Dynamic Nuclear Polarization (ME-DNP) has enormous potential for quantum sensing of nuclei through enhancement of NMR signal, but its current implementations are through radical clustering, a stochastic process that is difficult to control. A fine-tuned multiple-electron geometry with strong and asymmetric coupling would provide precise control over quantum coherences and polarization transfer for optimal ME-DNP. Here we share both our experimental and theoretical results on how to achieve controllable 1H ME-DNP in a designed electron geometry: a trityl-based tetra-radical. Our findings give us the design parameters necessary for electron geometries to achieve controllable ME-DNP for quantum sensing.

POSTER 156
SABRE Hyperpolarization with up to 200 bar Parahydrogen in Standard and Quickly Removable Solvents
Presenting Author: Sören Lehmkuhl

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In this work, we report on SABRE hyperpolarization up to 200 bar in standard and quickly removable solvents. We employ a recently introduced low-cost, versatile high-pressure setup, which enables spectroscopy measurements with a compact NMR magnet. With this setup, we achieved 2% SABRE polarization at a substrate concentration of 60 mmol/l, equal to a molar polarization of 1.2 mmol/l. Additionally, SABRE hyperpolarization in liquefied ethane and compressed CO2 at 200 bar was demonstrated. Eliminating standard SABRE organic solvents such as methanol in hyperpolarization techniques is a prerequisite for molecular medical research.

POSTER 157
Hyperpolarized Liquid-State 13C NMR Signals from 1H Dissolution-Dynamic Nuclear Polarization via INEPT Transfer
Presenting Author: James Tolchard

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The low sensitivity of NMR spectroscopy and imaging can be boosted by factors approaching ~10000x with hyperpolarization methodologies, such as dissolution-dynamic nuclear polarization. In small molecules, 13C-spins are typically polarized either directly, which is slow and yields low polarizations, or indirectly by pulse sequences such as cross-polarization, which is efficient but often unavailable. Here, we present the hyperpolarization of 1H-spins, which polarize rapidly and to a higher extent, before dissolution and a subsequent refocussed INEPT transfer to 13C polarization. Importantly, we exploit the minimal transfer time between our polarizing instrument and spectrometer (~1.7s) to overcome short 1H relaxation times. We will present our hardware setup, 13C polarization recoverable from solid-state 1H hyperpolarization, and simulations aiding the interpretation of 13C lineshapes.

POSTER 158
Hyperpolarization of 15N Betaine
Presenting Author: Magnus Karlsson

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NMR signals can be enhanced by several orders of magnitude with hyperpolarization techniques. A limitation of hyperpolarization is the lifetime of the signal; The signal created by hyperpolarization will decay towards the thermal level with the T1 of the nucleus. Hence, hyperpolarization molecules with long T1 nuclei are much preferred. Quaternary ammonium compounds can have long 15N relaxation time constants with several examples in the > 5 minutes range. Here we present results from experiments with one such compound: 15N labeled betaine.

POSTER 159
The Steady-State PHIP RASER: Generating a Continuous NMR Signal
Presenting Author: Jing Yang

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RASERs (Radio Amplification by Stimulated Emission of Radiation) allow to measure high-precision NMR spectra as well as study nonlinear phenomena. To operate a RASER, a population inversion is required, which can generated by hyperpolarization methods. With parahydrogen fueled RASERs, various new use-cases were unveiled in the recent years, all based on RASERs with multiple frequencies. However, parahydrogen fueled RASERs operating at high magnetic fields are burdened by the parahydrogen pumping itself.
In this work, we report on a steady-state multimode RASERs operating in different regimes pumped by hyperpolarized ethyl acetate. We demonstrate operating regimes dominated by five different scenarios depending on the population inversion: a starting RASER, the "normal NMR" two-mode RASER, frequency combs, period doublings, and chaos.

POSTER 160
Ultra-low Temperature Overhauser Dynamic Nuclear Polarization
Presenting Author: Scott A. Southern

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Overhauser DNP is possible in insulating solids when dynamics can result in the modulation of hyperfine couplings. We discovered that the methyl functionalization of the Blatter radical could activate the Overhauser Effect (OE), resulting from the methyl group dynamics modulating the hyperfine coupling to its protons, providing a mechanism for intramolecular cross-relaxation.
We predicted the free energy barrier for methyl rotation to define an approximate rovibrational wavefunction describing the dynamics, enabling us to predict the temperature dependence of the cross-relaxation.
Ultra-low temperature MAS-DNP experiments were used to study the mechanism of methyl-driven OE DNP. We observed increasing OE performance with decreasing temperature for CH_3 and CD_3 radicals, suggesting that at very low temperatures, librations are the dominant source of cross-relaxation.

POSTER 161
Clustering of P1 centers, and by proxy NV centers, observed by DNP and EPR
Presenting Author: Santiago Bussandri

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Through a collaborative study, we have found that P1 centers are clustering in more significant ways than previously anticipated using DNP profile measurements and EPR methods at room temperature and high field. Understanding the spatial distribution of P1 centers is critical because it directly reflects on the distribution of NV centers, which are essential tools for quantum information sensing. By decomposing the 7T DNP profile, we were able to identify different electronic spin populations and their respective EPR and DNP properties. ELDOR spectroscopy measurements and 13C DNP build-up experiments demonstrated the presence of a broad clustered species, in two common types of diamonds used widely in the community. These findings provide valuable insights for the development of advanced quantum technologies.

POSTER 162
Following Expected and Unexpected Molecular Interactions of Cucurbit[6]uril in a Molecular Relay with HyperCEST NMR
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Cucurbit[6]uril (CB6) and cucurbit[7]uril (CB7) are macrocyclic hosts that bind to different ends of the two-faced guest (TFG) AMADA-Put. Their cavities and portals can also be occupied by other competing guests and the details of the TFG transition from CB6 to CB7 were not yet fully known. Here, we investigate this transition using Xe-129 as a hyperpolarized monoatomic guest that is very sensitive to perturbations of the accessibility of CB6. We analyze anticipated and unexpected changes in saturation transfer responses for HyperCEST z-spectra that give insights into the rearrangements at the CB6 cavity. These insights should be included into the overall promiscuous binding behavior of CBs in various applications.

POSTER 163
Optimization of laser-induced OH• Radical Generation in Water
Presenting Author: Leon M. Geiger

Complete Author List:
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The use of radicals to transfer spin polarization to nuclei is an important technique to increase the signal intensity of NMR measurements. The classical methods require several tens of minutes and therefore suffer from the degradation of radical concentration during the generation process when spin-traps like DMPO are used. By using high-power pulsed laser systems, we can generate more radicals in a shorter timescale compared to the UV-generation method. Additional advantages are that only water is required to form OH• instead of hydrogen peroxide, and there is potential to locally generate radicals for in situ applications. From the EPR spectroscopy, the amount of bonded OH• radicals were calculated. Together with the number of spins, the DMPO capture efficiency was determined.

POSTER 164
Sustainable and cost-effective MAS DNP at 30 K with cryogenic sample exchange
Presenting Author: Gaël DE PAËPE

Complete Author List:
Subhradip Paul (CEA / Univ. Grenoble Alpes); Eric Bouleau (CEA / Univ. Grenoble Alpes); Quentin Reynard-Feytis (CEA / Univ. Grenoble Alpes); Jean-Pierre Arnaud (CEA / Univ. Grenoble Alpes); Christian Reiter (Bruker Biospin); Frank Engelke (Bruker Biospin); Armin Purea (Bruker Biospin); Sabine Hediger (CEA / Univ. Grenoble Alpes / CNRS); Gal De Pape (CEA / Univ. Grenoble Alpes)

We present a home built setup to perform sustainable and cost-effective fast Helium MAS DNP at 30 K with cryogenic sample exchange capability. Using cAsymPol-POK, a newly introduced polarizing agent for DNP, we report large signal-to-noise improvement for proton-dense methyl-containing organic powdered samples that are difficult to polarize at 100 K (currently the lowest temperature accessible in most DNP labs).

POSTER 165
Hyperpolarized glucose for non-invasive In-Cell measurements of glycolytic bottlenecks
Presenting Author: Pernille Rose Jensen

Complete Author List:
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The control and response of metabolic networks is still incompletely understood, even in highly studied model organisms. Without counterpart experimental data, computational mechanistic models remain hard to probe for their predictive value in vivo. Direct real-time measurements of metabolic flux can be obtained in cellular systems with 13C NMR using dissolution upon dynamic nuclear polarization in the solid state (dDNP-NMR). Here, we show recent strides in improving dDNP-NMR assays to include detection of the metabolites from upper part of the glycolysis, which have been shown as key steps in control of glycolytic flux.

POSTER 166
Overhauser DNP in Supercritical Ethane at High Magnetic Field
Presenting Author: thierry dubroca

Complete Author List:
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DNP in liquids is challenging, particularly at high magnetic fields, where resolution is needed to solve scientific questions. We present here a large sample volume pressure cell combined with a double resonance (13C-1H) liquid DNP probe capable of delivering significant microwave power at the sample operating at 150-600MHz (i.e.14.1T). This newly developed cell is applied to perform scalar Overhauser DNP with 13CCl4 and 13CHCl3 in supercritical ethane. The low viscosity fluid promotes short correlation times which increase the DNP efficiency. Efforts are on the way to increase the pressure range of the cell to accommodate a wide variety of supercritical fluids with the ultimate goal of being able to perform 1H ODNP at high magnetic fields.

POSTER 167
DNP-Enhanced NMR Analyses of Surface Group Interactions & Distributions on Colloidal Silica Particles
Presenting Author: Matthew Lertola

Complete Author List:
Matthew Lertola (University of California, Santa Barbara); Michael Schmithorst (University of California, Santa Barbara); Bradley F. Chmelka (University of California, Santa Barbara)

The surface functionalization of colloidal silica particles is a key determining factor of their macroscopic properties, including colloidal stability. Solid-state NMR is an important tool for understanding silica surface chemistry at the atomic level, which requires surface-selective techniques with enhanced sensitivity, especially for dilute loadings of covalently grafted organic groups. Here, we use solid-state DNP NMR methods to establish the surface distributions of two types of organic groups on silica nanoparticles. In particular, solid-state 2D DNP-enhanced 13C{1H} heteronuclear correlation (HETCOR) NMR analyses of freeze-dried silica nanoparticles reveal differences in the extents of commingling of surface organic groups between samples, which explain differences in their respective stabilities as colloidal suspensions.

POSTER 168
Investigation of DNP Mechanisms
Presenting Author: Ravi Shankar Palani

Complete Author List:
Ravi Shankar Palani (Postdoc, MIT); Michael Mardini (MIT); Yifan Quan (MIT); Robert G. Griffin (Massachusetts Institute of Technology)

Dynamic Nuclear Polarization (DNP) overcomes the issue of low sensitivity that Nuclear Magnetic Resonance (NMR) suffers from. The mechanism at play is determined by a host of factors including, but not limited to, the nature of the radical and microwave irradiation frequency. In this work, we discuss methods to investigate finer details of the underlying DNP mechanism and the polarization pathway in BDPA family of radicals and trityls. In BDPA our investigation led to designing Phe-d5-BDPA that specifically attenuates the DQ cross-relaxation pathway, improving enhancment by 50%. In trityls, we discuss the plausibility of thermal-mixing and characterize the mechanism under different conditions. We also investigate the competition between simultaneously active DNP mechanisms with multitone microwave irradiation and observe interesting spin-physics.

INSTRUMENTATION (Posters 169 – 193)

POSTER 169
Stripline Lenz lens add-on with one order of magnitude signal enhancement
Presenting Author: Jianyi Liang

Complete Author List:
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A Lenz lens is an electrically passive metallic radiofrequency interposer placed between a sample and a tuned or untuned nuclear magnetic resonance (NMR) detector. Its utility is to focus the B1 field of the detector onto a smaller sample space. Here we explore a novel embodiment of the Lenz lens, which acts as a non-resonant stripline interposer, i.e., the B 1 -field acts along the longitudinal volume of a sample container, such as a capillary or other microfluidic channel that is coincident with the axis of the stripline. The results show an enhancement up to one order of magnitude for a broadband application, i.e. from 130MHz to 500MHz.

POSTER 170
Automation in solid state NMR
Presenting Author: Jochem Struppe

Complete Author List:
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Herein we show a new strategy for automated experiment setup by using radio frequency reference fields, provided in lookup tables to automatically calculate rf pulses for excitation or recoupling, spinlock fields during cross polarization or decoupling fields for homo- or heteronuclear spin decoupling. This approach permits controlling the maximum decoupling field through simple field parameters, available in lookup tables, instead of abstract, machine oriented rf-power values. Often, these fields are synchronized with or have specific relationships to the magic angle spinning frequency. The approach permits easy experiment setup for any CPMAS experiment, whether it is a simple basic CPMAS experiment or a more complicated NMR experiments like any high-frequency MAS experiments with rotation rates of 111 kHz and above.

POSTER 171
Cryogen-free 9.4 T Solid state MAS NMR system
Presenting Author: Eugeny Kryukov

Complete Author List:
Eugeny Kryukov (Cryogenic Ltd); Denis Langlais (Cryogenic Ltd.); Dinu Iuga (Warwick University); Alexander Karabanov (Cryogenic Ltd); Paul Jonsen (Talaverascience); Rupert Reckless (Cryogenic Ltd.); Jeremy Good (Cryogenic Ltd.)

The ongoing crisis with liquid helium supply is getting more severe. We offer dry superconducting magnets based on cryogen-free cold heads that can replace the conventional magnets up to of 750 MHz 1H frequency. Our magnets are not only free from liquid cryogens, they are very compact and allow for probe insertion from the bottom or from the top of the magnet. The field value can be easily changed and made stable in an hour after the field ramp. This feature makes it possible to use the same magnet at many fixed fields or in a field sweep mode.
All of the above features were experimentally demonstrated on our in house 9.4 T MAS NMR system.

POSTER 172
Spin Echoes, Adiabatic Pulses, With Fantastic Sensitivity: Can Your Probe Do This?
Presenting Author: Paul Ellis

Complete Author List:
Paul Ellis (Doty Scientific); Daniel Arcos (Doty Scientific); F David Doty (Doty Scientific)

We have examined the performance of a standard Doty 4 mm MAS probe utilizing adiabatic (WURST) pulses. The performance of the probe demonstrated an excellent bandwidth and sensitivity. To accomplish these objectives, we have used two spin echo methods. The details of these methods are outlined within this presentation. We applied the spin echoes to three different samples, 119Sn in the form of SnO, 79Br as KBr, and 25Mg as Mg(O2CH)2•2H2O. The latter two samples are quadrupolar in nature with spins of 3/2 and 5/2, respectively. All the samples were at natural abundance for the spin systems of interest.

POSTER 173
Advances in Spherical Rotor DNP
Presenting Author: Lauren Price

Complete Author List:
Lauren Price (ETH Zurich); Nicholas Alaniva (ETH Zrich); Marthe Millen (ETH Zrich); Till Epprecht (ETH Zrich); Michael Urban (ETH Zrich); Alexander Dpp (ETH Zrich); Alexander Barnes (ETH Zrich)

Spherical rotors have many advantages for MAS DNP but the current 3D printed plastic stators are unable to withstand the cryogenic temperatures required. Here we introduce a probe assembly designed for spherical rotors at cryogenic temperatures. This system was used to successfully perform the first MAS DNP using spherical rotors. Using 9.5 mm spherical rotors, a maximum DNP enhancement of 256 on a sample of 20 mM AMUPol, 4 M 13C 15N Urea in 60/30/10 d-8 glycerol/D2O/H2O was obtained. The sample was spinning at a frequency of 2 kHz and a temperature of 107 K. This system is also being scaled for 6 mm spherical rotors which will increase spinning frequency to above 5 kHz.

POSTER 174
NMR Spectroscopy of Nanomole Amount Samples on a Homebuilt 2.1 Tesla NMR Magnet Using a Microsolenoid
Presenting Author: Sander Baas

Complete Author List:
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The past decades have brought increasingly strong magnetic fields to NMR, in order to improve resolution and sensitivity, with the current highest commercial field strength of 28.2 Tesla.. In recent years however, there has been a renewed interest in NMR at fields of several Tesla, making use of more affordable permanent magnets to produce the B0 field. We present a compact, cheap, homebuilt 2.1 Tesla NdFeB permanent magnet system for NMR spectroscopy. Sample handling occurs via a capillary-based microfluidic probe, with a microsolenoid transceiver coil. Currently the magnet system is operated in non-shimmed mode, with a movable probe stage for sample positioning. Coupled with hyperpolarization, samples amounts in the lower nanomole range (mM concentration) can be detected within a minute

POSTER 175
Advanced Integration of Batch-Mode Clinical-Scale SEOP Xenon-129 Generation-3 Hyperpolarizer
Presenting Author: Clementinah Oladun

Complete Author List:
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Hyperpolarized 129Xe gas is a revolutionary MRI contrast agent that has been recently FDA approved for clinical use. Hyperpolarized 129Xe is produced via spin-exchange optical pumping. Our clinical-scale batch-mode generation-3 SEOP hyperpolarizer employs batch-mode production, as well as in-situ polarimetry of Rb electron and 129Xe nuclear spin polarization. Here, we demonstrate next-generation advanced automation and systems integration embodied by our 129Xe hyperpolarizer. NMR and NIR polarimetry are performed in real time in a fully automated fashion using an ARM-based SMT32 microcontroller and a purpose-built low-field NMR spectrometer and a NIR spectrometer without any other devices. The resulting real-time 129Xe nuclear- and Rb electron polarization values are employed for auto-calibration and HP 129Xe production quality monitoring.

POSTER 176
Ball-Shift Automation to Achieve Reproducible Mapping of Transceiver Coils
Presenting Author: Jose Luis Uribe

Complete Author List:
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Achieving homogenous radiofrequency (rf) magnetic fields in solid-state NMR transceiver coils is essential for maximizing sensitivity during experimentation. Manual methods to measure coil homogeneity successfully and accurately have been a time-consuming and error-prone task, specifically with assessing spatial accuracy. An automated method that uses inexpensive and open-source equipment to create a modular, yet specialized tool, is presented, the Auto-Ball Shift (ABS). This mechanical apparatus is fully controlled by an Arduino UNO; designating pins, controlling direction, rotation speed, etc. The addition of an A4988 microstepper allows for precise fine

increments as low as 0.05mm. An automatized data collection scheme that uses Python scripting is used. This makes for a hands-free method which returns parsed rf coil data ready for mapping.

POSTER 177
Room-Temperature Overhauser DNP with Microwave Powers of Just 100's of Milliwatts at 7 T
Presenting Author: Alexander A. Nevzorov

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Currently, performing DNP at >200 GHz involves expensive gyrotrons. We demonstrate that all-dielectric photonic band-gap resonators (PBGR) make it possible to use compact, frequency-agile solid-state mm-wave sources with output powers of <400 mW to achieve appreciable solid and Overhauser DNP effects. We report on room-temperature Overhauser DNP gains of about 50 for 1M PhP3 and 100mM BDPA co-dissolved in d-toluene. Anodic Aluminum Oxide nanopores were employed as holders for creating thin, flat liquid samples to enhance the resonator performance. The results demonstrate the ability of a tuned resonator to effectively concentrate mm-waves for both liquid and solid samples. A dramatic increase in the PBGR Q-factor up to >1,000 can be achieved by utilizing full-defect photonic crystals. Supported by NIH 1R01GM130821.

POSTER 178
Development of a Novel 13C HTS Probe at 21.1 T
Presenting Author: Arthur S Edison

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Direct observation of 13C has many advantages in biological solution NMR spectroscopy. The primary limitation is the relatively low sensitivity of 13C compared to 1H. We report a new 13C HTS probe at 21.1 T with a novel 3-mm x 6.2-mm rectangular tube geometry. The volume of the rectangular sample tube is about 425 uL, allowing us to develop applications that were previously impractical with a smaller volume probe. We also have the option of using a 3-mm standard cylindrical tube with a reduced volume. The overall mass sensitivity is between about 2-3 times greater than a commercial 5-mm TXO cryogenic probe on the same instrument, depending on the sample. Data from urine and other biological samples will be shown.

POSTER 179
Improving Low (<100 mT) and Ultra-low (<10 mT) Field RF Fidelity with Active Transmit/Receive Switches and Q-Switching for Quantitative Magnetic Resonance
Presenting Author: Karl Stupic

Complete Author List:
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Low and Ultra-low magnetic field technology has seen expanded use in recent years for deployment as point-of-care systems for healthcare. As these systems continue to find further applications, issues such as phase stability and RF pulse fidelity at low RF frequencies is important to consider for quantitative results. Presented here are active switches utilized for both transmit/receive switches as well as for Q switching with the needed stability. These active switching systems are necessary as common passive switch designs such as crossed diodes can present phase instability due to low power requirements at low RF frequencies. Additionally, quality factor (Q) of RF coils plays a large role in pulse shape at low frequencies leading to added uncertainty in quantitative measurements.

POSTER 180
A Portable and Low-Cost MRI System Using an Impedance-Mismatched Front-End and SoC-Based Controller
Presenting Author: Soumyajit Mandal

Complete Author List:
David Ariando (University of Florida); Soumyajit Mandal (Case Western Reserve University)

In low-field NMR applications, such as emerging portable MRI devices, the benefit of impedance matching are not apparent, since reflections become insignificant at low frequencies (<10 MHz). However, the use of matched probes and transceivers is still standard practice at low frequencies (100 kHz-10 MHz), except for the use of audio transceivers at extremely low frequencies (<100 kHz). This work provides an alternative by using tuned but impedance-mismatched circuits for both signal transmission and reception and highlighting its benefits for portable MRI. A compact battery-powered pulsed-mode gradient driver is also described. The B0 field is generated by a low-cost Halbach array assembled from Ferrite magnets and 3D-printed holder. The system controller is an SoC-FPGA supporting autonomous measurement operations.

POSTER 181
Single-sided magnetic resonance sensor as a pre-clinical platform for analysis of complex tissue phantoms
Presenting Author: Sydney Sherman

Complete Author List:
Sydney Sherman (MIT); Alexa Zammit (MIT); Amena Khatun (MIT); Michael Cima (MIT)

Tissue microstructure can be indicative of pathology. The purpose of this work is to construct a portable permanent magnet array and RF coil sensor capable of acquiring signal from muscle tissue in clinical settings and differentiating microstructural differences. We have developed a portable, single-sided magnetic resonance sensor. The homogeneous region, B0 = 0.2T, is 12-15mm above the surface of the RF coil which allows for the clinical assessment of muscle tissue. Multi-compartment muscle and fat tissue phantoms were fabricated to have varying microstructural properties. A CPMG pulse sequence is used to acquire T2 relaxation metrics and diffusion weighted T2 signal. Microstructural differences in droplet size and distribution of phantoms does not affect T2 relaxometry data, but does affect diffusion-weighted T2.

POSTER 182
The Network for Advanced NMR: The Knowledgebase Progress
Presenting Author: Songlin Wang

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The mission of NAN, the Network for Advanced NMR, is to provide state-of-the-art NMR instrumentation to investigators in the US and abroad to advance relevant areas of science. The development of the NMR Knowledgebase (KB) in the areas of solution structural biology, metabolomics, biological solid-state NMR, and materials solid-state NMR is a part of the NAN mission, which includes writing standard protocols, standardizing regularly used pulse sequences, acquiring example datasets, and providing data processing templates. Here we present solid-state NMR KB content development to highlight key components of the KB and its application in the areas of biological and materials NMR.

POSTER 183
Development of Simplified Oxygenated System for in Vivo Solution State NMR
Presenting Author: Peter Costa

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The development of flow systems is critical for maintaining aquatic organisms for environmental toxicology, thus allowing for exposure studies in their native and unaltered state for in vivo solution state NMR. Current approaches use flow lines that have the potential to burst and clog, require large volumes of media, and multiple expensive HPLC pumps. The proposed "bubble pump" provides several unique advantages such as no clogging, small and constant solution volume, and no need for pumps or fluid lines in and out of the probe. The flow system is designed for in vivo solution state NMR experiments, for both large and smaller diameter probes with potential applications in toxicity and metabolomics-based research.

POSTER 184
Exploring the Potential of Broadband Complementary Metal Oxide Semiconductor Micro-Coil Nuclear Magnetic Resonance for Environmental Research
Presenting Author: Daniel Lysak

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Daniel Lysak (University of Toronto); Marco Grisi (Annaida Technologies SA); Kathryn Marable (Annaida Technologies); Carl Michal (UBC); William Wolff (University of Toronto); Peter Costa (University of Toronto); Katelyn Downey (University of Toronto); Kiera Ronda (University of Toronto Scarborough); Katrina Steiner (University of Toronto); Ronald Soong (University of Toronto); Andre Simpson (University of Toronto)

With sensitivity being the Achilles' heel of nuclear magnetic resonance (NMR), the superior mass sensitivity offered by micro-coils can be an excellent choice for tiny, mass limited samples such as eggs and small organisms. Here, the potential of broadband complementary metal oxide semiconductor (CMOS) based micro-coils for environmental research is investigated. Numerous heteronuclei such as 7Li, 11B, 13C, 19F, 23Na, 31P and 81Br were detectable and 13C and 19F were used to study two realistic environmental samples: a sprouting broccoli seed and a single Daphnia magna egg. The D. magna egg was exposed to hexafluorobenzene, and the contaminant was monitored within the egg by 19F NMR. Overall, broadband CMOS microcoils are shown to have significant potential for environmental research.

POSTER 185
Simplifying triple resonance experiment for high quality NMR spectra with Multi Frequency Drive System
Presenting Author: Hiroaki Sasakawa

Complete Author List:
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Organic compounds with phosphorus and boron nuclei often exhibit spectral complexity and reduced sensitivity in NMR analysis due to J couplings between hydrogen and carbon with these nuclei. We developed a triple resonance system called Multi Frequency Drive System (MFDS) to address this issue, enabling triple resonance experiments with a standard 2-channel NMR system. Using a JEOL JNM-ECZL600G spectrometer equipped with the ROYALPROBE™ P+, we conducted various solution NMR measurements. We present examples of signal enhancement and spectrum simplification achieved by triple resonance measurements of 1H, 31P, and 11B collected with a 2-channel NMR instrument.

POSTER 186
Development and applications of a 1.01 GHz (23.7 T) NMR system
Presenting Author: Yoshitaka Ishii

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We discuss development of an ultra-compact 1.01 GHz NMR magnet, and its preliminary NMR applications. The new ultra-compact 1GHz NMR magnet utilizes high-temperature superconducting (HTS) coils made of bismuth-based cuprates besides conventional low-temperature superconducting coils. Because of the high current density of the HTS coil, the magnet weighs only 1.6 tons and its footprint is the smallest among the existing 1 GHz NMR systems. The cryogenic refrigerator mounted on the magnet eliminates needs of regular liquid-helium refilling. We have successfully collected multi-dimensional solution NMR and solid-state NMR data for proteins at a 1H frequency of 1.01 GHz. The quality of the NMR data and other research progress from the ongoing project to develop 1.3 GHz NMR will be also discussed.

POSTER 187
SABRE Hyperpolarization and In Situ 1H NMR Signal Detection via Large Custom-Made Solenoid Coils
Presenting Author: Roman V. Shchepin

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Garrett L. Wibbels (South Dakota School of Mines & Technology); Clementinah Oladun (Wayne State University); Tanner Y. O'Hara (South Dakota School of Mines & Technology); Isaiah Adelabu (Wayne State University); Joshua E. Robinson (South Dakota School of Mines & Technology); Firoz Ahmed (Wayne State University); Zachary T. Bender (South Dakota School of Mines & Technology); Boyd Goodson (Southern Illinois University); W. Michael Snow (Indiana University Bloomington); Eduard Chekmenev (Wayne State University); Roman Shchepin (South Dakota School of Mines & Technology)

Hyperpolarization techniques require equipment with starting "price tag" of thousands of dollars in case of parahydrogen-based methodology (e.g. SABRE) and often exceeding millions in case of d-DNP, creating a substantial entrance barrier for many researchers. To tackle this issue, we develop a custom coil winding machine, which allowed us to prepare a number of solenoid magnet coils with high degree of magnetic field homogeneity. One of the solenoid coils was used (8 mT static field) in a 1H SABRE experiment, allowing signal detection at both external 1.4T magnetic field (tabletop NMR) as well as in situ when it is attached to low-field NMR spectrometer bringing the total cost of parahydrogen setup within few thousands of dollars!

POSTER 188
Control of Bruker NMR Spectrometers from Python with KovriginNMR
Presenting Author: Evgenii Kovrigin

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Evgenii Kovrigin (University of Notre Dame)

An object-oriented Python software package, KovriginNMR, has been designed to enable Bruker spectrometer control from Topspin Python interface. KovriginNMR is intended to help NMR users construct workflows incorporating multiple samples, experiments, as well as variable-temperature operation. A user is able to automate as much or as little of their workflow as desired, and the software may be started from user's own account (no need for NMR superuser privileges). KovriginNMR automatically logs events taking place during its operation and includes time stamps, sample and experiment names, data paths, thus assisting accurate record-keeping. The KovriginNMR Workflows module supports development of complex experimental routines involving multiple samples and experiments while the KovriginNMR VT module enables automated NMR measurements in a broad temperature range.

POSTER 189
Cryogen Reclamation System for NMR Magnets
Presenting Author: Takuya Matsumoto

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NMR magnets are usually cooled by two kinds of cryogens, ie.liquid helium and liquid nitrogen. The boil-off rates of the cryogen in general NMR magnets are typically around 20 cc/h for liquid helium and 200 cc/h for liquid nitrogen. We have developed new cryogen reclamation system that can greatly suppress the evaporation both of liquid helium and liquid nitrogen.
The system was tested with an NMR magnet, and it was confirmed that the noise generated by system vibration was at a level that would not interfere with NMR measurements. It has also confirmed that the magnet maintained stable zero boil-off status for more than 6 months.

POSTER 190
Fabricated Coplanar Waveguide Integrated with Microfluidics for Use in Nitrogen-Vacancy (NV) NMR
Presenting Author: Emma Huckestein

Complete Author List:
Emma Huckestein (University of Maryland); Johannes Cremer (University of Maryland); John Blanchard (Quantum Technology Center); Stephen DeVience (Quantum Technology Center); Ronald Walsworth (Quantum Technology Center); Declan Daly (University of Maryland)

Nitrogen-Vacancy (NV) centers have emerged as promising quantum sensors of local magnetic environments due to their spin-state dependent fluorescence, optical addressability, and room temperature behavior. In recent years, many pioneering works have demonstrated NV-based NMR, but have confined their experiments to bias fields less than 0.1 T due to engineering challenges associated with microwave delivery. In this work, we fabricate a coplanar waveguide with integrated microfluidics that homogeneously delivers microwaves with frequencies up to 6 GHz and with 15 – 30 MHz power.

POSTER 191
A Practical Approach to Passive Shimming of NMR Magnets
Presenting Author: Ilya Litvak

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ilya litvak (National High Magnetic Laboratory, Florida State University)

Shim systems supplied with commercial NMR magnets are designed to meet homogeneity specifications for particular applications. On the other hand, for home-built, custom, or otherwise out-of-spec magnets, inhomogeneity may be beyond the range of the active shims. Ferromagnetic passive shims take very little space and are field-deployable, thus being an attractive option.
We will share approach to designing ferromagnetic shims using a low-tech method. Our calculations were performed using Excel spread sheet; steel foil pieces were cut to size by hand and attached using consumer grade adhesive tape. The method can be used with little prior experience. The technique was tested while designing ferromagnetic shims for the 1500 MHz Series-Connected Hybrid (SCH) magnet at the National High Magnetic Field Laboratory.

POSTER 192
A Miniature Magnetic Field Sensor Utilizing Integrated NMR RF Transceiver
Presenting Author: Guang Yang

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Imperfections in gradient spatial constancy can affect MRI image quality.
To address this issue, we present a small magnetic field sensor designed for monitoring magnetic fields during imaging. The sensor includes a CMOS RF transceiver IC and a small NMR probe with a water sample, which are inserted into the MRI bore, and a controller with an FPGA module, PLL, and ADC that stays outside the bore and connects to the transceiver IC via power and SPI cables.
The field sensor prototype successfully captured FID using a 0.51-T permanent magnet.

POSTER 193
The LLWG: A Low-loss, Low-cost, Small-diameter THz Waveguide for MAS-DNP in NB Magnets
Presenting Author: F David Doty

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A novel waveguide for the 70-1500 GHz range will be presented that achieves loss more than two orders of magnitude below that of fundamental-mode waveguides at 400 GHz, comparable to that of corrugated waveguides of similar size, but is possibly two orders of magnitude more manufacturable at small diameters. The novel Laminate-Lined Waveguide (LLWG) is being integrated into a NB MAS probe for DNP that includes a high-mode THz cavity compatible with MAS and is expected to permit routine low-cost operation below 15 K. The goal is to enable MAS-DNP in NB high-field magnets using solid-state sources.

METABOLOMICS (Posters 194 – 209)

POSTER 194
Using NMR to Characterize Mitochondrial PolgD257A Mutator Mouse for Mitochondrial Diseases and Toxicity
Presenting Author: Qiuwei Xu

Complete Author List:
Connor Quinn (Merck Research Laboratories); Heather Vu (Merck Research Laboratories); Radha Desai (Merck Research Laboratories); Qiuwei Xu (Merck Research Laboratories)

Mitochondrial dysfunction is often a cause of many human diseases. PolgD257A impairs DNA replication "proofreading" and leads to progressive accumulation of mutation in mitochondrial DNA. The PolgD257A mouse provides an animal model for mitochondrial neuronal disease target validation. We have applied NMR metabolomics to identify metabolites and pathways related to mitochondrial dysfunction. Our internal library of over 700 endogenous metabolites provides an opportunity of quick chemical identification and shifts our focus on biochemical interpretation. In this presentation, we will show our recent work of metabolomics profiling of PolgD256A mutator mouse model and analysis of pathways under which significant changes were observed in mitochondria, glycolysis, and many other endogenous metabolites.

POSTER 195
13C NMR Spectroscopic Tracking of the Biochemical Changes in the Leloir and Glycolytic Pathways under Hypoxia in Liver Cancer
Presenting Author: Daniel Anable

Complete Author List:
Daniel Anable (University of Texas at Dallas); Lloyd Lumata (University of Texas at Dallas)

The Leloir pathway is the main metabolic pathway for the catabolism of the glucose epimer galactose [1,2]. The Leloir pathway involves 4 major enzymes in which glucose-1-phosphate is produced from galactose: 1, galactokinase; 2, galactokinase 1-phosphate uridyl transferase; 3, UDP-galactose 4-epimerase; 4, UDP-glucose pyrophosphate [1,2]. This pathway primarily occurs in the liver and serves as an alternate energy source for liver cells through conversion into glucose-6-phosphate and the glycolytic pathway. In this study, the metabolism of D-galactose was investigated using 13C NMR spectroscopy in liver cancer vis-à-vis the glycolytic pathway (glucose metabolism) in light of the Warburg effect under normoxic and hypoxic conditions.

POSTER 197
Revisiting Sample Preparation Protocols in Metabolomics
Presenting Author: Saraa Al Jawad

Complete Author List:
saraa Al Jawad (UGA); Mario Uchimiya (UGA); Arthur Edison (Network for Advanced NMR, University of Georgia, Complex Carbohydrate Research Center)

Sample preparation is a critical step in metabolomics, and standardization across labs is important for reproducible results. The current best practices in sample preparation for both urine and serum/plasma were optimized for room-temperature NMR probes and use phosphate buffers. Kelly (2002) has shown that phosphate buffers reduce the sensitivity of cryoprobes by about 2/3. This problem is even worse in urine, which has an overall salt concentration of >200 mM before any buffer is added. We previously found that DMSO minimizes the effects of salt and may improve sensitivity at high fields and with cryoprobes. We will present improved sensitivity in urine DMSO samples using a 5-mm cryoprobe at 600 MHz. At higher fields, the benefit should be even greater.

POSTER 198
Tracking the Influence of Transition and Lanthanide Metals on the Glycolytic Pathway of Neuroblastoma Cells Using 13C NMR
Presenting Author: Cody Larsen

Complete Author List:
Cody Larsen (University of Texas at Dallas); Lloyd Lumata (UT Dallas)

Despite their toxicity, lanthanides are a common starting material for developing contrast imaging agents to enhance the signal intensities of magnetic resonance imaging. Consequently, this work seeks to investigate the effects that lanthanide and paramagnetic transition metal ions have upon glucose metabolism and lactate production via the glycolytic pathway of cultured neuroblastoma cancer cells. The metabolic pathways were studied using 13C NMR spectroscopy by monitoring the conversion of glucose to lactate over 48 hours using a Bruker 600 MHz NMR spectrometer. Preliminary results indicating inhibition and excitation of certain lactate products will be presented, along with other experimental results.

POSTER 199
Hydrogen-Deuterium Addition and Exchange in N-Ethylmaleimide Reaction with Glutathione
Presenting Author: Daniel Raftery

Complete Author List:
Daniel Raftery (University of Washington); Vadim Pascua (University of Washington); Fausto Carnevale Neto (University of Washintgon); G. A. Nagana Gowda (University of Washington)

Glutathione is an ubiquitous thiol compound abundantly present in virtually every living cell. It is a powerful antioxidant critically required to protect cells from oxidative damage and free radical injury. It is very challenging to analyze glutathione in its native form from biological samples since the active form spontaneously becomes oxidized. To address this challenge, we developed a simple chemical derivatization method using N-ethylmaleimide (NEM) for the analysis of the notoriously unstable reduced glutathione antioxidant and its oxidized form. The chemical derivatization exhibited several intriguing phenomena including the generation of 12 different hydrogen-deuterium isotopomers of NEM-glutathione. The findings broaden the scope of metabolite profiling and impact areas of metabolomics, small molecule synthesis, and bioconjugation chemistry.

POSTER 200
13C NMR Study of the Effect of Sodium Dichloroacetate (DCA) in Glucose Metabolism of Cultured Colorectal Cancer Cells
Presenting Author: Emmanuel Ameh

Complete Author List:
Emmanuel Ameh (The University of Texas at Dallas); Lloyd Lumata (University of Texas at Dallas)

In this study, the effects of dichloroacetate on the metabolism in cultured colorectal cancer cells have been studied via 13C NMR spectroscopy. Cancer cells utilize glucose at a higher rate under the glycolytic pathway for ATP production as compared to normal cells. In particular, this study used carbon-13 NMR spectroscopy to track the glucose metabolism in the presence of dichloroacetate at different concentrations in cultured Colo-205 and LoVo colorectal cancer cell lines. The details of these results will be presented. This study is supported in part by the Welch Foundation grant AT-2111-20220331, the UT Dallas CoBRA and SPIRE seed grants, US Department of Defense CDMRP grants W81XWH-21-1-0176, W81XWH-22-1-0105, W81XWH-19-1-0741, HT9425-23-1-0062, and W81XWH-22-1-0003.

POSTER 201
Tracking the effects of LDH and hexokinase inhibitors on glucose metabolism in cancer cells using NMR
Presenting Author: Asiye Asaadzade

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Sodium oxamate is an inhibitor of lactate dehydrogenase (LDH), specifically LDH-A, an important enzyme that is active in cell and is responsible for catalyzing the reversible conversion of pyruvate to lactate.1 On the other hand, 2-deoxy-glucose (2DG) is a glucose analog that acts as hexokinase inhibitor suppressing glycolysis in tumor cells.2 In this work, we have investigated via carbon-13 nuclear magnetic resonance (NMR) spectroscopy the metabolic effect of varying concentration of sodium oxamate and 2DG administered separately on [1-13C] glucose metabolism in a variety of cultured cancers cell including renal cell carcinoma (786-O), hepatocellular carcinoma (HepG2), and glioblastoma (SfXL) cells.

POSTER 202
2D 1H-13C Experiments for Targeted Analysis of Structural Sub-classes in-vivo
Presenting Author: William Wynne Wolff

Complete Author List:
William Wolff (University of Toronto); Daniel Mathieu (Bruker Biospin Corporation); Kiera Ronda (University of Toronto Scarborough); Katrina Steiner (University of Toronto); Katelyn Downey (University of Toronto); Peter Costa (University of Toronto); Daniel Lysak (University of Toronto); Ronald Soong (University of Toronto); Andre Simpson (University of Toronto)

In-vivo NMR is a powerful tool for tracking the biochemical responses of organisms in response to environmental stress but has been limited by poor resolution as a result of the extreme inhomogeneity of living organisms, and in the chemical complexity of an organism. Targeted pulse programs can support environmental studies, but often discard information that might be useful in understanding the mode of action of environmental stressors. To address these challenges, we introduce two new targeted pulse sequences: an in phase anti-phase (IPAP) HSQC to separate amides and acids alongside a conventional HSQC, and a 2D HCCH-TOCSY to obtain additional correlations whilst maintaining high resolution in a narrow band.

POSTER 203
Exploration of Materials for Planar and Three-Dimensional Microcoil Production
Presenting Author: Vincent Moxley-Paquette

Complete Author List:
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5-axis CNC micromilling is an excellent prototyping tool for microcoil technology, allowing for custom made microcoils with built-in sample wells tailored to specific mass-limited samples. Although Cu-laminated Teflon was previously the material of choice for developing microcoils with this method, creating microcoils on other materials (including Acrylic, which cannot withstand the heat required for soldering) was not thoroughly explored. In addition, due to the limitations of 5-axis milling, machining more complex 3D volume microcoils (saddle coils and solenoids) required the use of a high-precision Elara 4-axis CNC milling machine. In this presentation, the performance of microstrips machined from number of Cu-laminated dielectrics will be compared, with the materials providing the best lineshape/SNR used to make microsolenoid and microsaddle coils.

POSTER 204
Mixing it Up with NMR: Low-Field NMR Undergraduate Mixture Analysis Experiments using Real-World Samples
Presenting Author: Katrina Steiner

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The current gaps in NMR education can largely be attributed to three barriers: 1) the lack of access to expensive high-field NMR facilities, 2) the highly specialized and complex nature of operating high-field instruments, and 3) the lack of readily available teaching materials. Benchtop NMR aids in addressing the first two barriers, while the two undergraduate experiments presented here address the third. The poster will introduce two benchtop laboratory experiments that involve the mixture analysis of real-world samples. The first uses 1D 1H NMR techniques to identify unique spectral fingerprints in three different cocktails. The second is more advanced and applies 1D, 2D, and specialized experiments to pattern match and identify the ingredients in the popular energy drink Red Bull.

POSTER 205
Low-Field, but Not Low Quality: 1D Simplification, Selective Detection, and Heteronuclear 2D Experiments for Improving Low-Field Environmental NMR
Presenting Author: Katelyn Downey

Complete Author List:
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NMR spectroscopy could be an effective environmental research tool, but high-field NMR is financially and physically inaccessible. Alternatively, low-field NMR is accessible but is also less sensitive and suffers from spectral overlap. Therefore, this work investigates experiment types (1D spectral simplification, selective, and heteronuclear 2D experiments) that can help overcome sensitivity and overlap challenges in low-field analysis of environmental samples. Notable findings include that JRES is more sensitive but less effective than PSYCHE at removing homonuclear coupling. Both GEMSTONE and DREAMTIME are efficient selective experiments, but GEMSTONE is simpler to operate, whereas DREAMTIME can perform multiple selection. Ultimately, low-field NMR spectroscopy has untapped potential in environmental research, and further optimization may make it a viable tool in future applications.

POSTER 206
Exploring Proton-only Experiments and Filters for In Vivo Samples: Potential and Limitations
Presenting Author: Kiera Ronda

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In order to understand the environmental impacts of anthropogenic activities, it is essential to assess the impacts of contaminants on living organisms. Therefore, environmental metabolomics has become an increasingly important area of research. In vivo Nuclear Magnetic Resonance spectroscopy is ideally suited to analyze the complex systems observed in living organisms. However, limitations associated with spectral overlap and overwhelming water signals make these studies difficult. Two-dimensional experiments can simplify the analysis, but often require 13C labeled organisms, which are more costly and less applicable to natural systems than their non-labeled counterparts. Thus, this work will examine the practicality of 1H filters and different suppression techniques in the analysis of non-labeled organisms as a complementary tool to increasingly expensive 13C enrichment.

POSTER 207
NMR compatible Bioreactor without Background Signal
Presenting Author: Julia B. Schulte-Hermann

Complete Author List:
Julia Schulte-Hermann (Karlruhe Institute of Technology); Monsur Islam (Karlruhe Institute of Technology); Jan G. Korvink (Karlsruhe Institute of Technology); Neil MacKinnon (Karlsruhe Institute of Technology)

Studying metabolic pathways or profiles of bacteria through NMR provides exciting opportunities in different fields, including medicine, environmental science, and biology. Since NMR is non-invasive and non-destructive, it allows long term monitoring of chemical processes without having to interrupt and extract samples, or even destroy the samples in the process. Therefore, time-course metabolic measurements of the same sample is possible.
In this work, we present an NMR compatible, rapid manufactured bioreactor for incubation of Escherichia coli. It is compatible with a 10 mm coil of a standard high field NMR spectrometer and can be inserted directly into the detection area. This bioreactor ensures the vitality of the organisms and provides a suitable bio- and magnetic field-compatible environment.

POSTER 208
NMR Methods for Determining Lipid Turnover via Stable Isotope Resolved Metabolomics
Presenting Author: Penghui Lin

Complete Author List:
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Here we report a quick and easy way to estimate the incorporation of 13C into different subunits of complex lipids by NMR using cellular phosphatidylcholine lipids (PCs) as internal standard in stable isotope tracer experiments. The ratios of peak intensities of other species to that of PC methyl groups in both the proton and the HSQC spectrum could be used for enrichment calculation. This method provides a simple tool for generating an overview of 13C incorporation into lipid molecules, which can be utilized as a standalone approach or to compliment targeted mass spectrometry-based lipidomics workflows. Further, with the detection limit as low as 2%, it provides very valuable information other techniques cannot easily generate.

POSTER 209
Development of MR Brain Phantoms to Improve Understanding of In-vivo Molecular Dynamics
Presenting Author: Mira Menon

Complete Author List:
James Collins (University of Florida); Joanna Long (University of Florida); Mira Menon (University of Florida)

To determine the quantitative accuracy and reliability of applying existing MR techniques in vivo for measuring metabolites concentrations, as well as to assist with the development of new methods for their quantitation, high quality phantoms that mimic the in vivo composition of metabolites and their local environment are required. A phantom based on multilamellar vesicles (MLVs) composed of lipids extracted from porcine brains were used to evaluate how each thickening agent affects small molecule metabolite linewidths and motion. To assess the various phantoms, T1, T2, T2* and diffusion measurements were made using optimally shimmed sample preparations. Comparisons are made with ex-vivo brain tissue samples at the same field strength, and potentially reveal details about metabolite local environment in brain tissue.

MRI MRS (Posters 210 – 226)

POSTER 210
Dual Signal Ultrasensitive Magnetic Resonance Probe for Precise Drug Delivery Monitoring
Presenting Author: Chenlu Yuan

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We synthesized an ultrasensitive 129Xe NMR probe with dual signals. It is a hollow nanoparticle composed of water-soluble CB[6]. It has two different hydrophobic cavities. Two 129Xe NMR signals with different chemical shifts can appear in a single detection, just like a mobile phone with dual SIM cards, which can interact with the electromagnetic waves from multiple "operators" transmitting towers with different frequencies at the same time. Therefore, it will not easily "lose contact" in complex environments, effectively avoiding the occurrence of false positive and false negative in complex biological environment detection, improving the accuracy and sensitivity of single detection, and has broad application prospects.

POSTER 211
Diffusion Tensor Imaging With Three Types Of Correction To Reveal Physiological And Morphological Differences in The Liver
Presenting Author: Artur T. Krzyżak

Complete Author List:
Weronika Mazur (AGH University of Science and Technology); Artur Krzyak (AGH University)

In pursuance of the liver parametrization based on the diffusive properties, diffusion tensor imaging (DTI) was proposed as a superior technique. DTI data were corrected for noise (increasing number of excitations), systematic errors (using B-matrix spatial distribution, BSD) and geometrical distortions (applying the affine registration relying on a 3D algorithm from a python library for the analysis of MR diffusion imaging). Diffusion tensors were calculated using four approaches, each encompassing different b-values sets. DTI metrics obtained after three types corrections evinced the largest reflection of the liver physiology and morphology. Based on the preliminary results with the age division, it can be suspected that distinguishing the perfusion- and diffusion-dominated signals, morphological and physiological differences in the livers are DTI-sensitive.

POSTER 212
The CaRAP, HISuc Hypotheses: Significant Advances at the Limits of 3T Proton MR Spectroscopy.
Presenting Author: Ralph E Hurd

Complete Author List:
Ralph Hurd (Stanford); Meng Gu (Stanford); Daniel Spielman (Stanford)

The neurological stress resulting from cardiac bypass can be followed by dynamic single voxel spectroscopy. Dynamic changes are reported in a CBP piglet model. Glutamine changes led to the Circulatory-arrest Recovery Ammonia Problem hypothesis. Results predicted a significant bolus of ammonia reaching the brain on re-perfusion, confirmed by blood ammonia assays. High Hypoxic-Ischemic Succinate has been established as a marker for elevated ROS and damage during re-perfusion. Succinate elevation was observed during circulatory arrest. In addition to discoveries with high potential clinical consequence, the model itself provides the controlled feedback and validation needed for the improvement of MR spectroscopy. In this presentation we show how the MR spectroscopy protocol has evolved over the past 3 years.

POSTER 213
Using MRI to Study High Pressure Nutrient Infusion
Presenting Author: Julia Kerr

Complete Author List:
Julia Kerr (Lawrence Livermore National Laboratory); Daniel M. Gruber (University of California, Davis); Matthew P. Augustine (University of California, Davis)

High pressure assisted infusion of nutrients into food is in situ monitored with magnetic resonance imaging (MRI). The model food used here is peeled apple flesh. The nuclear spin relaxation properties of the water surrounding the apple flesh are enhanced by adding paramagnetic manganese cations for MRI relaxation contrast during pressurization. This work tracks the efficiency of pressure induced nutrient infusion in situ, demonstrating that pressure gating and ramping offer no nutrient mass transport advantage over operation at constant pressure and that the presence of a peel expectedly disrupts solute transport into the fruit. High pressure assisted infusion, with all pressurization schemes studied here, yields nearly 100-fold faster infusion times than at ambient pressure.

POSTER 214
Association of MR-based functional and physical phenotyping with body impedance analysis
Presenting Author: Chetna Banga

Complete Author List:
Rama Jayasundar (Department of NMR, All India Institute of Medical Sciences); Dr. Preeti Bhosle (Ex Senior Research Fellow, Department of NMR, AIIMS); Dr. Dushant Kumar (Department of NMR, AIIMS); PROF. K.K Deepak (Ex HOD, Department of NMR, AIIMS); CHETNA BANGA (PhD Scholar, Department of NMR, AIIMS)

With growing demand for precision medicine, phenotyping is gaining much attention. This study has evaluated MR-based resting–state functional activity and body composition,and correlated with body impedance analysis in healthy volunteers (n=40). Resting state fMRI and mDIXON-Quant sequence for fat evaluation in abdomen and thigh were carried out at 3T. Body impedance analyzer was used to calculate body fat percentage, visceral fat area and mass, subcutaneous and segmental fat mass in different body regions i.e bilateral limbs (arms and legs) and trunk and visceral fat level. Volunteers were categorized into two different groups on the basis of their BMI values- < 25 kg/m2 (Group I) and > 25 kg/m2 (Group II). This study brings out the potential of MR in phenotying

POSTER 215
Monitoring The Molecular Status of Pinwheel [2]Rotaxanes Using 19F-NMR/MRI: A Mechanistic Study
Presenting Author: Zhongxing Jiang

Complete Author List:
Zhongxing Jiang (Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Xin Zhou (Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences)

Here, we have assessed the potential of 19F NMR and 19F MRI to monitor the molecular dynamics of [2]rotaxanes and provide insight into mechanical bonds (MB) and mechanical movements (MM). We developed novel 2-blade pinwheel [2]rotaxanes, containing symmetrical fluorine atoms on the wheel and axle to yield sensitive 19F NMR/MRI reporters of molecular dynamics. 1H/19F NMR studies revealed that the relaxation rates of the reporters strongly depend on the MB&MM. Solid-state 19F NMR measurements further demonstrated that the wheel in [2]rotaxanes exhibits a longer rotational correlation time and undergoes slower rotational motion than the macrocycle. The formation of MB in [2]rotaxanes leads to significant modulations of rotational correlation time and thus the relaxation rates of the fluorine atoms.

POSTER 216
Impact of Selected Polysaccharide Model Systems on the Relaxivity of Dissociated Gd-Ions from Gadolinium-based Contrast Agents (GBCAs)
Presenting Author: Patrick Werner

Complete Author List:
Patrick Werner (German Cancer Research Center (DKFZ)); Matthias Taupitz (Charite); Leif Schrder (German Cancer Research Center (DKFZ))

The use of GBCAs in medical imaging is widespread, but recent studies have raised concerns about the potential long-term retention of released Gd3+ ions. The mechanisms underlying this retention are not yet fully understood, but interactions with polysaccharides like glycosaminoglycans may play a critical role. We here demonstrate that the molecular weight of the polysaccharide in the microenvironment appears to impact the water accessibility of bound Gd3+, which in turn affects the observable r1 values in MRI scans. This means that the embedding of Gd in tissues may not always result in hyperintense signals in MRI scans as reported. Further research is necessary to understand the molecular interactions involved in Gd retention and relaxometry can provide important insights.

POSTER 217
Single-Sided NMR for Hydrogel Characterization
Presenting Author: Daniel Gruber

Complete Author List:
Daniel Gruber (CU Boulder/ NIST); Mark Ferris (CU Boulder/ NIST); Gary Zabow (NIST)

Hydrogels - hydrophilic polymers that maintain integrity when hydrated - have been synthesized to be responsive to external stimuli, such as temperature and pH and have found a wide variety of uses. Knowledge of their physical properties is crucial for effective design and integration. We describe a contactless technique to measure hydrogel swelling and swelling rate using the NMR-MOUSE and an inversion recovery-leveraged pulse sequence, resulting in spatial precision below 100 µm and temporal precision below 10 minutes per data point. A dephasing delay pulse sequence reveals diffusion and tortuosity, giving insight into physical structure. This is demonstrated with pH-sensitive "smart" hydrogel as a simple example elucidating changes resulting from different formulation and preparation strategies, as well as conditioning.

POSTER 218
MRS in Neurosciences – Recent advances in In-vivo spectroscopy methods and applications at the Stanford Center for Cognitive and Neurobiological Imaging
Presenting Author: Laima Baltusis

Complete Author List:
Laima Baltusis (Stanford University); Donna Murray (Stanford University); Ralph Hurd (Stanford University); Meng Gu (Stanford University); Hua Wu (Stanford University); Sache Coury (Stanford University); Ian Gotlib (Stanford University); Daniel Spielman (Stanford University)

The interest of measuring metabolic changes via MRS techniques and combining that information with functional MRI measurements continues to grow. The Stanford Center for Cognitive and Neurobiological Imaging (CNI) spectroscopy program has become a best practice through a community effort of spectroscopy expertise from CNI staff, MRI scientists, and an expanding user community each supporting spectroscopy research projects conducted at CNI.
We will present new recent developments and results of focal MRSI using the semi-LASER sequence in challenging and less well studied areas of the human brain such as the basal ganglia regions where complex functions related to movement, cognition, and emotion are carried out and where single voxel data is of generally lower quality.

POSTER 219
Lipid Nanoparticles Enhanced with Gadolinium Chelating Lipids as New, Potential Contrast Agents in Magnetic Resonance Imaging
Presenting Author: Karolina Gębicka

Complete Author List:
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The aim of the research was to develop new MRI contrast agents based on GMO (glyceryl monooleate) lipid nanoparticles loaded with two types of Gd-chelating lipids: DTPA-BSA (Gd) and DPPE-DTPA (Gd). The effect of these two types of high-molecular-weight Gd-chelating lipids on the structure of fabricated GMO-based lipid nanoparticles and their physicochemical properties, such as particle size, long-term colloidal stability and morphology were studied. The substantial results include MRI contrast properties studied by means of relaxation times measurements and MR imaging efficiency.
Results indicate that developed GMO/DTPA-BSA-Gd and GMO/DPPE-DTPA-Gd nanoparticles have the potential for efficient MR contrast agent, and further, considering their unique properties, for the development of multifunctional systems combining diagnostics and therapy in a single system.

POSTER 220
Imaging Protein-Ligand Interactions via 19F-MRI
Presenting Author: Dilara Faderl

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Ligands interact with proteins non-covalently, influencing the population distribution of conformational states, and thus modulate the function of the protein. Magnetic Resonance Imaging provides a window through which it is possible to characterize these molecular interactions.
As a model system for monitoring binding, we have used 4-trifluoromethyl)benzamidine (TFBA) and trypsin as the ligand and target protein, respectively. In presence of increasing trypsin concentrations, the 19F-MR signal changes of TFBA reflected the change in T2 upon binding. By adding in a dose-dependent manner an 19F MR-invisible competitor ligand, benzamidine (BA), we observed a corresponding recovery of the TFBA 19F signal intensity. By comparing 19F-T2-weighted MR images of TFBA in the presence of different BA concentrations, the TFBA-trypsin interaction could be characterized.

POSTER 221
Ultra-High Resolution fMRI in Awake Mice at 14T
Presenting Author: David Hike

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This study utilizes implantable RF coils as a novel head fixation mechanism in awake mouse functional magnetic resonance imaging. The implantable RF coils provide significantly higher SNR for fMRI studies and the head fixation method limits motion-induced artifacts considerably. SNR comparisons were done at 9.4T and 14T with a commercial coil as control at 9.4T. All fMRI studies were done at 14T using the implantable coils. Current whisker stimulation data shows strong barrel cortex activation with additional activation seen in the retrosplenial area, ventral posteromedial nucleus, and upper limb somatosensory cortex. Future work can utilize this setup to record real-time pupillometry and whisking movement for use as regressors, providing a behavior-driven mapping tool to study other animal models.

POSTER 222
NMR Characterization of Unfrozen Brine Vein Distribution and Structure in Frozen Systems
Presenting Author: Peng Lei

Complete Author List:
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Due to the freezing point depression, in frozen systems, unfrozen water persists between the interface of ice crystals and/or particles. This water forms a liquid vein network (LVN) in dynamic and complex frozen systems. By investigating the development of the distribution and structure of the LVNs, the process of ice recrystallization can be studied in frozen systems using magnetic resonance tools such as MRI, relaxation and self-diffusion measurements. The distribution and structure of the LVN are studied as a function of temperature, salinity, particle size and ice binding protein. These results help to understand problems as broad as how frost heave happens in cold regions or how to better preserve frozen food or cryopreserve cells.

POSTER 223
An innovative approach for the design of a 20 mT, 90 cm bore, Bitter-type electromagnet for earth field MRI.
Presenting Author: Gianni Ferrante

Complete Author List:
Gianni Ferrante (Stelar.); Ludovico Minati (Stelar); Marco Rabaioli (Stelar srl)

We present an innovative project of a Bitter-type electromagnet featuring 20mTesla B0 and 90cm inner bore, designed as magnetic polarization coil for a 'pre-polarized Earth-Field MRI' system.
The Bitter electromagnet offers an excellent homogeneity over 60 cm sphere and presents a very simple mechanical assembly.
The pre-polarized earth field MRI system is developed within the PRIMOGAIA, Horizon2020 project, funded by the European community. The main objective of the whole project is to develop a suitable technology 'to search for new contrasts linked to molecular events for the very early diagnosis of pathologies.
Details and spec of the magnet system are shown in the poster.

POSTER 224
Are we ready for in vivo Sodium(23Na+)-based functional MRI (SOBA-fMRI) under 14T?
Presenting Author: Yuanyuan Jiang

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Xin Yu (MGH); Yuanyuan Jiang (MGH)

Conventional fMRI methods map brain function based on the hemodynamic responses of vessels coupled to neuronal activity. How to directly measure neuronal activity through the NMR signal is an ongoing challenge of neuroimaging. In particular, to identify the "true fMRI" signal directly linked to the neuronal activity—e.g., the action potential (AP) or local field potential (LFP) mediated by transmembrane ion movement—x nuclei-NMR has the potential for improved mapping specificity compared to proton-based hemodynamic fMRI. Here, we applied a reshuffled k-t space FLASH sequence to directly map the Sodium-based (23Na) (SOBA) fMRI signal changes in rat brains with 0.4x0.4x2mm spatial resolution and 10ms TR. In contrast to positive BOLD fMRI signals, we detected negative SOBA signals in the activated barrel cortex.

POSTER 225
Multi-nuclear MRI and MRS Using 0.35 T Clinical MRI Scanner
Presenting Author: Md Raduanul H. Chowdhury

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Hyperpolarized 129Xe, propane and [1-13C]ketoisocaproate are potential hyperpolarized contrast agents for their utility in lung imaging and molecular imaging of cancer. We use low-field (0.35 T) MRI scanner to demonstrate the pilot feasibility of multi-nuclear imaging in phantoms imaging and spectroscopy using these HP contrast agents. For HP 129Xe, we used a natively supported 129Xe GRE sequence to demonstrate ultra-fast slice-selective 2D GRE MRI with 64x64 imaging matrix with in-phantom SNR of 180. For HP propane, we demonstrate a sub-second 2D GRE scan with 64x64 imaging matrix and SNR of 210. Moreover, a post-mortem injection

of hyperpolarized [1-13C]ketoisocaproate allowed dynamic MRS scan in a freshly euthanized mouse demonstrating the delivery of 9% polarization (starting polarization is 12% inside the polarizer).

POSTER 226
Comparing Diffusion Measurement Protocol of Polyvinylpyrrolidone (PVP) Solutions Across Three MR Systems
Presenting Author: Cassandra M Stoffer

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The apparent diffusion coefficient (ADC) of water is an important biomarker in tissue health that can be measured using NMR and MRI techniques. Aqueous solutions of polyvinylpyrrolidone (PVP) with well-defined ADC values are used in MRI calibration objects (phantoms) to ensure the accuracy of data across imaging systems. In this work, PVP solutions are prepared using solute from various manufacturers with various molecular weights. A molecular weight survey is conducted by measuring the ADC values of prepared solutions across 3 systems: 128MHz (3 T) NMR, 128 MHz (3 T) MRI, and 2.4MHz (56 mT) unilateral NMR. We compare diffusion measurement protocol across these systems and develop PVP solutions that better mimic human tissue for use in MRI phantoms.

ORGANIC INORGANIC AND HYBRID MATERIALS (Posters 227 – 253)

POSTER 227
29Si and 1H NMR structural study of Chrysocolla, an amorphous copper containing silicate: Application to archeological copper metallurgy.
Presenting Author: Claire Roiland

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A unique archeologic site located at Ujina-Collahuasi (Tarapacá, Chile) gives many clues to understand the functioning of the pre-hispanic furnaces to reduce ores to metal, especially in copper metallurgy. The main copper ore used appears to be Chrysocolla, a natural amorphous paramagnetic mineral with a proposed formula unit Cu2H2 (Si2O5)(OH)4·nH2O. In this work, we highlighted the necessity to employ adiabatic pulses to ensure a full excitation of the paramagnetic component. This is crucial to perfectly record broad lineshape and thus, to probe correlations between silicium and proton in this mineral. All those results allow us a good understanding of the Chrysocolla structure necessary to go further on the understanding of its thermal degradation during the metallurgy process.

POSTER 228
Solution State NMR-Guided Purification of Carbon Quantum Dots
Presenting Author: Cody Soper

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Carbon-based quantum dots have gained popularity over the last decade due to their low cost, scalable green production, biocompatibility, and favorable optoelectronic properties. Following bottom-up synthesis, the resulting product is often a mixture of carbon dots and partially-reacted precursor molecules with similar fluorescent signatures as the carbon dots. As such, these impurities are difficult to identify using fluorescent spectroscopy, and are too small to detect utilizing electron microscopy. Here, we present our initial results using solution-state 1H and 13C NMR to guide the purification of carbon dot mixtures via dialysis and size exclusion chromatography. Preliminary findings demonstrate that 1H and 13C NMR spectra of samples at different stages of purification show spectral signatures that are lacking in fluorescence spectroscopy.

POSTER 229
Dynamic Nuclear Polarization for Perovskite Photovoltaics
Presenting Author: Aditya Mishra

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Solid-state NMR has been used widely within the perovskite community to study cation incorporation, phase segregation, halide mixing, disorder, and dynamics. However, insensitivity prevents the study of application-relevant thin films, especially for surface coatings and additives. Firstly, we will show how tailoring the organic cation's relaxation properties helped us design impregnation dynamic nuclear polarization (DNP) experiments that achieve unprecedented bulk signal enhancements for hybrid perovskites. Secondly, we will show the possibility of DNP for three-dimensional inorganic perovskites doped with high-spin metal ions. The achieved sensitivity enhancements can be explained in terms of the dopant concentration, relaxation times, microwave absorption, and spin-diffusion. These DNP methods will pave the way to establishing structure-activity relationships in these materials for photovoltaic applications.

POSTER 230
Molecular Fingerprint of Wetland Soil Elucidated by Solid-State NMR and DNP
Presenting Author: Tuo Wang

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Wetland soil is important for carbon storage but it has been threatened by sea level rise. We employed DNP methods to collect high-resolution 2D 13C/1H-13C correlation solid-state NMR spectra on unlabeled soil collected from a brackish island in Louisiana. The island is only 55 km from New Orleans but has been rapidly shrinking over the past five years and finally disappeared in 2021. We identified a highly preserved lignocellulosic core in the surface soil layer and plants grown on top of it. Extending to a 2-m depth allowed us to examine the molecular changes of soil over an 11-century period. The composition and properties of the soil are influenced by many geological and historical factors as well as human activities.

POSTER 231
Impacts of Electrolyte Speciation on Ion Binding Environments in Aluminum-Quinone Batteries Elucidated by Dipolar-Mediated and Multiple-Quantum Solid-State NMR Methods
Presenting Author: Leo W Gordon

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Organic materials are earth-abundant, sustainable cathode alternatives for aluminum-metal batteries, however the electrolytes necessary to reversibly electrochemically plate and strip aluminum at room temperature contain a variety of polyatomic ions, leading to nuanced and complex charge storage mechanisms. Here, we determine electrolyte speciations with liquid-state NMR, and leverage multidimensional solid-state dipolar-recoupling NMR methods alongside multiple-quantum techniques to determine the nature and binding environments of solid aluminum-organic discharge products in three electrolytes. Complexed aluminum species are identified by their ^{27}Al-^{1}H dipolar interactions, and quadrupolar parameters determined by ^{27}Al{^{27}Al} MQ-MAS are linked with computationally-derived quantities to understand the physical characteristics of these bound ions. DFT calculated desolvation pathways were also validated by experimental measurements to conclusively reveal the ionic charge storage mechanism.

POSTER 232
Understanding the Solvation Structure of Li-Ion Battery Electrolytes Using DFT-Based Computation and 1H NMR Spectroscopy
Presenting Author: Julia Im

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Molecular dynamics simulations, density functional theory calculations, and 1H NMR spectroscopy were applied to understand the Li-ion electrolyte system, lithium bis(trifluoromethanesulfonyl)imide(LiTFSI) in tetraglyme(G4). By combining the computational and experimental methodologies, we show that the various solvation structures, dominated by the coordination between the tetraglyme solvent and lithium cation, directly influence the chemical shift separation of resonances in the 1H NMR spectra of the solvent. Thus, the 1H NMR spectra can be used to predict the fraction of tetraglyme involved in the solvation process, with a quantitative agreement with predictions from MD simulation snapshots. Overall, our results demonstrate the reliability of a hybrid computational and experimental methodology to understand the solvation structure and hence transport mechanism of the LiTFSI-G4 electrolyte system.

POSTER 233
Scalable nanoporous networks for carbon capture via solid-state NMR spectroscopy
Presenting Author: Haiyan Mao

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Carbon capture and sequestration reduce carbon dioxide emissions and is critical in accomplishing carbon neutrality targets. We demonstrate new sustainable, solid-state, polyamine-appended, cyanuric acid–stabilized melamine nanoporous networks (MNNs) via dynamic combinatorial chemistry (DCC) at the kilogram scale toward effective and high-capacity carbon dioxide capture. Polyamine-appended MNNs reaction mechanisms with carbon dioxide were elucidated with double-level DCC where two-dimensional heteronuclear chemical shift correlation nuclear magnetic resonance spectroscopy was performed to demonstrate the interatomic interactions. The coordination of polyamine and cyanuric acid modification endows MNNs with high adsorption capacity, fast adsorption time, low price, and extraordinary stability to cycling by flue gas. This work creates a general industrialization method toward carbon dioxide capture via DCC atomic-level design strategies.

POSTER 234
Paramagnetic Solid-State NMR: Direct Observation of the 55Mn Nuclei in Manganese Oxides for Batteries
Presenting Author: Anne Mirich

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Using traditional pulse sequences, direct observation of the transition metals (TM) in the cathode framework does not work. However, under certain conditions, the electrons spin pair to give S=0. By taking advantage of the spin pairing behavior in an extended lattice system, solid state NMR experiments were able to collect 55Mn spectra in an MnO2 solid. The amount of spin pairing was determined using SQUID experiments. Select ssNMR spectra were modeled using hybrid DFT calculations. Results showed that the total number of electron spins is less than what would be predicted, supporting the electron spin pairing theory.

POSTER 235
Structural Analysis of Nitrogen-Containing Hydrothermal Carbon by NMR
Presenting Author: Zhaoxi Zheng

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Molecular level detail is required for any attempt at rational design of N-hydrochar. 13C-glucose and 15N-glycine, model precursors that mimic major food waste components, were used to synthesize N-hydrochar characterized by NMR. Nonlabeled N-hydrochar synthesized from food waste was also studied and added for comparison. Quantitative composition analysis of labeled N-hydrochar was carried out both on 13C and 15N. Spectral editing showed >55% of carbons are not bonded to hydrogen, and >52% of carbons are aromatic. Rotational-echo double-resonance (REDOR) shows each nitrogen is bonded to two carbons in glucose and one carbon in glycine, and >65% of those carbons are not protonated. 15N NMR combined with 2D 13C-15N heteronuclear single quantum (HSQC) identified a wealth of possible forms of nitrogen.

POSTER 236
Mechanistic Insights into Processive Polyethylene Hydrogenolysis: An in-situ NMR Study
Presenting Author: Tommy Yunpu Zhao

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Chemical polymer upcycling by processive catalysts is a promising plastic waste remediation strategy, with the capability of producing selective, high-value products from waste plastics with minimal energy input. We employed in situ MAS NMR to study the underlying mechanism of processive polyethylene (PE) hydrogenolysis by mSiO2/Pt/SiO2 catalysts. We found that most PE-Pt interactions do not lead to C-C bond cleavage but rather release the polymer back into the melt. The hydrogenolysis to H/D exchange ratio increased with increasing shell thickness, indicating that longer pores inhibit the premature release of polymer thus afford higher extent of processivity. Coarse-grained molecular dynamics simulations were able to reproduce the trends observed in the experiments and further correlate pore geometry to processivity.

POSTER 237
Increased sodium mobility in sodium carbonphosphonitride thermosets from plasticization seen through Na-23 longitudinal relaxation behavior
Presenting Author: Christopher A. Klug

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Reacting phosphorus cyanide with lithium dicyanamide affords a conductive, thermosettable resin; however, the conductivity of the thermally cured resin at room temperature is too low to be practical for solid-state electrolyte applications. By exchanging out lithium with sodium and adding a plasticizer, the cured resin's conductivity is dramatically improved. Here, we characterize the cured resin's sodium variant with and without the plasticizer using solid-state NMR. Sodium-23 spectra contain a sharp peak and a broad band, suggesting two different types of Na-23 environments. Variable temperature spin-lattice relaxation measurements reveal broad band Na-23 relax faster for the plasticizer-containing sample, signifying an increase in sodium mobility achieved through plasticizer introduction. Kinetic parameters extracted from the relaxation rates will be presented.

POSTER 238
Structural verification and in-situ measurements of ZIF-67 in an electrochemical supercapacitor cell
Presenting Author: Mark O. Bovee

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Metal-organic frameworks (MOFs) are attractive as supercapacitor electrode materials because their high porosity promotes electrolyte-electrode interactions, boosting the system's capacitance. Previous work by our group characterized ZIF-67, a MOF displaying promise as an electrode material, using high resolution MAS NMR. These measurements revealed NMR can sense "guest" molecules in the pores. Here, we continue our study of ZIF-67 by measuring ZIF-8, a structural analog that replaces ZIF-67's paramagnetic Co2+ centers with diamagnetic Zn2+. Comparing the materials' 13C spectra provides further verification to our structural analysis of ZIF-67. Additionally, we present preliminary in-situ measurements of a supercapacitor cell that incorporates ZIF-67 as the positively charged electrode. Proton spectra of the electrolyte reveal features that change as a function of applied voltage.

POSTER 239
Probing the Ultrastructure of Oakwood Lignocellulose by Quantitative Solid-State NMR Analysis
Presenting Author: Zhenhuan Sun

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The lignocellulose ultrastructure of 13C-enriched young oakwood was analyzed using quantitative 13C NMR and spin diffusion. Proximities and domain sizes of 4 major components: lignin, acetylated hemicellulose, noncrystalline and crystalline cellulose, were probed using 2D 13C-13C exchange NMR. Spin diffusion out of crystalline cellulose documented 2.5±0.4 nm diameter crystal cores, corresponding to 3.5±1.5 nm thick cellulose microfibrils. The experimental data were matched by numerical simulation of spin diffusion in a quantitative model with a macrofibril of (6nm)2 cross-sectional area consisting of four cellulose microfibrils surrounded by hemicellulose and lignin. The macrofibril diameter was further validated by local equilibration in 1D 13C spin diffusion NMR and independently in a CHHC experiment with 13C-detected 1H spin diffusion after selective cellulose suppression.

POSTER 240
Distinguishing Degradation Products in Carbon Capture Polymers with ROCSA and Machine Learning
Presenting Author: Maxwell Marple

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Aminopolymers are promising sorbent materials for direct air capture applications that can be regenerated by exposure to elevated temperatures and steam. However, during regeneration the aminopolymer is susceptible to degradation that limits usable lifetime. Amine reactions with CO2 are complex and depend on temperature, humidity, and CO2 concentration, forming a wide variety of carbonyl-type environments. This complexity makes detecting degradation products difficult with standard solid-state NMR techniques. We use a 2D chemical shift anisotropy correlation measurement, ROCSA, to distinguish between chemisorbed and degraded products based on their 13C CSA parameters. To aid interpretation, we developed a machine learning model that classifies the type of carbonyl site based on its CSA values trained on over 100 crystal structures from DFT calculations.

POSTER 241
Direct Probing of Neighboring Sodium for Bridging Oxygen Atoms in Sodium Silicate Glasses with Solid-state NMR Spectroscopy
Presenting Author: Jeongjae Lee

Complete Author List:
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Sodium, silicon, and oxygen, constituting the majority of Earth's crust and mantle in forms of silicate minerals and melts, also find common use in many commercial glasses such as amorphous soda-lime glass. In this study, we show that the sodium distribution in amorphous sodium silicate glasses is likely to be more homogeneous than previously expected from the accepted modified random network (MRN) model. NMR correlation spectroscopy between the oxygen and sodium species in form of 17O→23Na triple quantum heteronuclear correlation experiments between quadrupolar nuclei unambiguously demonstrate the existence of sodium atoms in the immediate vicinity of bridging oxygen (Si–O–Si species), a feature unexpected from the MRN model.

POSTER 242
Investigating the Role of Solid Acid Catalyst in Depolymerization of Polyolefins using SSNMR
Presenting Author: Jinlei Cui

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Chemical recycling with catalysts can break the polymers into smaller monomers, which can then be reformed into high-quality materials. Fluorinated alumina oxide (F-Al2O3) is a catalyst that can improve the depolymerization of polyolefin, and its effectiveness is related to the proximity between the polymer and the catalyst's surface. In this study, the copolymer(ethylene-co-dodecene) was introduced into two distinct catalysts: fluorinated F-Al2O3 and Al2O3. Under a spinning rate of 10 kHz, 13C{27Al} TRAPDOR was

utilized with dynamic nuclear polarization (DNP) under 100k at 9.4T. The data indicated that F-Al2O3 exhibits a substantially faster dephasing compared to Al2O3, suggesting that the copolymer is in closer proximity to the former's surface.

POSTER 243
Understanding Oxidative Degradation Mechanisms of Ethyl and Propyl Amines
Presenting Author: Hannah Eshelman

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Polyethyleneimine (PEI) is a promising candidate for direct air capture (DAC) due to low volatility, low cost, and high selectivity and adsorption capacity for CO2. However, oxidative degradation of amine-containing adsorbents is a concern for DAC processes due to the abundance of oxygen in the air and temperature swings used for adsorbent regeneration. Solution state NMR was used to study a variety of aminooligomers containing either ethyl or propyl spacers and taking on either a branched or linear configuration to represent fragments of PEI. Corresponding 1H, 13C, 1H-1H COSY, and 1H-13C HSQC NMR of aminooligomers throughout their degradation was used to identify products and help determine the most stable configuration for new amine-containing DAC adsorbents.

POSTER 244
Strategies for Oxygen-17 and Calcium-43 NMR studies of biomaterials: isotopic labeling, (ultra)-high field NMR and DNP
Presenting Author: Danielle Laurencin

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Oxygen and calcium are highly abundant elements in living organisms. They are notably found within mineralized tissues like bone and teeth, and in pathological calcifications like kidney stones. Thus, to help understand the structure of these materials, 17O and 43Ca NMR appear as valuable probes, due to the high sensitivity of the NMR parameters of these nuclei to their local environment. Yet, both isotopes are very challenging. Here, some of our recent work aiming at using 17O and 43Ca NMR to investigate the structure of synthetic biomaterials will be presented.

POSTER 245
Investigating the structure of synthetic opioid precursors with single crystal X-ray diffraction and multi-nuclear solid-state NMR spectroscopy
Presenting Author: Harris E. Mason

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With the current synthetic opioid epidemic raging in the U.S., there is a significant need to understand the structure and chemistry of fentanyl, its analogues, and its precursors. The compound N-phenyl-4-piperidinone, referred to as NPP, is a critical precursor in the "Siegfried method" for the synthesis of the synthetic opioid fentanyl and its analogues. This material can serve as a "safe" alternative to fentanyl for structural studies. Here, we present the results of a systematic multinuclear solid-state NMR and single crystal X-ray diffraction (SC-XRD) study of the NPP crystal structure as a function of the coordinating anion strength.

POSTER 246
Understanding the Electrochemical Discharge Mechanism in Li-CFx Batteries from the Atomic to Macroscopic Scales by Solid-State NMR Spectroscopy
Presenting Author: Loleth Robinson

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NASA is vetting ultra-high-energy-density Li-CFx batteries for its mission concept to Europa, a moon of Jupiter with liquid water underneath its icy surface. The main challenges associated with this mission are battery aging during the long journey to Jupiter and high gamma radiation. These factors can have significant negative impacts on battery materials and electrochemical performance. As such, much remains to be understood about the electrochemical discharge processes at a molecular level and how the local compositions, structures and interfaces of the electrode materials change at different states-of-charge. Here, we combine quantitative and multi-dimensional, dipolar-mediated NMR measurements, coupled with electrochemical impedance spectroscopy, to enable elucidation of molecular-level environments and quantification of the electrode composition as a function of state-of-charge.

POSTER 247
Understanding Speciation in Ionic Liquid Analogue Electrolytes for Rechargeable Al Batteries
Presenting Author: Jonah Wang

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Rechargeable aluminum metal batteries are attractive due to aluminum's earth abundance, high theoretical capacity, low cost, and inherent safety. Ionic liquid analogue (ILA) electrolytes have been investigated as lower cost and less corrosive alternatives to state-of-the-art chloroaluminate ionic liquid electrolytes. However, much remains to be understood regarding how the types and populations of electrolyte species affect their physical and electrochemical properties. In this work, we use quantitative 1D 27Al and 1H single-pulse NMR measurements, as well as 2D 27Al{27Al} EXSY NMR experiments, to reveal molecular-level environments, populations, and dynamics present in Lewis acidic AlCl3-urea-[EMIm]Cl electrolyte mixtures of varying compositions. The results reveal insights into how electrolyte speciation and dynamics relate to its electrochemical properties.

POSTER 248
Quantification monitoring of drug loading in metal-organic layers by 129Xe MRI
Presenting Author: Xin Zhou

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Metal-organic framework layers (MOLs) are two-dimensional nanosheets of metal-organic frameworks (MOFs). Compared with the three-dimensional MOF materials, MOL may provide more surface area for 129Xe exchange due to its ultra-thin sheet structure. This specific capability makes MOL to be a potential 129Xe MRI contrast agent with improved availability. The porous ultra-thin structure and high specific surface area make MOL as a multifunctional drug-carrying cage. After DOX molecules occupied the surface and pores area of MOL, the chemical microenvironment and exchange process between MOL and 129Xe atoms is affected depending on aperture occupancy rate dominated by DOX-loading concentration. A 129Xe MRI method is provided for the quantification monitoring of drug loading concentration.

POSTER 249
Indirect detection of solid-state platinum species by {1H}195Pt PE-RESPDOR NMR
Presenting Author: Anna Pischer

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Nanoscale platinum is commonly used as a heterogeneous catalyst to facilitate chemical reactions that are the foundation for many industrially important energy and chemical manufacturing applications. Characterizing the electronic and bonding environments of solid-state platinum species by 195Pt NMR provides insights into their atomic-level compositions and structures. Historically, direct detection and resolution of 195Pt NMR signals have been limited due to their extremely broad lineshapes and the low Pt contents of industrially relevant materials. Nevertheless, indirect detection of 195Pt through a sensitive dipolar-coupled nucleus, such as 1H, enables solid-state 195Pt NMR spectra to be acquired with improved sensitivity. Results will be presented on the use of 1H{195Pt} PE-RESPDOR NMR and related techniques to analyze platinum species dispersed on heterogeneous catalysts.

POSTER 250
Fast Field Cycling NMR applied to polymers: tacticity and molecular weight
Presenting Author: Donald Bouchard

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In this study, we want to show the power of the Fast Field Cycling [1,2] NMR relaxometry (FFC) technique by addressing the assessment of one of the key properties of polymers, the tacticity,
We present a case study on the most widely used and well-known industrial polymer: the polypropylene. The "Tacticity" has important implications on its physical and mechanical properties. In this study, we investigate the potential of the FFC technique to discriminate commercially available isotactic polypropylene (PP) from atactic PP.

POSTER 251
Proton NMR to Evaluate Changes in Activity of Water in High Concentrated Aqueous Zinc Battery Electrolyte
Presenting Author: Alexis Scida

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Aqueous-based battery systems have been highly studied due to their inherent increase in safety; however, they face major issues regarding limitations in the electrochemical stability window and parasitic hydrogen evolution reaction (HER). Strategies, including increasing salt concentration, and the addition of co-solvents can help decrease free water molecules' reactivity, and form protective interface layers, thereby limiting unwanted side reactions. Herein, proton NMR and T1 spin-lattice relaxation are utilized to corroborate the changes in the chemical environment associated with water in a unique highly concentrated, multi-salt electrolyte for zinc-ion batteries. Changes in chemical shifts denote a decrease in acidity and polarization, whereas a reduction in relaxation time is evident in changes in long-range order of water.

POSTER 252
Unusual Crystal Chemistry in High-rate Battery Electrodes via 23Na NMR and DFT
Presenting Author: Kent Griffith

Complete Author List:
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In this work, we focus on multinuclear solid-state NMR characterization to explore the relationship between composition, crystal structure, and defect chemistry in a series of complex sodium and lithium niobium oxides. The role of defects on electrochemical transport properties in these new high-rate electrode or solid electrolyte materials will be discussed. Starting with average structure models from X-ray and neutron diffraction, we then turn to a local structure perspective from NMR that is more sensitive to defects and disorder. One- and two-dimensional 6,7Li and 23Na NMR spectra provide insights on mobile cation positions and dynamics as well as alkali sublattice vacancies. (Ir)reversible changes upon cycling are identified. DFT calculations and numerical simulations support the spectral assignments in these complex oxides.

POSTER 253
2D J- and Dipolar-mediated Correlation NMR Techniques for Structural Characterization of Dilute Heteroatom Sites in Zeolite Catalysts
Presenting Author: Michael B Schmithorst

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Aluminosilicate zeolites are crystalline, nanoporous materials used as catalysts in a variety of applications, including environmental emission control. NMR is well suited to the characterization of the local environments of 29Si and crucial 27Al atoms in the zeolite framework, however, their relative distributions are challenging to establish due to their non-stoichiometric compositions, low natural abundance of 29Si, non-periodic ordering and quadrupolar nature of 27Al, and inhomogeneous broadening of NMR signals. We demonstrate that the combined use of low-temperature J- and dipolar-mediated 2D 27Al-29Si HMQC and 2D 29Si-29Si INADEQUATE correlation NMR techniques provide detailed and complementary new understanding of dilute structural features of zeolite frameworks, which correlate with their macroscopic catalyst reaction properties.

SMALL MOLECULES NATURAL PRODUCTS (Posters 254 -271)

POSTER 255
"Dark" phosphate no longer dark?
Presenting Author: Jiaqi Lu

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Phosphate is the most abundant anion in the human body as integral structural components of cell membranes and bones. When bound with calcium, phosphates are hypothesized to form cluster. However, the underlying assembly mechanism of phosphate species and other factors that lead to bone formation processes are not well understood. We have found evidence that phosphate species including orthophosphates, pyrophosphates and adenosine phosphates associate into dynamic assemblies in dilute solutions that are spectroscopically 'dark', and that the aggregation propensity increases with rising temperature. Recently, we found pH also played an important role in affecting the phosphate aggregation in 'dark state', and chemical exchange saturation transfer (CEST) was employed in this study to provide new spectroscopic evidence for phosphate aggregation.

POSTER 256
NMR Relaxation and Solution Dynamics of Four-/Five-coordinate Co(II) Complexes
Presenting Author: Matthew P. Grindle

Complete Author List:
Matthew Grindle (Miami University); David L. Tierney (Miami University)

The physical properties of systems employed in solid state are derived from inherent properties of the molecules and the constraints on the molecule that effect crystallization. We use NMR to measure the characteristics of two related compounds in solution at 207-347K. Solution NMR relaxation rates are measured to provide electronic dynamics information which relates to magnetic relaxation in the solid state. Data is presented on two similar five-coordinate complexes (TpPh,MeCo(guaiacol) and TpPh,MeCo(thioguaiacol) (TpPh,Me = tris-3-phenyl-5-methyl-1-pyrazolylborate)). They differ by the substitution of one atom (S for O) coordinated to the central metal. Both complexes show approximate TBP geometry in crystalline form. In solution, TpPh,MeCo(thioguaiacol) is stable under all conditions, while TpPh,MeCo(guaiacol) is dynamic, converting between four- and five-coordination at higher temperatures.

POSTER 257
^{19}F-centered NMR Spectroscopy for the Analysis of Complex Mixtures

Presenting Author: Nicholle Bell

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We present NMR methodology that uses ^{19}F as a 'spy' for the structure determination of mono-fluorinated compounds in complex mixtures. This ^{19}F-centred NMR analysis consists of a complementary set of broadband, phase-sensitive NMR experiments that utilize the substantial sensitivity of ^{19}F and its far reaching heteronuclear couplings to obtain ^{1}H, ^{13}C and ^{19}F chemical shifts, values of J_{HF}, J_{HH}, and J_{FC} coupling constants and the sizes of ^{13}C induced ^{19}F isotopic shifts – parameters that underpin the structure elucidation process. This new methodology is illustrated on solving the structures of disinfectant by-products produced by chloramination of a single mono-fluorinated phenolic compound.

POSTER 258
NMR metabolomics based chemosensory differentiation of anti-obese phytocompounds
Presenting Author: Ankita Singh

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Obesity is a global epidemic. Recent research on phytochemicals and plants with nutritional and medicinal values has gained interest in obesity management. In this study, NMR metabolomics was used to differentiate 22 phytochemicals and 38 medicinal plants in pungent and non-pungent groups. Anti-lipase assays were conducted to study their anti-obesity properties. Multivariate analysis of NMR data demonstrated the potential of proton NMR metabolomics in identifying and differentiating medicinal plants and their active phytochemicals with anti-obesity properties which was confirmed with anti-lipase assays and alkaloid analysis.

POSTER 259
Identifying Individual Molecules in a Small Molecular Mixture via Unsupervised Analysis
Presenting Author: Madhur Srivastava

Complete Author List:
Aritro Sinha Roy (Cornell University); Madhur Srivastava (Cornell University)

Resolving small molecule mixtures by NMR spectroscopy has been of great interest for a long time for its precision, reproducibility, and efficiency. However, spectral analyses for such mixtures are often highly challenging due to overlapping resonance lines and limited chemical shift windows. The existing experimental and theoretical methods to produce shift NMR spectra in dealing with the problem have limited applicability owing to sensitivity issues, inconsistency, and/or the requirement of prior knowledge. We resolved the problem by decoupling multiplet structures in NMR spectra by the wavelet packet transform (WPT) technique, followed by a scheme to deploy the method in predicting the composition of the corresponding molecular mixtures from their 1H NMR spectra in an automated fashion.

POSTER 260
31P NMR Spectroscopy Parameters for Structural Analysis
Presenting Author: Markéta Tichotová

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Phosphorus is a biogenic element and a crucial component in modern organic synthesis. However, a stereogenic centre on the phosphorus atom causes formation of two enantiomers. These are difficult to separate, and stereoselective synthesis is usually challenging. The determination of the absolute configuration is also often hindered. Therefore, an NMR method able to assign the stereochemistry on phosphorus would be a solution.
In this work, we examine 31P NMR parameters for the structural analysis of model compounds. We complement experimental NMR data with quantum-chemical calculations. Furthermore, we present a new route for profound conformational sampling using artificial intelligence. 31P-13C J-coupling analysis unequivocally assigned the relative configuration, while 31P-based RDC analysis requires further investigation. We tested a new molecular-dynamics-based method, MDOC.

POSTER 261
^{15}N-Mediated J-Couplings in Conformational Analysis of ^{15}N-Glycomimetics
Presenting Author: Radek Pohl

Complete Author List:
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In this contribution, we would like to present a joint NMR & DFT study dealing with ^{15}N-labeled glycomimetics – compounds structurally resembling natural carbohydrates with a ^{15}N label in the position that is close to the location of the conformational change. The labeling provides extra NMR parameters, especially *J*-couplings that are sensitive to the conformation. The ^{15}N labeling enables exploring (a) aminomethyl group rotation, (b) aglycone conformation, or (c) N-disaccharide conformation. The approach combines molecular dynamics simulations, DFT calculations of NMR parameters, and fitting the calculated *J* couplings with experimental values. The results of the study reveal redundant ^{15}N-mediated *J*-couplings sensitive to conformational changes and show that the approach can predict conformers of such ^{15}N-glycomimetics in solution.

POSTER 262
Something from Nothing: Automated Compound Sparing NMR
Presenting Author: Francisco Silva

Complete Author List:
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clear Magnetic Resonance (NMR) Spectroscopy has played a key role in Pharmaceutical R& D as a gold standard in structure identity; however, the amount of material tends to be in the low milligrams. In our previous Direct-to-Weigh (DTW) NMR workflow, laborious NMR sample preparation was used where samples were manually weighed out from a standard vial and remaining valuable material was used in various screening cascades. The spent vial was then discarded with unweighable material. Here in a new Automated Compound Sparing (ACS) NMR, the spent unweighable vials were utilized as the source of NMR sample. A high recovery vial on a modified liquid handler, tasked with solvation and transfer, enabled automation of NMR sample preparation. A comparison of the

POSTER 263
NMR and Machine Learning Study of Crude Oil Stability
Presenting Author: Vilko Smrecki

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The stability of crude oils and their components is one of the biggest challenges in petroleum industry, as there is no single method to determine stability of all fractions. To explore possibilities to predict crude oil stability, statistical multi-way analysis (MWA) and machine learning (ML) methods were coupled with DOSY NMR spectroscopy and compared with various parameters affecting crude oil stability. An extensive ML multivariate linear regression was performed to model crude oil stability in terms of various measured properties, such as aromaticity, API gravity, percentage of aliphatic chains, asphaltene content and relative diffusivities. The correlations obtained for mixtures as complex as crude oil were exceptionally good, proving that this new and robust model can accurately predict crude oil stability.

POSTER 264
Changes in components of T2 relaxation in chicken breast meat after slaughtering
Presenting Author: Hong Zhuang

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Changes in T2 relaxation in chicken breast fillets (pectoralis major) were investigated during the conversion of muscle to meat and postmortem storage. Fillets were removed from bones within 5 min after bleeding and placed in ice. T2 relaxation in muscle was measured using Bruker LF-NMR 90II over the postmortem period (from 0.5 to 168 h). Results from distributed analysis show four components with relaxation times of 5, 42, 148, and 378 ms, respectively, at the early postmortem phase (< 24 h), but only three components were noted at the end of storage. Relative content of 42-ms component increased and 378-ms component decreased during the conversion of muscle to meat. Findings reveal that T2 components change both quantitatively and qualitatively postmortem.

POSTER 266
Unequivocal Identification of Two-bond Heteronuclear Correlations by i-HMBC to Facilitate the Elucidation of Complex Natural Product Structures at Nanomole Scale
Presenting Author: Mikhail Reibarkh

Complete Author List:
Mikhail Reibarkh (Merck); Xiao Wang (Merck); Yunyi Wang (Merck); Ryan Cohen (Merck); Guilherme Dal Poggetto (Merck)

HMBC is an essential experiment for determining multiple-bond heteronuclear correlations in organic molecules, including natural products, yet its major limitation is differentiating two-bond from longer-range correlations. Previous approaches to overcome this suffer drawbacks such as restricted utility and poor sensitivity. Here we present a sensitive and universal i-HMBC (isotope shift detection HMBC) methodology to identify two-bond HMBC correlations using isotope shifts. Experimental utility was demonstrated at the nanomole scale with only a few hours of acquisition time for structure elucidation of several complex proton-deficient natural products, which were otherwise too challenging for conventional 2D NMR experiments. i-HMBC overcomes the key limitation of HMBC without significant reduction in sensitivity and thus could replace the latter as an essential structure elucidation experiment.

POSTER 267
Quantifying Hidden Fluorine in Aqueous Film-Forming Foams via 19F NMR
Presenting Author: Esteban Hernandez

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Aqueous film-forming foams (AFFFs) are mixtures used in extinguishing high-hazard flammable liquid fires. These mixtures have historically contained long chain per- and polyfluoroalkyl substances (PFAS), which have been a topic of interest due to being linked with a variety of health problems. The use of AFFFs in both military (MilSpec) and commercial (non-MilSpec) applications has led to PFAS being found in high concentrations near population centers. Current analytical methods to quantify PFAS in AFFFs are often complicated, time-consuming, and expensive. As the concern of PFAS contamination grows, rapid and inexpensive methods are needed to quantify total fluorine in environmental mixtures. This work focuses on the use of 19F qNMR as a means to quickly and reliably deduce total fluorine concentration.

POSTER 268
Internuclear Distance Measurements between 1H and 14N in Multi-Component Rigid Solids at Fast MAS
Presenting Author: Yutaro Ogaeri

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1H-14N internuclear distances are readily and accurately measured using the symmetry-based phase modulated resonance-echo saturation-pulse double-resonance (PM-S-RESPDOR) method in rigid solids. Analytical equation of the fraction curve easily provides 1H-14N couplings. However, this treatment is only applicable when NH proton resonance is well separated from the other proton peaks, which is not necessarily satisfied even at fast MAS >60kHz, especially in multi-component systems. To overcome this problem, T-HMQC filtering is applied to suppress the 1H signals other than NH proton prior to the PM-S-RESPDOR experiments. The method is well demonstrated on two components acetaminophen-oxalic acid (APAP-OXA) systems.

POSTER 269
P-chirogenic Compounds Analyzed by 31P NMR Parameters
Presenting Author: Anna Hruzíková

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P-chirogenic molecules with another stereogenic center are present as a mixture of two diastereomers. Their separation and crystallization for determination of stereochemistry is challenging by X-ray diffraction. NMR spectra of 31P isotope can be easily recorded on commonly used NMR probes due to 100% natural abundance, spin ½ and high sensitivity. In this work, we search for new approaches employing 31P NMR parameters in structural analysis complemented by quantum-chemical calculations. At first, we used conformational sampling of studied DSIs to generate all potential conformers. The low-energy conformers were found by DFT calculations. Subsequently, we investigated the possibility of using 31P J-couplings and residual dipolar couplings (RDCs) to assign relative configuration on phosphorus.

POSTER 270
31P Derivatization and NMR Detection Strategies for Tannin Analysis
Presenting Author: Luke Fulton

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Tannins are biomolecules that present as mixtures of highly similar structures which poses a challenge for both general characterization and content determination. Three quantitation strategies were compared: PULCON, deconvolution, and 2D qNMR. PULCON does not have a reference compound alongside the tannin to simultaneously serve as a chemical monitor. For deconvolution, the extent of overlap contributes to less accuracy than typically associated with internal standard methods. 1H and 31P correlations observed through 2D NMR exhibited more overlap in the aliphatic region than was expected. Due to the lack of aromatic proton correlations, we investigated phenol as an alternative reference compound, an option not available to 1D methods.

POSTER 271
Eliminating Ambiguity in Small Molecule Structural Characterizations with J-Couplings and Computational Methods
Presenting Author: James Harper

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Certain J-couplings provide structural information (e.g. the Karplus relationship relating three-bond 1H/1H couplings (3JHH) to dihedral angle). However, finding other general rules has been challenging. The development of accurate computational methods for computing J-couplings provides an alternative path that involves building all feasible structures then comparing computed and experimental values. This process encompasses one, two and three bond couplings to identify unknown structures with high confidence. This poster describes measurements of 1JCC in two partially characterized natural products and provides information to finalize these characterizations. A second study provides complete structural characterization of a novel natural product using only 1JCC data and 81 candidate structures. Work evaluating the merit of 2JCH and 3JCH values in structural studies is also described.

THEORY COMPUTATION AND DATA PROCESSING IN NMR (Posters 272 – 302)

POSTER 272
Can Three-Site Relaxation-Exchange Maps be Asymmetric?
Presenting Author: Bernhard Bluemich

Complete Author List:
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Asymmetric relaxation maps in three-site exchange report circular flow between the relaxation sites. This disagrees with detailed balance according to which the exchange between any pair of sites must be balanced in thermodynamic equilibrium. Confined vacancy diffusion by random jumps on a 2D checkerboard grid and confined gas diffusion were modelled to explore the impact of topological constraints on diffusion. Both models produce density variations across the pore. Moreover, in equilibrium diffusion up to 1% of the molecules appear to move in circular paths near the walls. This motion may result from pore resonance corresponding to diffusion eigenmodes. If confirmed by experiment, then detailed balance of multi-site exchange does not necessarily apply when the exchange is impacted by topological constraints.

POSTER 273
Acquisitions with random shim values enhance AI-driven NMR shimming
Presenting Author: Moritz Becker

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Shimming is still a time-consuming and cumbersome burden preceding most NMR experiments. Meanwhile, artificial intelligence is a promising approach to speed up and improve signal-based shimming algorithms. We present multiple enhancements for the applicability of AI-driven shimming, focusing on reference sample peaks for benchtop magnets. Our improvements include randomized dataset collection, upscaling from linear shims to an additional higher-order shim, and a novel flexible neural network architecture. We utilize a temporal history combining previous spectra and random shim offsets to explore the shim space, before applying predictive shimming steps guided by our neural network. The application reduces the linewidth from ~ 4 Hz to below 1 Hz, within less than 10 acquisitions, and helps avoid local minima of traditional algorithms.

POSTER 274
REDEN: Multi-Fitting NMR Peaks Deconvolution-based Peak Picking Software
Presenting Author: Abigail Chiu

Complete Author List:
Abigail Chiu (University of Colorado Denver); Mehdi Rahimi (University of Colorado Denver); Woonghee Lee (University of Colorado Denver)

In NMR analysis, the accurate detection of signals, known as "peak picking," is essential for subsequent procedures. To address the issue of lower intensity peaks being obscured by more intense peaks, we present REDEN, a software that effectively identifies peaks in both 2D and 3D NMR spectra. Our integrated, cross-platform and open-source software can be accessed independently or as part of the Poky suite. It utilizes four fitting algorithms to deconvolute specified regions of an NMR spectrum, allowing for precise identification of all peaks. REDEN also provides options for fine-tuning and troubleshooting through Basic and Advanced modes. The advanced mode offers a wealth of data and fine-tuning capabilities, including 3D and contour plots of intermediate processing steps.

POSTER 275
Post-acquisition correction of NMR spectra distorted by dynamic and static field inhomogeneity of cryogen-free magnets
Presenting Author: Alexander Karabanov

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We describe analytical and numerical mathematical methods for post-acquisition correction of NMR spectra distorted by static and dynamic magnetic field inhomogeneity, typical for cryogen-free magnets. For the dynamic inhomogeneity, we apply a variant of the reference deconvolution method. For the static inhomogeneity, we apply the method of a delayed Fourier acquisition time. We verify our approach by post-processing experimental NMR spectra of liquid water and ethanol samples and obtain good results in both static and dynamic cases. This work complements our previous work on instrumental suppression of the cold head distortions. The results presented contribute to the general field of processing NMR spectra and serve towards a more extensive use of cryogen-free magnets in
high-resolution NMR spectroscopy.

POSTER 276
Automated Chemical Shift Assignments of MAS Solid-State NMR Spectra of Complex Protein Systems by ssPINE/ssPINE-POKY
Presenting Author: Andrea Estefania Lopez Giraldo

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Andrea Lopez Giraldo (University of Colorado Denver); Adilakshmi Dwarasala (University of Colorado Denver); Mehdi Rahimi (University of Colorado Denver); John L. Markley (University of WisconsinMadison); Woonghee Lee (University of Colorado Denver)

We have developed a software package called ssPINE that automates the process of recognizing, categorizing, and assigning signals from various types of multidimensional ssNMR spectra of proteins. Additionally, we have developed a graphical user interface called ssPINE-POKY, which is a plugin integrated with the POKY suite. This interface allows users to easily submit ssPINE jobs, visualize, verify, download, and browse results. The combined package supports automated peak assignments and facilitates three-dimensional structure calculations. The ssPINE web server is available as a free alpha version accessed through a web submission form.

POSTER 277
The Free Energy Landscape of Flexible Molecules studied using MD Simulations with Tensorial Orientational Constraints
Presenting Author: Ulrich Sternberg

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Dipolar interactions are often measured as RDC tensors encoding orientations of molecules or molecular groups with respect to the external magnetic field. Used as constraints in MD simulations, the RDC tensors initiate molecular re-orientations and rotations of molecular groups. Such MDOC simulations can be augmented by scalar NMR parameters as 3J-couplings and NOE distances. They generate trajectories of molecules at ambient temperatures that are controlled by the energy and the entropy terms of the system. MDOC simulations demonstrated their power in elucidating chiral configurations, NMR parameter assignments and conformer distributions even for highly flexible molecules. In well studied molecules like strychnine new conformers were found that appear only with rising entropy.

POSTER 278
Towards an Unlimited Number of Analytical Solutions for the Bloch Equations
Presenting Author: Christian Bonhomme

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christian bonhomme (Sorbonne University); Pierre-Louis Giscard (ULCO)

In this presentation, we tackle the problem of finding new analytical solutions for the Bloch equations, distinct from the celebrated Rabi and Rosen-Zener models. The solutions are obtained by combining the newly developed Path-Sum approach and reverse-engineering of the system of coupled linear differential equations. An unlimited number of analytical solutions is obtained consequently.

POSTER 279
The POKY Suite for Biomolecular Solution and Solid-State NMR Spectroscopy
Presenting Author: Woonghee Lee

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POKY is a professional-grade software suite for NMR-based structural biology and drug design. It offers advanced algorithms and modern technologies, including artificial intelligence and machine learning, to maximize user experience and productivity. POKY supports a wide range of NMR data and experiments, and can be easily integrated with other tools and databases. It is suitable for researchers and developers working in structural biology and drug design, and supports recent advances in the field, including 13C-detected solution NMR for intrinsically disordered proteins and 1H/13C/19F detected solid-state NMR for large, insoluble, and membrane proteins. POKY is freely available at https://poky.clas.ucdenver.edu and includes web servers and plugins for automated assignments and structure calculation.

POSTER 280
Broadband Adiabatic Inversion Cross-Polarization: Theory and Applications
Presenting Author: James J. Kimball

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Large anisotropic interactions make it challenging to acquire uniform ultra-wideline (UW) solid-state NMR spectra with high S/N ratios. Conventional CP experiments are well known to aid in signal enhancement; however, they typically operate over limited bandwidths. The broadband adiabatic-inversion cross-polarization (BRAIN-CP) sequence, which utilizes WURST pulses for polarization transfer, has been shown to compensate for these bandwidth limitations; however, to date, comprehensive experimental and theoretical investigations have only been conducted for static spectra of spin-1/2 and spin-1 nuclei. In this work, we demonstrate significant broadband CP enhancements for both static UW NMR spectra of half-integer quadrupolar nuclei and MAS spectra of spin-1/2 nuclei. Analytical and numerical simulations, along with applications to extremely unreceptive nuclei (99Ru and 103Rh), are presented.

POSTER 281
EM simulation-based detector modeling and pulse compensation for parallel NMR experiments
Presenting Author: Mengjia He

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In parallel NMR probes, radio frequency interference becomes a significant problem due to the coil's inductive coupling and matching network's enhancement, especially among the open coil system without excellent electrical isolation\cite{kupceParallel2021b}. This issue may cause pulse sequence coupling and signal transfer between multiple homonuclear channels. Based on the electromagnetic simulation, we present a theoretical framework to evaluate the coupling effects, calibrate the parallel pulse sequences with compensated cooperative pulse and split the composited FIDs from multiple channels. These results would help walk close to the capacity limit of designing parallel NMR probe in fixed magnet system.

POSTER 282
Zero- to Ultra-low field NMR: Probing the Selection Rules of Angular Momentum
Presenting Author: Adam Ortmeier

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Zero- to Ultra-low Field (ZULF) NMR is an emerging tool that can be used as a cheap alternative to conventional high-field NMR for a fraction of the cost without forfeiting sensitivity to molecular structure. Another remarkable aspect of ZULF NMR is that it creates spaces where the J-coupling Hamiltonian is the predominant interaction of the system. In this system, at ultra-low field, the coupled basis's energy states are close enough together where we can probe them. This gives us a unique opportunity to look at the selection rules of angular momentum. Where, depending on the axis of detection relative to the axis of a weak perturbing magnetic field, we observe different ZULF NMR spectra.

POSTER 283
Robust Automated Backbone Triple Resonance NMR Assignments of Proteins Using Bayesian-Based Simulated Annealing
Presenting Author: Anthony C Bishop

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Assignment of individual resonances of nuclear magnetic resonance (NMR) spectra to specific atoms within a protein remains a labor-intensive and often challenging task. Here we present a new algorithm BARASA for the automated assignment of backbone triple resonance spectra of proteins. The algorithm utilizes a Bayesian statistical analysis of predicted and observed chemical shifts in combination with simulated annealing to search for an optimal solution. The algorithm is tested against systems ranging in size to over 450 amino acids including examples of intrinsically disordered proteins (IDPs). BARASA is robust, accommodates incomplete and incorrect information, is sufficiently fast to allow for real-time evaluation of data acquisition, and outperforms currently employed algorithms – especially in cases of sparse data.

POSTER 284
NMR Metabolite Quantification by Neural Networks Using Explainable AI Approach
Presenting Author: Hayden Johnson

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Neural networks show promise for improving speed and automation of data processing for quantitative NMR analysis; however, neural networks are commonly thought of as a black-box approach – potentially discouraging usage in research. We examine this issue in a metabolomics context by generating a dataset from simulated metabolite signals, training a multi-layered perceptron for metabolite quantification, and using the integrated gradients method to facilitate interpretation of our model. Using integrated gradients, we obtain scores attributing model predictions to input features to gain insight into how spectral intensity at each chemical shift contributes to model-estimated concentrations. Results show the model recognizes target metabolite signals in a mixture spectrum and determine concentrations by assessing relative peak intensities, much like a human spectroscopist.

POSTER 285
NUScon: New Tools for Evaluating and Optimizing Data Processing on Nonuniformly Sampled NMR
Presenting Author: Adam Schuyler

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Darien Craft (UConn Health); Adam Schuyler (UConn Health)

NUScon (nuscon.org) was founded as a community-driven contest with open challenges for reconstructing spectra from nonuniformly sampled (NUS) experiments. The original NUScon evaluation workflow starts with uniformly sampled NMR data, injects synthetic peaks, subsamples to produce NUS challenge data, reconstructs the spectrum with contestant scripts, and tests how the resulting spectra recover the injected peaks. We present here new modules to expand the scope of NUScon to evaluate sampling schemes and peak pickers. The NUScon software enables anyone to easily explore the modular tasks and evaluate how they impact spectral quality, thereby improving how we design and process NMR experiments. The NUScon software is delivered on the NMRbox platform (nmrbox.org), so any of its 250+ software packages can be utilized.

POSTER 286
Fastcoords for Structural Biology Surveys in NMRbox
Presenting Author: Hamid R. Eghbalnia

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Computation plays a critical role in nuclear magnetic resonance spectroscopy (NMR) applications in structural biology, and more broadly, all applications of structural biology. Many advancements have been enabled by access to data archives such as the Protein Data Bank (PDB), the Biological Magnetic Resonance Data Bank, and the AlphaFold Structural Database (ASB). Here we describe fastcoords, a software package that builds on the computational resources available on NMRbox (NMRbox.org) to simplify and accelerate search and analysis of structural data in archives such as the PDB and ASB, as well as federation of data from different resources. We demonstrate the significant gains in computational speed by performing search and processing operations on approximately 500 thousand AlphaFold protein structures.

POSTER 287
Molecular Dynamics Approach in Modeling NMR Lineshape of non-Markovian Fluids
Presenting Author: Mohamad Niknam

Complete Author List:
Mohamad Niknam (UCLA); Louis S. Bouchard (UCLA)

Direct measurements of non-Markovian self-diffusion behavior are, to our knowledge, not currently possible with existing methods. In this work, we look at non-Markovian dynamics of diffusing spins with Molecular Dynamics simulations, present two methods for the prediction of NMR lineshape, and compare the results to the experimental observations. In the second part, we study the relationship between the lineshape and temperature, wall separation, and interaction with the wall.

POSTER 288
Quadrupolar NMR Crystallography – Crystal Structure Prediction (QNMRX-CSP)
Presenting Author: Robert Schurko

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A new Quadrupolar NMR Crystallography Crystal Structure Prediction (QNMRX-CSP) protocol will be presented, which uses experimentally measured and theoretically determined 35Cl electric field gradient (EFG) tensor parameters for the prediction, refinement, and validation of crystal structures of organic HCl salts, including pharmaceutical compounds. The protocol comprises three modules, which feature methods for molecular fragment selection, crystal packing with Monte-Carlo simulated annealing, and plane-wave DFT calculations (of EFG parameters and for structural refinement). Aspects including benchmarking, blind predictions of unknown structures, determinations of uncertainties of atomic positions, and possible applications using other quadrupolar nuclei (e.g., 14N, 17O, and 23Na) will be discussed.

POSTER 289
ChemisTwin: A Novel Online Platform for electronic Reference Materials for NMR applications
Presenting Author: Albert Farre Perez

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ChemisTwin is a digital portal which will contain an extensive database of electronic reference materials (eRM) acting as digital twins of the physical reference materials. These eRM are produced from physical certified reference materials (CRM) and the eRM is based on a digital package of datasets that defines a physical material. The use eRMs in the platform allows customers to perform the identity verification and content determination of their analytes of interest by uploading the raw data from the corresponding instrument. This solution allows the customer to automatically compare their sample with the eRM and provides a detailed report. This product provides our customers with a readily available, more sustainable, significantly less involved, and more error-proof solution.

POSTER 290
Flow Encoding Established by Optimal Control RF Pulse
Presenting Author: Mehrdad Alinaghian Jouzdani

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Mehrdad Alinaghian Jouzdani (PhD Student); Mazin Jouda (Dr); Jan Gerrit Korvink (Professor)

Flow encoding MRI is traditionally achieved by applying bipolar gradients. In this study, we show that velocity can be encoded into phase during the excitation time. First, a mathematical model is designed. Then, optimal control (OC) theory is used to design the required flow encoding RF pulse. Also, GRAPE algorithm is employed to minimize the cost function in the OC problem, and a constraint is added to make the RF pulse slice selective. This method enables one to achieve shorter echo times hence enhance signal-to-noise ratio. Furthermore, a non-linear relation can be made which improves phase-SNR of regions with lower flow rates, for example close to vessel walls.

POSTER 291
Network for Advanced NMR Data Handling
Presenting Author: Chris Bontempi

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The Network for Advanced NMR (NAN) provides many resources for simplifying and democratizing the use of high-field NMR. Data Transport automatically transmits acquisition data (VNMRJ/OpenVJMR and TopSpin) to a central repository. A sophisticated Data Browser allows users to browse and manage the repository contents and seamlessly integrates with NMRBox, BMRB and Globus. The Knowledge Bases allow users of all levels to conduct new experiments and better understand the process, while allowing experts to share their vast experience with the community. Resource Connector allows users to see the spectrometers and other resources available for their use. Dashboards ease administrative tasks and provide visibility into all aspects of the facilities and the system. NAN aims to be essential to any NMR lab.

POSTER 292
Structure-Based Resonance Assignment Strategy for Sparsely Labeled Proteins
Presenting Author: James H. Prestegard

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Recent advances in computational prediction of protein structure offer new opportunities for NMR characterization of ligand binding, protein-protein interaction and dynamics. However, assignment of NMR resonances reporting on these phenomena is a prerequisite. Traditional triple resonance assignment often requires expression in bacterial hosts, precluding work on proteins requiring extensive post-translational modification (glycoproteins) or folding chaperones, and for large proteins extensive overlap of resonances from uniform labeling can limit resolution. Here, we illustrate a sparse-labeling strategy applicable to mammalian cell expression along with a software tool (ASSIGN_SLP) for sequence specific assignment of resonances. New additions to the tool, including the use of data dependent on paramagnetic tags and the use of accelerated molecular dynamics to account for internal motion are discussed.

POSTER 293
The BMRB archive of Protein, Nucleic Acid and Metabolite NMR Data
Presenting Author: Kumaran Baskaran

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Biological Magnetic Resonance data Bank (BMRB: https://bmrb.io) serves the biomolecular NMR community by maintaining a curated archive of primary and derived data and metadata linked to scientific investigations under the "FAIR Principles" (Findable, Accessible, Interoperable, and Reusable). The goal of BMRB is to empower scientists in their analysis of the structure, dynamics, and chemistry of biomolecular systems and to support further developments in the field of biomolecular NMR spectroscopy. BMRB is a member and a core archive of the Worldwide Protein Data Bank (wwPDB: https://www.wwpdb.org). As of February 2023, BMRB holds over 11 million chemical shifts from 15729 macromolecule entries and a library of carefully curated NMR spectroscopic data of over 1000 small molecules.

POSTER 294
Towards Shorter Composite Refocusing and Inversion Pulses for NMR
Presenting Author: Stephen Wimperis

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Stephen Wimperis (Department of Chemistry, Lancaster University)

Novel composite 180° pulses are designed for NMR and MRI. Rather than being constructed from 180° pulses, the new sequences are constructed from 90° pulses, with the aim of finding shorter sequences overall. The primary focus is on composite pulses that are dual compensated – broadband with respect to both rf inhomogeneity and resonance offset – and have antisymmetric phase schemes, hence forming spin echoes without phase errors. In particular, new dual-compensated refocusing pulses are presented that are constructed from ten 90° pulses, whereas the existing equivalents effectively consist of eighteen 90° pulses. The use of 90° pulses creates a number of theoretical difficulties for the design process and these are tackled here using average Hamiltonian theory and numerical searching.

POSTER 295
A Flexible and Automated Approach to NMR Spectral Assignment using Full Bayesian Inference
Presenting Author: Joseph Courtney

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Assigning NMR spectra can be challenging, especially for complex systems like Intrinsically Disordered Proteins (IDPs) and high-molecular weight complexes. Existing algorithms like Probabilistic Interaction Network of Evidence (PINE) are fast and accurate for solution-state spectra of small globular proteins but becomes less useful for larger and more complex molecules. To address this challenge, we have developed a flexible approach to NMR spectral assignment using full Bayesian inference based on probabilistic programming. Our approach is more general and flexible, enabling the inclusion of additional prior knowledge and new experimental schemes. Utilizing probabilistic programming enables the evaluation of a wide range of hypothetical structures and assignments, providing a powerful tool for NMR analysis.

POSTER 296
SelEx – a fast and easily setup 1D exchange NMR spectroscopy experiment
Presenting Author: Markus Rotzinger

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Markus Rotzinger (University of Graz); Nathalie Schuster (University of Graz); Klaus Zangger (University of Graz)

Observing chemical exchange in a variety of media is a challenge occasionally associated with isotope exchange to monitor dynamic processes. One frequently used method is EXchange SpectroscopY (EXSY) which gives information about chemical exchange processes on a variety of timescales. EXSY requires the acquisition of time-consuming two-dimensional spectra. In this work we provide a faster alternative, via an experiment which uses spatial encoding to extract similar information in a 1D experiment. Therein, all protons are observed at once, but in different slices of the detection volume. The experiment can be carried out in a single scan to identify exchanging sites in a 1D spectrum.

POSTER 297
Quantum optimal control module of Spinach library
Presenting Author: Ilya Kuprov

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In magnetic resonance, optimal control theory is used to generate pulses and pulse sequences that achieve instrumentally difficult objectives (for example, uniform 13C excitation in a 1.2 GHz magnet) with high precision under stringent time and radiofrequency/microwave power constraints. At the moment, the most popular framework is GRAPE (gradient ascent pulse engineering, 10.1016/j.jmr.2004.11.004).
This poster reports our recent mathematical and software engineering work on the various extensions and refinements of the GRAPE framework, and on its implementation as a module of Spinach library. Recently implemented functionality includes: fidelity Hessians and regularised Newton-Raphson optimisation, generalised curvilinear waveform parametrisation, prefix and suffix pulse sequences, multi-target and subspace control, keyhole states and subspaces, cooperative pulses and phase cycles, and piecewise-linear control sequences.

POSTER 298
Sensitivity of Nonuniform Sampling Experiments
Presenting Author: Yulia Pustovalova

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All common techniques for spectral reconstruction of multidimensional NMR experiments employing nonuniform sampling (NUS) are nonlinear. Low-intensity signals are often lost during spectrum reconstruction. Increasing the number of transients can improve sensitivity at the expense of lower NUS coverage. However, there is a trade-off between the number of sampled points and transients for time-limited experiments. The standard metric for uniformly sampled data, signal-to-noise ratio, is unreliable for NUS spectra reconstruction. So, here we use intrinsic receiver operating characteristic (IROC) analysis to explore the nonlinearities caused by NUS reconstruction algorithms, NUS sampling and noise level. We find evidence for noise-dependent phase transitions reminiscent of the Donoho-Tanner phase transition for nonuniform sampling coverage.

POSTER 299
Improved Signal Detection in Magnetic Resonance with Pseudorandom Phase Encoding
Presenting Author: Michael W. Malone

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Magnetic resonance based signal detection can be greatly complicated by radio frequency (RF) interference, especially when frequency components of the interference are close to the target signal. By pseudorandomly varying the phase of a magnetic resonance signal, however, a "fingerprint" can be encoded in the target signal that distinguishes it from the RF interference. We describe a modification to the Carr-Purcell Meiboom-Gill sequence and show how it allows us to reject strong interference even at our target signal frequency. This has the potential to improve fieldable deployments of magnetic resonance systems for substance detection.

POSTER 300
A New Generation of NMR Data Processing Software for a New Generation of Chemists
Presenting Author: Alex Waked

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To close the skills gap reported in chemical sciences graduates, students need educational experiences that mimic their future careers. However, logistical and financial barriers often make such work difficult to replicate in an academic environment. In particular, NMR and other analytical data processing software have historically been difficult to implement in chemical education. In response, we present the first commercially available browser-based NMR and hyphenated chromatography/MS processing application—Spectrus JS. Designed to address the barriers presented by its predecessors, it provides a convenient and cost-effective way to deploy and access NMR and analytical data processing tools in academic environments, helping educators better equip the next generation of chemists.

POSTER 301
Analyzing the use of \textit{in situ} Receiver Operator Characteristic to Evaluate Nonuniform Sample Reconstructions
Presenting Author: D. Levi Craft

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Nonuniform sampling (NUS) has allowed spectroscopists to tailor their experiments to reduce data collection time and improve spectral quality. The Nonuniform Sampling and Reconstruction Contest (NUScon) has released a workflow for evaluating spectral reconstructions. We extend this workflow to include a new evaluation metric using \textit{in situ} receiver operating characteristic (IROC). This study evaluates how to interpret the results of an IROC analysis alongside the standard approach of computing a point-spread-function and calculating its peak-to-side-lobe ratio, a known standard for approximating the relative intensity of signals to artifacts introduced into a reconstructed spectrum. This study presents tools for assessing spectral quality and optimizing ML-based techniques, which served to jump start a spectroscopist's workflow.

POSTER 302
Nutation-Based Longitudinal Sensing Protocols for High-Field NMR With Nitrogen Vacancy Centers in Diamond
Presenting Author: Declan Daly

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Nitrogen vacancy (NV) centers in diamond allow for accessible NMR experiments on samples of just a few picoliters. NV-NMR has been unable to work in the high-field regime due to the challenge of making NV's sensitive to high frequency Larmor signals. We investigate the experimental viability of NV-NMR at high field with a new experimental protocol called DRACAERIS (Double Rewind ACquisition Amplitude Encoded Radio Induced Signal). We will discuss how finite pulse lengths and spin-spin couplings affect the resulting NMR spectra and identify reasonable experimental parameters. Additionally, we will highlight recent progress towards the experimental realization of DRACAERIS NMR.

LATE

POSTER 303
Contrasting lanthanide and actinide complexation by polyoxometalates via solution-state NMR
Presenting Author: Christopher Colla

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Deciphering the solution chemistry and speciation of actinides is inherently difficult due to radioactivity, rarity, and cost constraints, especially for transplutonium elements. In this context, the development of new chelating platforms for actinides and associated spectroscopic techniques is particularly important. In this study, we investigate a relatively overlooked class of chelators for actinide binding, namely the polyoxometalates (POMs). We provide the first NMR measurements on americium-POM and curium-POM complexes, using 1D 31P NMR, variable temperature NMR, and spin-lattice relaxation time (T1) experiments. Reaction constants, reaction enthalpy, and reaction entropy were extrapolated from the NMR data. The proposed POM-NMR approach allows for the study of trivalent f-elements even using microgram amounts and in phosphate-containing solutions where they are typically insoluble.