CONTENTS

Major Conference Sponsors

BRUKER

JEOL
Solutions for Innovation

Major Sponsor of the Student Travel Stipend Program

NORELL®

The student travel stipends are made possible by major program sponsor **Norell** with additional support from the following: Annaida Technologies, Cambridge Isotope Labs, Doty Scientific, Isotec/MilliporeSigma, New Era Enterprises, PhoenixNMR, and Invited Speakers.

GENERAL INFORMATION

PROGRAM. Short abstracts for the talks and posters appear in this document and are arranged in the order of presentation. Long abstracts may be viewed in the mobile app and desktop online planner. Session recordings of all orals will be available in the mobile app and online planner approx. 24 hours after sessions conclude. These will remain available through June 1.

POSTERS. Posters are located in the Fireside Pavilion (underground garage) along Asilomar Blvd. All posters should be set up by 8:30 AM on Monday and removed at 4:00 PM on Thursday. Please place posters in the poster space number shown in this program. Authors are encouraged to attend posters according to this schedule:

- Monday and Wednesday: Authors of posters in odd numbered spaces (001, 003, 005, etc).
- Tuesday and Thursday: Authors of posters in even numbered spaces (002, 004, 006 etc).

TALKS. Sessions are in Merrill Hall or Chapel. There will be an ENC Speaker PC and Mac (with the latest OS and software) on stage for presentations. **Please bring your presentation on a USB drive at least 30 minutes before your session to load onto an ENC speaker laptop.** You can even load your presentation the day before your session, *the earlier the better!* Name your file with your name and code (i.e. John Doe MOA 830). *Use your own laptop as a last resort.* The aspect ratio of the screen is 16 x 9.

There is a speaker test station set-up in Triton (the registration building) with same laptops used in each session rooms.

PROGRAM HIGHLIGHTS

Sunday, 2:30 – 4:00 PM, Merrill Hall
Young Scientists Symposium in honor of Suraj Manrao

Monday - Thursday, 7:00 - 7:50 AM, Merrill Hall
Early Morning Lecture Series by Warren Warren

Thursday, 4:00 – 5:35 PM, Merrill Hall
Machine Learning Tutorial Session

FRIDAY BOX LUNCHES. If you are lodging at Asilomar and checking-out on Friday, there will be box lunch available for you to pickup on Friday morning in Crocker Dining Hall at 7:30am. Ask attendant taking tickets on Friday morning for assistance.

HOSPITALITY SUITES & EXHIBIT BOOTHS. Vendor hospitality suites are open in the evenings and are located throughout the Asilomar grounds. Exhibit booths are in the Fireside Underground (underground garage) across Asilomar Blvd from the main Asilomar entrance. Use the mobile app to view Maps OR from an exhibitor listing tap the location icon for a pin drop.

ADVENTURE RAFFLE. Embark on an exhilarating journey around the Asilomar campus as you visit each vendor hospitality suite and exhibit booth with our Explore the ENC Adventure Raffle! Open 'Adventure Raffle' on the mobile app to scan and play.

SPECIAL EVENTS & INTEREST-DRIVEN GATHERINGS

Sunday, 4:00 - 6:00 PM, Fireside Pavilion (Poster Garage)
Welcome Gathering, Fireside Pavilion (Underground Poster Garage). All registrants are invited. No extra ticket is required, only your ENC name badge. Please use the drink ticket tucked into your name badge holder for wine or beer.

Regular Asilomar dinner begins at 6:00 pm for those lodging on-site.

Monday, 1:15 – 2:00 PM, Chapel
International Emerging Female Leaders in Biological Magnetic Resonance Forum, All are welcome.

Monday, 7:00 – 8:30 PM, Chapel
AMMRL, All are welcome.

Monday, 9:00 – 10:30 PM, Chapel
OCMRS, All are welcome.

Tuesday, 1:00 – 2:00 PM, Chapel
Ultra High-Field Workshop, All are welcome.

Thursday, 6:30 - 9:30 PM
Evening at the Barns at Cooper Molera in downtown Monterey. Ticket includes transportation and buffet dinner. Buses will start at 6:15 PM from behind the Social Hall. Event ticket required for bus. Cost: $75. Wear comfortable shoes and warm jacket. **Ticket sales close at 2pm on Monday.**

Regular dinner service will be offered for those lodging at Asilomar but who have not purchased tickets.

EMPLOYMENT CENTER. Look for special poster boards in the 'aclove' area of the Fireside Pavilion (underground poster garage) located in the exhibit booth area. Use these boards to post employment notices. If you are seeking a position, please bring at least 3-5 copies of your cv and place in the 'CV Binder' located on tables in the alcove.

ANIL KUMAR TRAVEL AWARD

The ENC Anil Kumar Travel Award is created to honor Professor Anil Kumar, currently Professor Emeritus, Department of Physics, Indian Institute of Science (I.I.Sc.), Bengaluru, Karnataka, India. Prof. Anil Kumar obtained his Master's degree from Agra University (1961) and PhD degree in 1969 from the Indian Institute of Technology, Kanpur, under the supervision of Prof. B.D.N. Rao. He did his Post-doctoral research in the United States at the Georgia Institute of Technology (Sydney Gordon) and at the University of North Carolina (Charles Johnson). He then moved to the laboratory of Prof. R.R. Ernst, (ETH Zurich) where he performed the first 2D NMR experiment in liquids and the first 2D Fourier imaging experiment. He was also the first one to observe transient oscillations in cross-polarization dynamics in solids. Anil Kumar returned to India and joined Indian Institute of Science, Bangalore as a Faculty member in January 1977. Here he started to work on further developments in two-dimensional NMR spectroscopy. He returned to Zurich for the academic year 1979-80, in a joint project of Prof. Ernst and Prof. Wuthrich. During this period, he was the first one to apply the two-dimensional Nuclear Overhauser Effect experiment, (popularly known as NOESY) in a protein, which opened the field for the determination of three-dimensional structures of biomolecules in solution by NMR. In his laboratory in India, he continued to explore challenging problems such as the study of cross-correlations in relaxation. He has major contributions in the field of NMR quantum information processing and NMR quantum computing and has pioneered several experimental aspects. Additionally, Prof. Anil Kumar has played a major role in popularizing the power of the modern NMR methodology and has been largely responsible for the current wide-spread use of the technique in India.

The Anil Kumar Travel Award of $1,000 is funded with contributions from Professor Anil Kumar's former Ph.D. students, collaborators, short-term workers, colleagues, and well-wishers. The Travel Award will be focused on reflecting Prof. Anil Kumar's broad interests in the development of multi-dimensional NMR methods and the application of NMR in chemical, biological and imaging sciences.

The 2024 recipient of the ENC Anil Kumar Travel Award is **Jacob Lindale**, Ph.D., Duke University. *This award will be presented on Thursday afternoon, 3:20pm in Merrill Hall (immediately following the Tutorial Session).*

MANRAO-RASTOGI TRAVEL AWARDS FOR YOUNG SCIENTISTS

Dr. Suraj Manrao is a longtime supporter of young researchers at the ENC. This new initiative with Dr. Vinit Rastogi is envisioned to specifically support Indian students and postdocs with costs associated with conference attendance. Generously funded by Suraj Manrao and Vinit Rastogi.

The 2024 recipients of the Manrao-Rastogi Travel Awards for Young Scientists

These awards will be presented on Thursday afternoon, 3:20pm in Merrill Hall (immediately following the Tutorial Session).

Fatema Bhinderwala, Ph.D.
University of Pittsburgh

Ritik Modi
George Mason University

Arka Prabha Ray
University of Florida

MELANIE ROSAY STUDENT & POSTDOC SUPPORT PROGRAM

Dr. Melanie Rosay was a scientific and industry leader in NMR, in particular, for developing instruments methods and applications of solid-state DNP. Nearly 50 labs worldwide now run instruments that she developed working with colleagues at Bruker, MIT and CPI (Communications and Power Industries). Melanie was an enthusiastic force who enabled and participated in a wide range of NMR advances, and also a great friend in the community. In particular, she was a genuine supporter of young scientists, and the Melanie Rosay Travel Awards suitably honors her interest and enjoyment of the energy and innovation they continually breathe into the field of hyperpolarized NMR. The awards are $1,000 each and are funded by Bruker.

The 2024 recipients of the Melanie Rosay Student & Postdoc Support Program

These awards will be presented on Thursday afternoon, 3:20pm in Merrill Hall (immediately following the Tutorial Session).

Nesreen Elathram
University of California, San Diego

Adam Altenhof, Ph.D.
Los Alamos National Laboratory

SHIMON VEGA TRAVEL AWARD

The Shimon Vega Travel Award was established by the Weizmann Institute of Science in Israel in memory of Prof. S. Vega, who for many decades was an active lecturer and participant at the ENC. The travel award consists of $1,000 and will be given to a PhD student or postdoctoral fellow researching in the fields of solid state NMR and/or DNP.

The 2024 recipient of the Shimon Vega Travel Award is **James Kimball**, Florida State University. *This award will be presented on Thursday afternoon, 3:20pm in Merrill Hall (immediately following the Tutorial Session).*

JMR/JMRO AWARDS

Sponsored by Journal of Magnetic Resonance, Elsevier Science Publishing

The annual JMR/JMRO Awards are selected from abstracts submitted by graduate students or postdoctoral fellows. Each award includes a prize of $500 funded by Elsevier Science Publishing.

2024 JMR / JMRO Award Receipients.

These awards will be presented on Thursday afternoon, 3:20pm in Merrill Hall (immediately following the Tutorial Session).

Shannon Eriksson
Duke University

Hannah Gerbeth
German Cancer Research Ctr

Jihyun Kim
Weizmann Institute of Science

Christopher Williams
University of California, Riverside

ENC STUDENT TRAVEL STIPENDS

The student travel stipends are made possible by major program sponsor Norell with additional support from the following:
Annaida Technologies, Cambridge Isotope Labs, Doty Scientific, Isotec/MilliporeSigma,
New Era Enterprises, PhoenixNMR, and Invited Speakers.

NORELL®

IMAGINATION - INNOVATION - RESULTS...
...IN SCIENCE AND IN BUSINESS

2024 Student Travel Stipend Recipients

Mustapha Abdulmojeed
North Carolina State University

Abubakar Abdurraheem
Wayne State University

Kathrin Aebischer
ETH Zurich

Caitlyn Agee
U. of North Carolina Wilmington

Luisa Almeida
Sao Paulo University

Jaafar Ansari
George Mason University

Emma Borthwick
University of St Andrews

Isaac Eason
Texas Tech University

Ioana Fidel
Extreme Light Infrastructure Nuclear-Physics

Simon Fleischer
Karlsruhe Inst. of Technology

Emma Gates
University of Manchester

Daniel Gebrezgiabhier
U. of California, San Francisco

Alexandria Guinness
Syracuse University

David Hernandez
Charite Universität Berlin

Beining Jin
University of Florida

David Joseph
Max Planck Institute

June Kenyaga
SUNY at Binghamton

Samuel Lehr
German Cancer Research Ctr

Yuan Li
San Diego State University

Yunke Liu
Rice University

Daniel Lysak
University of Toronto

Kelsey Marr
New York University

Shiraz Nantogma
Wayne State University

Lorenzo Niccoli
CERM - University of Florence

Rajan Rai
Clemson University

Ishani Senanayake
Southern Illinois University Carbondale

Spiridon Sevdalis
University of Maryland School of Medicine

Florin Teleanu
New York University

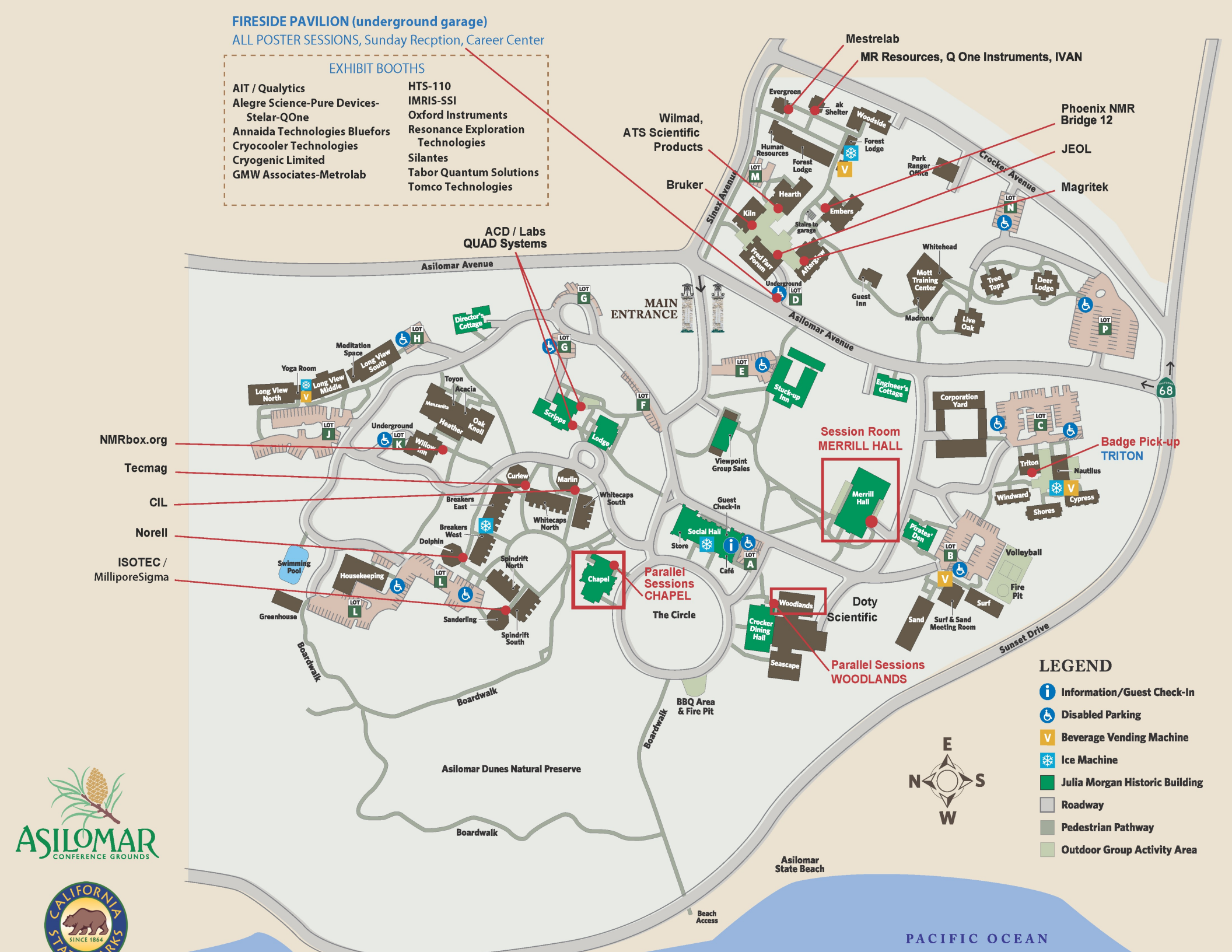
Jake Williams
University of Chicago

Embark on an exhilarating journey around the Asilomar campus as you visit each vendor hospitality suite and exhibit booth with our **Explore the ENC Adventure Raffle**!

Open 'Adventure Raffle' on the mobile app to scan and play.

EXHIBITOR		LOCATION ON ASILOMAR CAMPUS
ACD/Labs		Scripps Living Room (Hosp Suite)
AIT / Qualytics		Exhibit Booth 8 (Fireside Garage w-Posters)
Alegre Science-Pure Devices-Stelar-QOne		Exhibit Booth 3-4 (Fireside Garage w-Posters)
Annaida Technologies		Exhibit Booth 7 (Fireside Garage w-Posters)
Bluefors Cryocooler Technologies, Inc.		Exhibit Booth 14 (Fireside Garage w-Posters)
Bruker		Kiln (Hosp Suite)
Cambridge Isotope Labs		Marlin (Hosp Suite)
Cryogenic Limited		Exhibit Booth 6 (Fireside Garage w-Posters)
Doty Scientific		Surf & Sand (Hosp Suite)
GMW Associates - Metrolab		Exhibit Booth 2 (Fireside Garage w-Posters)
HTS-110 LP		Exhibit Booth 10 (Fireside Garage w-Posters)
IMRIS-SSI		Exhibit Booth 13 (Fireside Garage w-Posters)
ISOTEC/MilliporeSigma		Sanderling (Hosp Suite)
JEOL USA, Inc.		Fred Farr (Hosp Suite)
Magritek Inc.		Afterglow (Hosp Suite)
Mestrelab Research		Evergreen (Hosp Suite)
MR Resources - QOne - IVAN		Oak Shelter (Hosp Suite)
National NMR Resources in the Cloud		Willow Living Room (Hosp Suite)
Norell, Inc.		Dolphin (Hosp Suite)
Oxford Instruments		Exhibit Booth 11 (Fireside Garage w-Posters)
PhoenixNMR - Bridge 12 Technologies		Embers Living Room (Hosp Suite)
QUAD Systems		Scripps Conference Room (Hosp Suite)
Resonance Exploration Technologies		Exhibit Booth 12 (Fireside Garage w-Posters)
Silantes GmbH		Exhibit Booth 9 (Fireside Garage w-Posters)
Tabor Quantum Solutions		Exhibit Booth 5 (Fireside Garage w-Posters)
Tecmag, Inc		Curlew (Hosp Suite)
Tomco Technologies		Exhibit Booth 1 (Fireside Garage w-Posters)
Wilmad, ATS Scientific Products FTS		Hearth Living Room (Hosp Suite)

SEE NEXT PAGE FOR ASILOMAR CAMPUS MAP


FIRESIDE PAVILION (underground garage)
ALL POSTER SESSIONS, Sunday Recption, Career Center
EXHIBIT BOOTHS
AIT / Qualytics
Alegre Science-Pure Devices-Stelar-QOne
Annaida Technologies Bluefors
Cryocooler Technologies
Cryogenic Limited
GMW Associates-Metrolab
HTS-110
IMRIS-SSI
Oxford Instruments
Resonance Exploration Technologies
Silantes
Tabor Quantum Solutions
Tomco Technologies
Mestrelab
MR Resources, Q One Instruments, IVAN
Wilmad, ATS Scientific Products
Bruker
ACD / Labs
QUAD Systems
Phoenix NMR
Bridge 12
JEOL
Magritek
NMRbox.org
Tecmag
CIL
Norell
ISOTEC / MilliporeSigma
Asilomar Avenue
Sinex Avenue
Crocker Avenue
MAIN ENTRANCE
Session Room
MERRILL HALL
Badge Pick-up
TRITON
Parallel Sessions
CHAPEL
Parallel Sessions
WOODLANDS
The Circle
Doty Scientific
Sunset Drive
Asilomar Dunes Natural Preserve
Asilomar State Beach
PACIFIC OCEAN
BBQ Area & Fire Pit
Boardwalk
Beach Access
Evergreen
Oak Shelter
Woodside
Forest Lodge
Human Resources
Hearth
Kiln
Embers
Fred Farr Forum
Afterglow
Park Ranger Office
Whitehead
Mott Training Center
Madrone
Guest Inn
Tree Tops
Deer Lodge
Live Oak
Director's Cottage
Meditation Space
Long View South
Long View Middle
Long View North
Yoga Room
Toyon
Acacia
Manzanita
Heather
Oak Knoll
Willow
Underground
Scripps
Lodge
Stuck-up Inn
Engineer's Cottage
Corporation Yard
Viewpoint Group Sales
Curlew
Marlin
Whitecaps South
Whitecaps North
Breakers East
Breakers West
Dolphin
Swimming Pool
Housekeeping
Greenhouse
Spindrift North
Spindrift South
Sanderling
Chapel
Social Hall
Store
Café
Guest Check-In
Merrill Hall
Pirates' Den
Triton
Nautilus
Windward
Cypress
Shores
Volleyball
Fire Pit
Surf
Sand
Surf & Sand Meeting Room
Woodlands
Crocker Dining Hall
Seascape
LEGEND
Information/Guest Check-In
Disabled Parking
Beverage Vending Machine
Ice Machine
Julia Morgan Historic Building
Roadway
Pedestrian Pathway
Outdoor Group Activity Area
ASILOMAR
CONFERENCE GROUNDS
CALIFORNIA STATE PARKS
SINCE 1864

PROGRAM OVERVIEW, For detailed program, use the mobile app or online planner.

There are THREE oral session rooms color-coded on this overview. ***See map on reverse for locations on the campus.***

Merrill Hall | **Chapel** | **Woodlands (next to Crocker)**

Posters, Exhibit Booths, Employment Center, and Sunday Welcome Reception are in Fireside Pavilion (underground garage). ***See map on reverse for location.***

Fireside Pavilion (underground garage)

Flip over for campus map

Flip over for campus map

SUNDAY

2:30 - 4:00 pm, Young Scientist Symposium, **Merrill Hall**

4:00 - 6:00 pm, Welcome Reception with Exhibitors, **Fireside Pavilion (underground garage)**

6:00 - 7:00 pm, Asilomar Dinner Service (ticket req'd)

From 7:00 pm, Hospitality Suites (optional opening day), **various locations see map on reverse**.

MONDAY

7:00-7:50
Hyperpolarization, Part 1: Warren S. Warren
Merrill Hall

8:45 - 10:05
Welcome
Laukien Prize Session
Merrill Hall

10:10-10:45 am Coffee Break

10:45am-12:35pm MOD: Biomolecular Solids I **Merrill Hall**	10:45am-12:35pm MOE:Small Molecules I **Chapel**	10:45am-12:35pm MOF: MOF: The Dynamics of Life! **Woodlands**

12:45-2:00 pm Asilomar Lunch Service (ticket req'd)

1:15-2:00 pm, Int'l Emerging Female Leaders in Biological Magnetic Resonance, **Chapel**

2:00 - 3:45 pm Poster Session & Exhibit Booths
Fireside Pavilion (underground garage)

4:00-5:50 pm MOG: Biomolecular Solutions I **Merrill Hall**	4:00-5:50 pm MOH: Instrumentation I **Chapel**	

6:00 - 7:00 pm Asilomar Dinner Service (ticket req'd)

From 7:00 pm Hospitality Suites
Various locations, **SEE MAP ON REVERSE**

7:00 - 8:30 pm, AMMRL, Chapel
9:00 - 10:30 pm, OCMRS, Chapel

TUESDAY

7:00-7:50
Hyperpolarization, Part 2: Warren S. Warren
Merrill Hall

8:45 - 10:10 TOA: Biomolecular Solids II **Merrill Hall**	8:45 - 10:15 TOB: Materials I **Chapel**	8:45 - 10:15 TOC: Adding Contrast **Woodlands**

10:10-10:45 am Coffee Break @ Merrill & Chapel

10:45am-12:35pm TOD: Biomolecular Solutions II **Merrill Hall**	10:45am-12:35pm TOE: Instrumentation II **Chapel**	10:45am-12:35pm TOF: Spin behavior and Sequence Optimization **Woodlands**

12:45-2:00 pm Asilomar Lunch Service (ticket req'd)

13:00 - 14:00 UHF Workshop, **Merrill Hall**

2:00 - 3:45 pm Poster Session & Exhibit Booths
Fireside Pavilion (underground garage)

4:00-5:50 pm TOG: Hyperpolarization I **Merrill Hall**	4:00-5:50 pm TOH: Metabolomics I **Chapel**	

6:00 - 7:00 pm Asilomar Dinner Service (ticket req'd)

From 7:00 pm Hospitality Suites
Various locations, **SEE MAP ON REVERSE**

WEDNESDAY

7:00-7:50
Hyperpolarization, Part 3: Warren S. Warren
Merrill Hall

8:45 - 10:10 WOA: Theory and Computation **Merrill Hall**	8:45 - 10:15 WOB: Materials II **Chapel**	8:45 - 10:15 WOC: Innovations in MRI **Woodlands**

10:10-10:45 am Coffee Break @ Merrill & Chapel

10:45am-12:35pm WOD: Biomolecular Solids/Materials **Merrill Hall**	10:45am-12:35pm WOE: Eclectica **Chapel**	10:45am-12:35pm WOF: Small Molecules II **Woodlands**

12:45-2:00 pm Asilomar Lunch Service (ticket req'd)

2:00 - 3:45 pm Poster Session & Exhibit Booths
Fireside Pavilion (underground garage)

4:00-5:50 pm WOG: Biomolecular Solutions III **Merrill Hall**	4:00-5:50 pm WOH: Hyperpolarization II **Chapel**	

6:00 - 7:00 pm Asilomar Dinner Service (ticket req'd)

From 7:00 pm Hospitality Suites
Various locations, **SEE MAP ON REVERSE**

THURSDAY

7:00-7:50
Hyperpolarization, Part 4: Warren S. Warren
Merrill Hall

8:45 - 10:10 am ThOA: Biomolecular Solutions IV **Merrill Hall**	8:45 - 10:10 am ThOB: Metabolomics II **Chapel**	8:45 - 10:10 am ThOC:Computation for SS NMR **Woodlands**

10:10-10:45 am Coffee Break @ Merrill & Chapel

10:45am-12:35pm ThOD: Biomolecular Solids III **Merrill Hall**	10:45am-12:35pm ThOE: Hyperpolarization III **Chapel**	10:45am-12:35pm ThOF: Materials III **Woodlands**

12:45-2:00 pm Asilomar Lunch Service (ticket req'd)

2:00 - 3:45 pm Poster Session & Exhibit Booths
Fireside Pavilion (underground garage)

4:00 - 5:35 pm
PLENARY
2 Tutorials, Varian & Awards
Merrill Hall

6:00 - 7:00 pm, Asilomar Dinner Service (ticket req'd) for those **not** attending Social event at The Barns (offsite).

6:30-9:30 pm, Social Event @
The Barns at Cooper Molera
Ticket required, **ticket sales close Monday at noon.**

Explore the ENC Adventure Raffle 2024

Use the ADVENTURE RAFFLE feature in the mobile app to scan a special QR code at each booth or suite location.
High scorers will be entered into the Vendor Raffle held during THURSDAY POSTER SESSION @ 3:30 pm. Must be present to win. Fun prizes (we promise!)

2:30 – 4:00 PM SUNDAY AFTERNOON
YOUNG SCIENTISTS SYMPOSIUM in honor of Suraj Manrao
Fatema Bhinderwala (University of Pittsburgh) and Christopher Williams (UC Riverstide) presiding
Merrill Hall

Sunday 2:30-2:45 pm	**Pyruvate vs glucose as metabolic imaging agents: Is one better than the other?**; Elton Montrazi; *Weizmann Institute of Science*
Sunday 2:45-3:00 pm	**Integrated Low-Field Polarimetry Device for Hyperpolarized Contrast Media Applications**; Clementinah Oladun; *Wayne State University*
Sunday 3:00-3:15 pm	**The Structure and Dynamics of Defects in Metal-organic Frameworks**; David Gomez-Cabeza; *IBEC*
Sunday 3:15-3:30 pm	**Parallel Metabolic Imaging Using MRI and Microfluidics for Personalised Medicine**, Kevin Chalek, *San Diego State University*
Sunday 3:30-3:45 pm	**Solving and Targeting 5_SL5 - the Translational Start Site of SARS-CoV-2**, Klara Mertinkus, *Goethe University-Frankfurt*
Sunday 3:45-4:00 pm	**Investigating the Conformational Dynamics of the Human A2A Adenosine Receptor in Lipid Vesicles by 19F MAS Solid-State NMR**; Beining Jin, *University of Florida*

4:00 - 6:00 PM, SUNDAY AFTERNOON
WELCOME RECEPTION WITH EXHIBIT BOOTHS
Fireside Underground (across Asilomar Blvd.)

6:00 – 7:00 PM, SUNDAY
ASILOMAR DINNER SERVICE
For onsite lodgers or those with tickets, Crocker Dining Hall

From 7:00 PM, SUNDAY
HOSPITALITY SUITES (optional)
See mobile app for list of suites open on Sunday evening.
See map in this PDF for locations around the Asilomar Campus.

7:00 – 7:50 AM, MONDAY
BEYOND BOLTZMANN AND BLOCH: Understanding Hyperpolarization, Hamiltonian manipulation, and Nonlinear NMR
Part 1: Essentials of hyperpolarization (overview of different technologies; fundamental physical limitations of each technology; how far are we, in each case, from the fundamental limits)

Presented by Warren Warren (Duke University)

Merrill Hall

Coffee service starts at 6:45 am.

8:45 – 10:10 AM, MONDAY
WELCOME AND LAUKIEN PRIZE SESSION
Merrill Hall

10:10 – 10:45 AM, MONDAY
COFFEE BREAK
Outside Merrill Hall

10:45 AM - 12:35 PM, MONDAY
MOD: Biomolecular Solids I
Rasmus Linser (TU Dortmund University) presiding
Merrill Hall

MOD 10:45-11:10	**Assemblies of Biomolecules: Good, Bad and Ugly** Presenter: Chad Rienstra (University of Wisconsin-Madison)
MOD 11:10-11:30	**Structural Basis of HIV-1 Maturation Inhibitor Binding and Activity by MAS NMR** Presenter: Roman Zadorozhnyi (University of Delaware)
MOD 11:30-11:50	**NMR Crystallography of Toho-1 β-Lactamase Enabled by Nearly Complete Backbone and Sidechain Assignments** Presenter: Christopher Williams (Department of Chemistry, University of California, Riverside)
MOD 11:50-12:10	**Structure and Dynamics of Phosphatidylinositol Phosphates in Lipid Bilayers** Presenter: Andrew Nieuwkoop (Rutgers University)
MOD 12:10-12:35	**Role of a bacterial glycolipid in Sec-independent membrane protein insertion** Presenter: Kaoru Nomura (Suntory Foundation for Life Sciences)

10:45 AM - 12:35 PM, MONDAY
MOE: Small Molecules I
Jeffrey Peng (University of Notre Dame) presiding
Chapel

MOE 10:45-11:10	**Novel NMR Methods: the Art of Simplifying Complexity** Presenter: Laura Castanar (Complutense University of Madrid)
MOE 11:10-11:30	**Phase-incremented Steady State Free Precession as an Alternate Route to High Resolution 1D NMR** Presenter: Lucio Frydman (Weizmann Institute)
MOE 11:30-11:50	**Single-Scan Ultraselective NMR Experiments with Preserved Sensitivity** Presenter: Margherita Bazzoni (University of Nantes)
MOE 11:50-12:10	**R2D3, a Fast and Accurate approach to 1-Dimensional Quantitative NMR: combining the Old and the New.** Presenter: Margot Sanchez (CEISAM UMR CNRS 6230)
MOE 12:10-12:35	**The Wonders of a 400 MHz HTS Magnet NMR System, How It Works and Our Results at Amgen** Presenter: Maria Victoria Silva Elipe (Amgen, Inc.)

10:45 AM - 12:35 PM, MONDAY
MOF: The Dynamics of Life!
Gosia Marjanska (University of Minnesota) presiding
Woodlands

MOF 10:45-11:10 **Direct imaging of neuronal activity (DIANA) with high temporal and spatial resolution**
Presenter: Jang-Yeon Park (Sungkyunkwan University)

MOF 11:10-11:30 **Steady State T1rho-weighted Breast MRI at Ultra-low Field (6.5 mT)**
Presenter: Sheng Shen (1. A. A. Martinos Center for Biomedical Imaging, Department of Radiology, MGH, 2.Harvard Medical School)

MOF 11:30-11:50 **Correlation and Coordination during Membrane-Crossing of a Peptide-Based Magnetic Resonance Imaging Agent for Molecular Imaging Revealed by Spectroscopy, Imaging and Theoretical Calculation**
Presenter: Shangwu Ding (National Sun Yat-sen University)

MOF 11:50-12:10 **Investigating The Impact Of Diet, Gender And Genetics On Liver Metabolism Using Hyperpolarized MRSI**
Presenter: Irene Marco-Rius (Institute for Bioengineering of Catalonia (IBEC))

MOF 12:10-12:35 **The Role of Clinical Proton MR Spectroscopy of the Central Nervous System**
Presenter: Eva-Maria Ratai (Massachusetts General Hospital/Harvard Medical School)

12:45 – 2:00 PM
ASILOMAR LUNCH SERVICE
For onsite lodgers or those with tickets, Crocker Dining Hall

1:15 – 2:00 PM
International Emerging Female Leaders in Biological Magnetic Resonance
Chapel, All are welcome

2:00 – 3:45 PM
POSTER SESSION AND EXHIBIT BOOTHS
Fireside Pavilion (Underground garage)

4:00 PM - 5:50 PM, MONDAY
MOG: Biomolecular Solutions I
Rieko Ishima (University of Pittsburgh) presiding
Merrill Hall

MOG 04:00-04:25 **Rewiring Allosteric Crosstalk and Specificity in CRISPR-Cas9**
Presenter: George Lisi (Brown University)

MOG 04:25-04:45 **Visualizing a Two-State Conformational Ensemble in Stem-Loop 3 of the Transcriptional Regulator 7SK RNA**
Presenter: Catherine Eichhorn (University of Nebraska Lincoln)

MOG 04:45-05:05 **Mechanistic roles of enzyme tails through the lens of semi-synthesis and NMR**
Presenter: Thibault Viennet (Aarhus University)

MOG 05:05-05:25 **Structure of LARP7 protein p65–telomerase RNA complex in telomerase revealed by cryo-EM and NMR**
Presenter: Yaqiang Wang (University of California Los Angeles)

MOG 05:25-05:50 **Combining Methyl-TROSY and NMR Relaxation Experiments to Probe the Mechanisms of Chaperone Dysfunction in Disease**
Presenter: Rina Rosenzweig (Weizmann Institute of Sceince)

4:00 PM - 5:45 PM, MONDAY
MOH: Instrumentation I
Rachel Martin (University of California, Irvine) presiding
Chapel

MOH 04:00-04:25 **Mechanical Modulation of DC Magnetic Fields: Instrumentation and Biological Applications**
Presenter: Shoujun Xu (University of Houston)

MOH 04:25-04:45 **SSNMR Spinning Module Optimization for Minimimal Perturbance of the Applied Magnetic Field achieving Sub-Hertz Resolution on 13C Adamantane**
Presenter: Jasmin Schoenzart (Colorado School of Mines)

MOH 04:45-05:05 **A High-Volume and High-Frequency Resonator for DNP-NMR**
Presenter: Adam Altenhof (Los Alamos National Laboratory)

MOH 05:05-05:25 **Benchtop NMR Spectroscopy at Record High Fields**
Presenter: John Price (University of Colorado, Boulder)

MOH 05:25-05:45 **Novel Approaches in NanoMRI for Probing Atomic-Scale Material Structure**
Presenter: Raffi Budakian (University of Waterloo)

6:00 – 7:00 PM, MONDAY
ASILOMAR DINNER SERVICE
For onsite lodgers or those with tickets, Crocker Dining Hall

From 7:00 PM, MONDAY
HOSPITALITY SUITES
See map in this PDF for locations around the Asilomar Campus.

7:00 – 8:30 PM, MONDAY
AMMRL
Marc ter Horst presiding
Chapel

9:00 – 10:30 PM, MONDAY
OCMRS
Chapel

7:00 – 7:50 AM, TUESDAY
BEYOND BOLTZMANN AND BLOCH: Understanding Hyperpolarization, Hamiltonian manipulation, and Nonlinear NMR
Part 2: Altering reality for nuclear spins
(very brief review of density matrix formalism; average Hamiltonian theory; recent applications)

Presented by Warren Warren (Duke University)

Merrill Hall

Coffee service starts at 6:45 am.

8:45 AM - 10:10 AM, TUESDAY
TOA: Biomolecular Solids II
Len Mueller (University of California, Riverside) presiding
Merrill Hall

TOA 08:45-09:10	**Solid-State NMR Studies of DNA-Protein Complexes** Presenter: Christopher Jaroniec (The Ohio State University)
TOA 09:10-09:30	**Studies of Interactions Between Biological Membranes and Amyloid-Beta (Aβ) Aggregates by Solid-State NMR Spectroscopy** Presenter: June M. Kenyaga (Binghamton University)
TOA 09:30-09:50	**Modulation of Protein Dynamics by Protein-Protein Interactions Through an Intermolecular Dynamic Network Observed by Solid-State NMR.** Presenter: Sara Medina Gomez (TU Dortmund University)
TOA 09:50-10:10	**The KirBac1.1 inward-rectifier K+ channel is opened cooperatively by anionic lipids as observed by solid-state NMR** Presenter: Benjamin Wylie (Texas Tech University)

8:45 AM - 10:10 AM, TUESDAY
TOB: Materials I
Yusuke Nishiyama (Riken-JEOL, JEOL Resonance) presiding
Chapel

TOB 08:45-09:10	**Probing Oxygen Exchange and Water Stability of Metal-Organic Frameworks using 17O solid-state NMR** Presenter: Frédérique Pourpoint (UCCS - Centrale Lille)
TOB 09:10-09:30	**SMARTER Crystallography of Porous Functional Materials – Strategies to Reveal Supramolecular Assemblies in Host Pore Systems** Presenter: Eric Breynaert (NMRCoRe, KU Leuven)
TOB 09:30-09:50	**In Situ Chemical Shift Imaging Investigation of ZIF-67/Activated Carbon Electrochemical Supercapacitor Cell** Presenter: Mark Bovee (US Naval Research Laboratory)
TOB 09:50-10:10	**Revealing Molecular Mechanisms in Hierarchical Nanoporous Carbon via Nuclear Magnetic Resonance** Presenter: Haiyan Mao (Stanford University)

8:45 AM - 10:10 AM, TUESDAY
TOC: Adding Contrast
Laura Walkup (Cincinnati Children's Hospital) presiding
Woodlands

TOC 08:45-09:10	**Simple hyperpolarization chemistry for more substrates, new NMR physics, and first in-vivo metabolic MRI** Presenter: Thomas Theis (North Carolina State University)
TOC 09:10-09:30	**Whole Abdominopelvic Variable Resolution DNP 13C MRI Imaging for Advanced Prostate Cancer Patients** Presenter: Tanner Nickles (UCSF)
TOC 09:30-09:50	**A Family of Novel DNP Probes to Non-invasively Detect 2-Hydroxyglutarate by in vivo Magnetic Resonance Spectroscopy** Presenter: Norikazu Koyasu (National Institutes of Health / National Cancer Institute)

TOC 09:50-10:10 **High-Sensitivity Glutamate Quantification with CEST, Water-Resonant Spin-Locking, and MR Fingerprinting**
Presenter: David Korenchan (Athinoula A. Martinos Center for Biomedical Imaging)

10:10 – 10:45 AM, TUESDAY
COFFEE BREAKS
Outside Merrill Hall and Ouside Chapel
Attendees in the Woodlands session room should go to Merrill or Chapel for coffee break.

10:45 AM - 12:30 PM, TUESDAY
TOD: Biomolecular Solutions II
Lucia Banci (University of Florence-CERM) presiding
Merrill Hall

TOD 10:45-11:10 **NMR spectroscopy of Biomolecular Condensates**
Presenter: Markus Zweckstetter (German Center for Neurodegenerative Diseases)

TOD 11:10-11:30 **Biomolecular Condensate Remodels the Conformational Equilibria of SOD1 Towards Aggregation-Prone States**
Presenter: Rashik Ahmed (Hospital for Sick Children)

TOD 11:30-11:50 **Using CS-ROSETTA to Probe Assembly of Intrinsically Disordered Spider Silk Proteins in Solution**
Presenter: David Onofrei (San Diego State University)

TOD 11:50-12:10 **In-situ electric field in NMR to study charge, ligand binding and orientation of molecules**
Presenter: Ulrich Scheler (Leibniz-Institut für Polymerforschung Dresden e.V.)

TOD 12:10-12:30 **Structure Determination of RNA Tetraloop Ensembles with Integrated NMR/Molecular Dynamics Based Methods**
Presenter: David Leopold (Johann Wolfgang Goethe-University Frankfurt)

10:45 AM - 12:35 PM, TUESDAY
TOE: Instrumentation II
Thorsten Maly (Bridge 12 Technologies, Inc.) presiding
Chapel Hall

TOE 10:45-11:10 **A Semiconductor-chip Based Miniature Magnetic Field Sensor (MMFS) aimed at MRI Image Calibration**
Presenter: Guang Yang (Harvard University)

TOE 11:10-11:30 **From Low-Cost, Homebuilt Laboratory Prototypes to Industrial Magnetic Resonance Systems for Online Monitoring at Mine-Sites**
Presenter: Thai Ly (CSIRO)

TOE 11:30-11:50 **47 Tesla Handheld Magnet and a Path Towards Widely Available NMR >2 GHz**
Presenter: Alexander Barnes (ETH Zurich)

TOE 11:50-12:10 **Extreme Line Narrowing with Matched Decoupling**
Presenter: Matthew Augustine (UC Davis)

TOE 12:10-12:35 **Frugal NMR Spectroscopy**
Presenter: Aldrik Velders (Wageningen University)

10:45 AM - 12:35 PM, TUESDAY
TOF: Spin Behavior and Sequence Optimization
Sharon Ashbrook (University at St. Andrews) presiding
Woodlands

TOF 10:45-11:10 **Is it time to revise the NMR Signal Processing?**
Presenter: Vladislav Orekhov (University of Gothenburg)

TOF 11:10-11:30 **Pulsed Dynamic Nuclear Polarization Sequence Design via Effective Hamiltonian Optimization**
Presenter: José P. Carvalho (Interdisciplinary Nanoscience Center (iNANO) and Department of Chemistry, Aarhus University)

TOF 11:30-11:50 **Optimal control pulses for improving filtered NOESY experiments**
Presenter: David Joseph (Max Planck Institute for Multidisciplinary Sciences, Göttingen, Germany)

TOF 11:50-12:10 **Characterizing Internal Dynamics Using Partially Averaged Anisotropic Interactions**
Presenter: Kathrin Aebischer (ETH Zurich)

TOF 12:10-12:35 **Solid-state NMR Tools in Crystal Engineering**
Presenter: Michele R. Chierotti (University of Torino)

12:45 – 2:00 PM, TUESDAY
ASILOMAR LUNCH SERVICE
For onsite lodgers or those with tickets, Crocker Dining Hall

1:00 – 2:00 PM, TUESDAY
ULTRA HIGH-FIELD WORSHOP
Merrill Hall, All are welcome
Bring your own lunch. If you have Asilomar lunch ticket, go to Crocker and request your lunch 'to-go', then bring with you to

Program
1:00pm, Tatyana Polenova, Introduction
1:00 - 1:10 pm, Chris Jaroniec: "Update on the 1.2 GHz NMR Instrument & National Gateway Ultrahigh Field NMR Center at OSU"
1:10 - 1:20 pm, Lucia Banci: "Exploring the 1.2 GHz NMR potentialities"
1:20 - 1:30 pm, Rainer Kuemmerle: "Update from Bruker on UHF NMR"
1:30 - 1:40 pm, Chad Rienstra: "Solid-State NMR on the 1.1 GHz Spectrometer at U. Wisconsin-Madison"
1:40 - 1:50 pm, Rob Schurko: "The Need for High-Field NMR"
1:50 - 2:00 pm, Lucio Frydman "NASEM Committee: Update"

2:00 – 3:45 PM, TUESDAY
POSTER SESSION AND EXHIBIT BOOTHS
Fireside Pavilion (Underground garage)

4:00 PM - 5:45 PM, TUESDAY
TOG: Hyperpolarization I
Laura Walkup (Cincinnati Children's Hospital) presiding
Merrill Hall

TOG 04:00-04:25 **Recent advances in magnetic resonance imaging for chemical and bioprocess engineering**
Presenter: Alexander Penn (Hamburg University of Technology)

TOG 04:25-04:45 **Using Computationally Optimized Three-Dimensional Field Sequences to Improve SABRE Hyperpolarization**
Presenter: Shannon Eriksson (Duke University)

TOG 04:45-05:05 **First Hyperpolarized [1-13C]alpha-ketoglutarate MR Spectroscopy of the Human Brain**
Presenter: Yaewon Kim (University of California, San Francisco)

TOG 05:05-05:25 **Solution-State NMR signal enhancement of small molecules via intermolecular cross relaxation from PHIP hyperpolarized source molecules**
Presenter: Ilai Schwartz (NVision Imaging Technologies GmbH)

TOG 05:25-05:45 **Live Magnetic Observation of Parahydrogen Hyperpolarization Dynamics**
Presenter: Michael Tayler (ICFO - The Institute of Photonic Sciences)

4:00 PM - 5:50 PM, TUESDAY
TOH: Metabolomics I
David Rovnyak (Bucknell University) presiding
Chapel

TOH 04:00-04:25	**New Approaches for Studying Known and Unknown Complex Metabolomics Mixtures by NMR** Presenter: Rafael Bruschweiler (The Ohio State University)
TOH 04:25-04:45	**NMR Metabolomics Best Practices: What We Are Doing, What We Should Be Doing, and Some Recent Advancements** Presenter: Robert Powers (University of Nebraska-Lincoln)
TOH 04:45-05:05	**Unraveling Human CD8+ T-Cell Metabolism: A 13C NMR Perspective on Targeting Glycolysis for Autoimmune Disease Intervention** Presenter: Gaurav Sharma (UNIVERSITY OF FLORIDA)
TOH 05:05-05:25	**Impact of Exposure to Environmental Pollutants on Placental Metabolism** Presenter: Lindsay Cahill (Memorial University of Newfoundland)
TOH 05:25-05:50	**Ultra-High Resolution NMR: a Robust Method for Determining Quantitative Metabolic Profiles in Biofluids** Presenter: Nicolas Giraud (Université Paris Cité)

6:00 – 7:00 PM, TUESDAY
ASILOMAR DINNER SERVICE
For onsite lodgers or those with tickets, Crocker Dining Hall

From 7:00 PM, TUESDAY
HOSPITALITY SUITES
See map in this PDF for locations around the Asilomar Campus.

7:00 – 7:50 AM, WEDNESDAY

BEYOND BOLTZMANN AND BLOCH: Understanding Hyperpolarization, Hamiltonian manipulation, and Nonlinear NMR

Part 3: Effects of large magnetization (coherence, correlation and entanglement; radiation damping; dipolar field; differences between concentrated thermal and dilute hyperpolarized magnetization)

Presented by Warren Warren (Duke University)

Merrill Hall

Coffee service starts at 6:45 am.

8:45 AM - 10:15 AM, WEDNESDAY

WOA: Theory and Computation for Structural and Dynamic Aspects

Len Mueller (University of California, Riverside) presiding

Merrill Hall

WOA 08:45-09:10	**Transformative Acceleration of Putative Structure Generation** Presenter: Chris Pickard (University of Cambridge)
WOA 09:10-09:30	**Neural Net Analysis of Strongly Coupled Spin Systems** Presenter: James Prestegard (University of Georgia)
WOA 09:30-09:50	**First Principles Calculations of Molecular Properties in Electrolytes from Spin and Molecular Dynamics Simulations Coupled with Experimental Relaxation Rates** Presenter: Florin Teleanu (New York University)
WOA 09:50-10:15	**Development of QM/DFT Computational Tools to Accelerate NMR based Molecular Structure Elucidation and Solid Form Identification** Presenter: Nina Gonnella (Boehringer Ingelheim Inc.)

8:45 AM - 10:15 AM, WEDNESDAY

WOB: Materials II

Robert W. Schurko (Florida State University) presiding

Chapel

WOB 08:45-09:10	**Structure and Dynamics of Photochromic Rare-Earth Oxyhydrides** Presenter: Arno Kentgens (MRRC, Radboud University)
WOB 09:10-09:30	**Exploiting In Situ Solid-State NMR Spectroscopy to Follow Non-Traditional Zeolite Formation** Presenter: Emma A. L. Borthwick (University of St Andrews)
WOB 09:30-09:50	**Massive CQ's and Fast Cation Dynamics: 23Na, 25Mg and 11B NMR Studies of "Paddlewheel" Antiperovskite Solid Electrolytes** Presenter: David Halat (UC Berkeley & LBNL)
WOB 09:50-10:15	**Probing Structural Subtleties in Anti-Perovskite Solid Electrolytes** Presenter: Karen Johnston (Durham University)

8:45 AM - 10:15 AM, WEDNESDAY

WOC: Innovations in MRI Methods and Instruments

Matthew Rosen (Mass General / Martinos Ctr for Biomedical Imaging, Harvard Medical School) presiding

Woodlands

WOC 08:45-09:10	**20-year odyssey of the 11.7T human MRI project** Presenter: Nicolas Boulant (University of Paris-Saclay, CEA, NeuroSpin)
WOC 09:10-09:30	**Dynamic Noise Cancellation for Unshielded, Single-Sided, Fourier and Spatiotemporally Encoded Low-Field MRI** Presenter: Kartiga Selvaganesan (Promaxo Inc.)
WOC 09:30-09:50	**Multiband Spatiotemporal Encoding with minimized slab boundary artifacts** Presenter: Jaeyong Yu (Sungkyunkwan University)
WOC 09:50-10:15	**Dipolar Order and Disorder in Model Systems and In Vivo** Presenter: Scott Swanson (University of Michigan)

10:15 – 10:45 AM, WEDNESDAY
COFFEE BREAKS
Outside Merrill Hall and Ouside Chapel
Attendees in the Woodlands session room should go to Merrill or Chapel for coffee break.

10:45 AM - 12:35 PM, WEDNESDAY
WOD: Biomolecular Solids-Materials
Sharon Ashbrook (University at Andrews) presiding
Merrill Hall

WOD 10:45-11:10	**A close look at the surface of nanocelluloses with DNP-enhanced solid-state NMR** Presenter: Sabine Hediger (Univ. Grenoble Alpes / CEA / CNRS)
WOD 11:10-11:30	**Hydration water dynamics in globular villin headpiece and amyloid fibrils, a joint H-2 and O-17 NMR study** Presenter: Liliya Vugmeyster (CU Denver)
WOD 11:30-11:50	**Proton Relaxation Dispersion at fast MAS, a Tool with Important Prospects** Presenter: Rasmus Linser (TU Dortmund University)
WOD 11:50-12:10	**Novel Dipolar Recoupling Elements** Presenter: Evgeny Nimerovsky (MPI-NAT)
WOD 12:10-12:35	**Optimal Control Methods for Multidimensional Solid-State NMR of Proteins** Presenter: Zdenek Tosner (Charles university)

10:45 AM - 12:35 PM, WEDNESDAY
WOE: Eclectica
Carl Michal (University of British Columbia) presiding
Chapel

WOE 10:45-11:10	**Ultra-sensitivity with radiation-detected NMR using unstable nuclei** Presenter: Magdalena Kowalska (CERN/UNIGE)
WOE 11:10-11:30	**Towards Ultra-high-quality-factor Wearable RASER MR Sensing Using Parametric Pumping** Presenter: Eduard Chekmenev (Wayne State University)
WOE 11:30-11:50	**Moving MRI (mMRI): Imaging a Moving Body with Synchronized Magnet Movement** Presenter: Jingting Yao (Massachusetts General Hospital/Harvard Medical School)
WOE 11:50-12:10	**Deconstructing Porous Media: Magnetic Resonance Insights into the Heterogeneous, Fractal-like Kinetics of Chemically Upcycled Polymers** Presenter: Sophia Fricke (University of California, Berkeley)
WOE 12:10-12:35	**Probing singlet states in frustrated magnets using muons as a magnetic resonance probe** Presenter: Stephen Blundell (University of Oxford)

10:45 AM - 12:30 PM, WEDNESDAY
WOF: Small Molecules II
Yusuke Nishiyama (Riken-JEOL, JEOL Resonance) presiding
Woodlands

WOF 10:45-11:10	**Protein dynamics from relaxation and paramagnetic NMR** Presenter: Christian Griesinger (MPINAT)
WOF 11:10-11:30	**Free Ligand Solution Conformations Guided Design of Potent and Bioavailable Drug Candidates** Presenter: Amber Balazs (AstraZeneca)
WOF 11:30-11:50	**The Field and Temperature Dependence of 1H and 13C Relaxation Rates in Rapidly-Rotating Methyl Groups and the Effect of Spin Rotation** Presenter: Kelsey Marr (New York University)
WOF 11:50-12:10	**Slice Through the Water – Exploring the Fundamental Challenge of Water Suppression for Benchtop NMR Systems** Presenter: Ronald Soong (University of Toronto Scarborough)

WOF 12:10-12:30 **Parahydrogen Polarization of Biological Molecules in Homogeneous and Heterogeneous Media**
Presenter: Christian Hilty (Texas A&M University)

12:45 – 2:00 PM, WEDNESDAY
ASILOMAR LUNCH SERVICE
For onsite lodgers or those with tickets, Crocker Dining Hall

2:00 – 3:45 PM, WEDNESDAY
POSTER SESSION AND EXHIBIT BOOTHS
Fireside Pavilion (Underground garage)

4:00 PM - 5:50 PM, WEDNESDAY
WOG: Biomolecular Solutions III
Rasmus Linser (TU Dortmund University) presiding
Merrill Hall

WOG 04:00-04:25 **Understanding Periplasmic Protein Quality Control at Atomic Level in Live Cells**
Presenter: Alejandro Vila (IBR (CONICET, UNR))

WOG 04:25-04:45 **In-cell NMR: a Powerful Approach for Drug Discovery**
Presenter: Lucia Banci (University of Florence)

WOG 04:45-05:05 **Cell-free synthesis of trifluoromethionine labeled proteins for 19F NMR studies**
Presenter: Wenkai Zhu (University of Pittsburgh)

WOG 05:05-05:25 **NMR Observation of Membrane-Associated H-Ras in the Native Cellular Environment**
Presenter: Takanori Kigawa (RIKEN Center for Biosystems Dynamics Research)

WOG 05:25-05:50 **19F NMR spectroscopy: a powerful approach for studying biological systems in vitro and in cells**
Presenter: Conggang Li (Chinese Academy of Sciences)

4:00 PM - 5:45 PM, WEDNESDAY
WOH: Hyperpolarization II
Daniel Raftery (University of Washington) presiding
Chapel

WOH 04:00-04:25 **High-Field Dynamic Nuclear Polarization from the Electron Spin Perspective**
Presenter: Ilia Kaminker (Tel-Aviv University)

WOH 04:25-04:45 **First Example of Nitrogen-14 Hyperpolarization**
Presenter: Roman Shchepin (South Dakota School of Mines & Technology)

WOH 04:45-05:05 **Frequency-Chirped MAS DNP and Combination with Electron Decoupling**
Presenter: Nicholas Alaniva (ETH-Zürich)

WOH 05:05-05:25 **Feasibility of HyperCEST Spectroscopy With Caged Xenon Under Enhanced Relaxation Conditions**
Presenter: Hannah Gerbeth (German Cancer Research Centre (DKFZ))

WOH 05:25-05:45 **Optically Enhanced Solid-State 1H NMR Spectroscopy**
Presenter: Lyndon Emsley (EPFL)

6:00 – 7:00 PM, WEDNESDAY
ASILOMAR DINNER SERVICE
For onsite lodgers or those with tickets, Crocker Dining Hall

From 7:00 PM, WEDNESDAY
HOSPITALITY SUITES
See map in this PDF for locations around the Asilomar Campus.

7:00 – 7:50 AM, THURSDAY
BEYOND BOLTZMANN AND BLOCH: Understanding Hyperpolarization, Hamiltonian manipulation, and Nonlinear NMR
Part 4: Enhancing hyperpolarization (long lived states, pulsed DNP, complex fields in PHIP/SABRE)

Presented by Warren Warren (Duke University)

Merrill Hall

Coffee service starts at 6:45 am.

8:45 AM - 10:10 AM, THURSDAY
ThOA: Biomolecular Solutions IV
Jeffrey Peng (University of Notre Dame) presiding
Merrill Hall

ThOA 08:45-09:10	**A Complete Set of Cross-Correlated Relaxation Experiments for Structural Studies of Intrinsically Disordered Proteins** Presenter: Anna Zawadzka-Kazimierczuk (University of Warsaw)
ThOA 09:10-09:30	**Disentangling NOE from 1H relaxation measurements reveal a third state in Watson-Crick-Franklin to Hoogsteen DNA base pair dynamics.** Presenter: Rubin Dasgupta (Uppsala University)
ThOA 09:30-09:50	**New applications of TOCSY NMR for pursuing the structure and dynamics of protein sidechains** Presenter: Peter Hwang (Departments of Medicine, Biochemistry, University of Alberta)
ThOA 09:50-10:10	**Enhanced Sensitivity and Resolution CEST NMR by the Extended Hadamard Scheme** Presenter: Jihyun Kim (Weizmann Institute of Science)

8:45 AM - 10:10 AM, THURSDAY
ThOB: Metabolomics II
Daniel Raftery (University of Washington) presiding
Chapel

ThOB 08:45-09:10	**Development and Applications of a Novel 13C High-Temperature Superconducting Probe at 21.1 T** Presenter: Art Edison (University of Georgia)
ThOB 09:10-09:30	**Developing HRMAS 13C NMR Reactor for Monitoring Real-Time Live Cell Metabolomic Reactions** Presenter: Rajshree Ghosh Biswas (MGH Harvard Medical School)
ThOB 09:30-09:50	**Advancing towards monitoring bacteria metabolism through SABRE** Presenter: Julia Schulte-Hermann (Karlruhe Institute of Technology)
ThOB 09:50-10:10	**From Conventional to High-Power-Laser-Driven Irradiation: Timely Detection of Magnetic Resonance Biomarkers in Cells Cultures** Presenter: Ioana Fidel (Extreme Light Infrastructure Nuclear-Physics)

8:45 AM - 10:15 AM, THURSDAY
ThOC: Computation for SS NMR
Danielle Laurencin (CNRS Montpellier) presiding
Woodlands

ThOC 08:45-09:10	**Modulation of Structure and Dynamics in Solids via Directional Non-Covalent Interactions. The Roles of NMR Experiments and Theory** Presenter: David Bryce (University of Ottawa)
ThOC 09:10-09:30	**Biexponential I = 3/2 Spin-Lattice Relaxation in the Solid State: Multiple-Quantum 7Li NMR as a Probe of Fast Ion Dynamics** Presenter: Stephen Wimperis (Department of Chemistry, Lancaster University)
ThOC 09:30-09:50	**An Exploration of Dipolar Order in Wideline Solid State NMR** Presenter: James Kimball (Florida State University)
ThOC 09:50-10:15	**Cryogenic Solid-State NMR and MAS DNP Investigation of Metastable Species Formed along Crystallization Processes** Presenter: Giulia MOLLICA (CNRS, ICR)

10:10 – 10:45 AM, THURSDAY
COFFEE BREAKS
Outside Merrill Hall and Ouside Chapel
Attendees in the Woodlands session room should go to Merrill or Chapel for coffee break.

10:45 AM - 12:35 PM, THURSDAY
ThOD: Biomolecular Solids III
Rachel Martin (University of California, Irvine) presiding
Merrill Hall

ThOD 10:45-11:10	**DNP-enhanced solid-state NMR in mechanistic Membrane Protein Research** Presenter: Clemens Glaubitz (Goethe University, BMRZ)
ThOD 11:10-11:30	**Solid-State and Solution NMR Comparison of GPCR Energy Landscapes in Membrane Mimetics** Presenter: Matthew Eddy (University of Florida)
ThOD 11:30-11:50	**Mapping the energy landscape, stages, and domains of membrane protein unfolding using solid-state NMR spectroscopy** Presenter: Vlad Ladizhansky (University of Guelph)
ThOD 11:50-12:10	**A Targeted DNP Approach to Study the Activity of the Membrane Protein Ail in Whole Bacterial Cells** Presenter: Nesreen Elathram (UC San Diego)
ThOD 12:10-12:35	**Adventures in Biomolecular MAS DNP** Presenter: Joanna Long (University of Florida)

10:45 AM - 12:35 PM, THURSDAY
ThOE: Hyperpolarization III
Thorsten Maly (Bridge 12 Technologies, Inc.) presiding
Chapel

ThOE 10:45-11:10	**Synchronizing a High-Rep Rate Tunable Pulsed Laser with MAS NMR Experiments for the Development of Optically Pumped NMR Methods** Presenter: Claudia Avalos (New York University)
ThOE 11:10-11:30	**Scaling Up Hyperpolarization of 117Sn and 1H for Neutron Optics-Based Time-Reversal Symmetry Investigations.** Presenter: Abubakar Abdurraheem (Wayne State University)
ThOE 11:30-11:50	**Magnetic-Field Dependence of LC-Photo-CIDNP Nuclear-Spin Hyperpolarization via a Rapid-Shuttling Device** Presenter: Silvia Cavagnero (University of Wisconsin-Madison)
ThOE 11:50-12:10	**Optimizing Signal Amplification by Reversible Exchange: Recyclable Perfluorinated Iridium Catalysts for Enhanced [1-13C]Pyruvate Hyperpolarization in Fluorinated Solvents and its Metal-Free Aqueous Delivery** Presenter: Jess Benjamini-Ettedgui (NHLBI/NIH)
ThOE 12:10-12:35	**Continuous-wave NMR for Nuclear Physics Polarized Scattering Experiments** Presenter: James Maxwell (Jefferson Lab)

10:45 AM - 12:35 PM, THURSDAY
ThOF: Materials III
Gaël De Paëpe (CEA / Univ. Grenoble Alpes) presiding
Woodlands

ThOF 10:45-11:10	**Advanced Solid-State NMR Spectroscopy of Materials using Fast Magic Angle Spinning and Dynamic Nuclear Polarization** Presenter: Amrit Venkatesh (National High Magnetic Field Laboratory, Florida State University)
ThOF 11:10-11:30	**Local structure and diffusion of sodium in a hybrid glass from high-temperature 23Na MAS NMR at 20 T** Presenter: Dominik Kubicki (University of Birmingham)

ThOF 11:30-11:50 **Surface Sensitive Solid-State NMR Spectroscopy Reveals Local Structural Features of an Industrial Catalyst**
Presenter: Sonja Egert (University of St Andrews)

ThOF 11:50-12:10 **Unifying finally all NMR/DNP data related to substituted hydroxyapatite by extensive NMR/DNP crystallography**
Presenter: Christian Bonhomme (Sorbonne University)

ThOF 12:10-12:35 **Magnetic Resonance for Li-ion Batteries: From NMR to EPR Imaging**
Presenter: Bingwen Hu (East China Normal University)

12:45 – 2:00 PM, THURSDAY
ASILOMAR LUNCH SERVICE
For onsite lodgers or those with tickets, Crocker Dining Hall

2:00 – 3:45 PM, THURSDAY
POSTER SESSION AND EXHIBIT BOOTHS
Fireside Pavilion (Underground garage)

4:00 – 5:35 PM, THURSDAY
MACHINE LEARNING TUTORIAL SESSION
Followed by Awards Presentations for Students & Postdocs
And
Closing Remarks: Intro to the 66th ENC Joint ISMAR in 2025

Merrill Hall

ThOG 4:00-4:45 **Machine Learning for Magnetic Resonance: From Bayesian Methods to Modern Neural Networks for Large-Scale Analysis of MRI Data**
Presenter: Juan Eugenio Iglesias, *Massachusetts General Hospital & Harvard Medical School*

ThOG 4:45-5:20 **Transforming and Analyzing Complex NMR Spectra with Deep Neural Networks**
Presenter: D. Flemming Hansen, *University College London & Francis Crick Institute*

5:20-5:35 **Presentation of Student and Postdoc Awards**

Closing Remarks: Intro to the 66th ENC Joint ISMAR in 2025

6:00 – 7:00 PM, THURSDAY
ASILOMAR DINNER SERVICE
For onsite lodgers **not** attending the social event offsite, Crocker Dining Hall

6:30 – 9:30 PM, THURSDAY
SOCIAL EVENT OFFSITE
at
THE BARNS AT COOPER MOLERA

Advance purchase ticket is required. **Ticket sales close at 2:00 pm on Monday.**

Location is 535 Polk Street in Downtown Monterey.

Doors will open at 6:30 pm. You are welcome to arrive by uber/lyft, your own car, etc. OR take our shuttle buses.

Shuttle buses. Look for buses behind the Social Hall. First bus will load at 6:15 pm. Buses will circulate in a loop until all guests are transported to the Barns. Shuttles will be running to return guests to Asilomar.

10:45 AM - 12:35 PM, MONDAY
MOD: Biomolecular Solids I
Rasmus Linser (TU Dortmund University) presiding
Merrill Hall

MOD 10:45-11:10
Assemblies of Biomolecules: Good, Bad and Ugly
Presenter: Chad Rienstra (University of Wisconsin-Madison)
All Authors: Chad Rienstra (University of Wisconsin-Madison)
In this talk, I will present recent results from our team focusing on understanding the biological role of macromolecular assemblies in health and disease. Assemblies for good include amphotericin B, an antifungal drug that acts by sequestering ergosterol in a high molecular weight extramembranonous sponge, which we have engineered for improved therapeutic index. Assemblies for bad include alpha-synuclein fibrils, the accumulation of which can lead to a variety of neurodegenerative pathology. We have studied in vitro as well as tissue amplified forms to gain insights into the structures and ligand binding sites in various fibrils. Although these preparations are sometimes macroscopically ugly, with state-of-the-art solid-state NMR instrumentation and pulse sequences, beautiful spectra are most often the result.

MOD 11:10-11:30
Structural Basis of HIV-1 Maturation Inhibitor Binding and Activity by MAS NMR
Presenter: Roman Zadorozhnyi (University of Delaware)
All Authors: Roman Zadorozhnyi (University of Delaware); Caitlin M. Quinn (University of Delaware); Sucharita Sarkar (Graduate student); Ryan W. Russell (University of Delaware); Kaneil K. Zadrozny (University of Virginia School of Medicine); Juan Sebastian Rey Lancheros (University of Delaware); Hamed Meshkin (University of Delaware); Brandon J. Kennedy (Lotus Separations LLC, Princeton University); Sherimay Ablan (HIV Dynamics and Replication Program, Center for Cancer Research, National Cancer Institute); Glenn P. A. Yap (University of Delaware); Daniel Sanner (Lotus Separations LLC, Princeton University); Christina Kraml (Lotus Separations LLC, Princeton University); Juan R. Perilla (University of Delaware); Eric O. Freed (HIV Dynamics and Replication Program, Center for Cancer Research, National Cancer Institute); Barbie K. Ganser-Pornillos (University of Utah School of Medicine); Owen Pornillos (University of Utah); Angela Gronenborn (University of Pittsburgh School of Medicine); Tatyana Polenova (University of Delaware)
HIV-1 maturation inhibitors (MI) are a novel class of anti-retroviral compounds that bind to and stabilize a junction between capsid protein C-terminal domain (CACTD) and spacer peptide 1 (SP1) preventing its proteolytic cleavage. Here we report atomic-resolution structures of microcrystalline CACTD-SP1 assembly with MI PF-46396 and inositol hexakisphosphate (IP6) using magic angle spinning (MAS) NMR. Our results reveal unique binding modes of PF-46396 and simultaneous binding of the inhibitor and IP6 with reduced mobility of the cofactor inside the binding pore. The unique interactions between PF-46396 enantiomers with IP6 and CACTD-SP1 in the six-helix bundle pore allowed for identification of each enantiomer. Our study allows for structural explanation of PF-46396 resistance mechanisms and provides guidance for a new MIs design.

MOD 11:30-11:50
NMR Crystallography of Toho-1 β-Lactamase Enabled by Nearly Complete Backbone and Sidechain Assignments
Presenter: Christopher Williams (Department of Chemistry, University of California, Riverside)
All Authors: Christopher Williams (Department of Chemistry, University of California, Riverside); Songlin Wang (University of Wisconsin-Madison); Jacob Holmes (Department of Chemistry, University of California, Riverside); Rittik Ghosh (Department of Biochemistry, University of California - Riverside, CA 92521); Chad Rienstra (University of Wisconsin-Madison); Len Mueller (University of California Riverside)
Nearly complete backbone and sidechain chemical shifts are reported for a microcrystalline sample of the 28 kDa Toho-1 β-Lactamase in its free and avibactam-bound forms. Using only two 2D and three 3D experiments at 900 MHz, 98% of the backbone and 94% of the nonaromatic sidechains can be assigned, including the mechanistically important active site sidechain residues. These chemical shifts allow 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. The resulting structures are consistent with a proposed mechanism in which Glu166 is the general base catalyst.

MOD 11:50-12:10
Structure and Dynamics of Phosphatidylinositol Phosphates in Lipid Bilayers
Presenter: Andrew Nieuwkoop (Rutgers University)
All Authors: Ashley Bernstein (Rutgers University); Yanxing Yang (Rutgers University); Tom Osborn Popp (Rutgers University); Gertrude Asante Ampadu (Rutgers University); Andrew Nieuwkoop (Rutgers University)
Phosphatidylinositol phosphates (PIPs) are eukaryotic membrane lipids that tightly regulate cellular processes such as cellular growth, metabolism, immunity, and development, through direct interactions with partner proteins. In this work, we seek to directly observe the structure and dynamics of PIP3 in lipid bilayers. We probe the effects of the anionic lipid phosphatidylserine (PS) and the divalent cation Ca2+. We use solution and solid-state 1H, 31P, and 13C NMR combined with MD simulations to characterize the structure and dynamics of PIPs. Overall, we note the presence of PS restricts the lateral diffusion of PIPs. PIP3s, like PS, form dimers (and larger oligomers) in the presence of Ca2+ which explains differential changes in the rotational dynamics of the PIP headgroup phosphates.

MOD 12:10-12:35
Role of a bacterial glycolipid in Sec-independent membrane protein insertion
Presenter: Kaoru Nomura (Suntory Foundation for Life Sciences)
All Authors: Kaoru Nomura (Suntory Foundation for Life Sciences)
An endogenous glycolipid in the E. coli membrane called membrane protein integrase (MPIase) regulates membrane protein insertion. Here, we focused on the Sec translocon-independent pathway and examined the mechanisms of MPIase-facilitated protein insertion. We determined the membrane insertion efficiency of a protein using solid-state NMR. The insertion strongly correlated with membrane physicochemical properties. Diacylglycerol reduced the acyl chains mobility and inhibited the insertion, whereas MPIase increased the mobility and restored the insertion. The negatively charged pyrophosphate of MPIase attracted the positively charged residues of a protein near the membrane surface. This becomes the trigger of the insertion during the early stage. Thus, MPIase supports the membrane insertion by its unique molecular structure in various ways.

10:45 AM - 12:35 PM, MONDAY
MOE: Small Molecules I
Jeffrey Peng (University of Notre Dame) presiding
Chapel

MOE 10:45-11:10
Novel NMR Methods: the Art of Simplifying Complexity
Presenter: Laura Castanar (Complutense University of Madrid)
All Authors: Laura Castañar (Complutense University of Madrid)
Here several novel NMR approaches to solving some of the most challenging problems encountered in the analysis of complex systems will be shown. The methods proposed use the following approaches to facilitate the extraction of key spectroscopic information:

i) factorization of complex proton NMR spectra into simplified subspectra for individual spin systems by homonuclear (GEMSTONE) or heteronuclear (FESTA) spectral editing, and

ii) spectral simplification by using pure shift NMR spectroscopy, where the effect of scalar couplings is suppressed, providing ultrahigh resolution spectra.

The usefulness of these new methods will be illustrated across a wide range of applications, including the structural analysis of natural products and pharmaceuticals, and the study of physically inseparable mixture.

MOE 11:10-11:30

Phase-incremented Steady State Free Precession as an Alternate Route to High Resolution 1D NMR

Presenter: Lucio Frydman (Weizmann Institute)

All Authors: Tian He (Weizmann Institute); Yuval Zur (Insightech Ltd); Elton T. Montrazi (Weizmann Institute); Mark Shiff (Weizmann Institute); Lucio Frydman (Weizmann Institute)

For over five decades, pulse-acquire experiments have been the choice for collecting high resolution 1D NMR spectra, providing excellent resolution with good sensitivity over arbitrary bandwidths. This study presents an alternative providing the same information, with comparable (or slightly higher?) sensitivity. The starting point of this phase-incremented steady-state (PISS) NMR experiment is Carr's 1958 steady-state free precession (SSFP), known to provide the highest possible sensitivity per √acquisition_time for liquids fulfilling T2≈T1. SSFP however, lacks spectral resolution and shows strong distortions in the peak's amplitudes depending on their position peaks. PISS relies on this sensitivity to offset to deliver high resolution NMR spectra, while avoiding bandwidth penalties and preserving other desirable aspects of SSFP's performance.

MOE 11:30-11:50

Single-Scan Ultraselective NMR Experiments with Preserved Sensitivity

Presenter: Margherita Bazzoni (University of Nantes)

All Authors: Margherita Bazzoni (University of Nantes); Rituraj Mishra (University of Nantes); Jean-Nicolas Dumez (University of Nantes)

Ultraselective 1D NMR experiments allow the selection of a 1H multiplet of interest even when it overlaps with other multiplets. The challenge in the implementation of ultraselective experiments is to achieve high selectivity within short experimental times and with good sensitivity.

The GEMSTONE (Gradient-Enhanced Multiplet-Selective Targeted-Observation) pulse sequence element allows the acquisition of ultraselective experiments in a single scan, through a position dependent dephasing of off-resonance signals. However, high selectivity with GEMSTONE requires long gradient pulses that result in important diffusion losses.

Here we describe the GEMSTONES approach, which largely mitigates diffusion-induced sensitivity losses in single-scan ultraselective experiments and significantly broaden their applicability.

MOE 11:50-12:10

R2D3, a Fast and Accurate approach to 1-Dimensional Quantitative NMR: combining the Old and the New.

Presenter: Margot Sanchez (CEISAM UMR CNRS 6230)

All Authors: Sanchez Margot (CEISAM UMR CNRS 6230); Julien Pontabry (RS2D); Assemat Gaëtan (RS2D); Martinez Anthony (R2SD); Akoka Serge (CEISAM)

In order to decrease the experimental time in quantitative experiments, while preserving its precision and/or trueness, we combined the DEFT pulse sequence with the R2D2 method (Recovery time Reduction to Decrease experimental time Duration) that we recently developed (R2D3). With this approach, a 2-dimensional spectrum is recorded, with TR decreasing in the indirect dimension. After FT in both dimensions and projection, a 1D spectrum is obtained.

This way, a huge gain in time can be obtained, up to 18, depending on the targeted precision and trueness. Acquisition time can even be divided by 40 if only a trueness of 3% is expected. This time saving is better than that obtained with 1D polarization transfer technique but without its main drawback.

MOE 12:10-12:35

The Wonders of a 400 MHz HTS Magnet NMR System, How It Works and Our Results at Amgen

Presenter: Maria Victoria Silva Elipe (Amgen, Inc.)

All Authors: MARIA VICTORIA SILVA ELIPE (Amgen, Inc.)

The discovery of new ceramic materials containing Ba-La-Cu oxides in 1986 exhibiting superconductive properties at high temperatures (35 K or higher), opened a new world of opportunities for NMRs and MRIs to move away from liquid cryogens, recognized with the Nobel Prize in Physics in 1987. A prototype 400 MHz high temperature superconducting (HTS) power-driven magnet NMR spectrometer was installed at Amgen's chemistry laboratory to be tested for a variety of applications, structure analysis, reaction monitoring, and CASE-3D studies with RDCs. The HTS "cryofree" magnet does not required liquid cryogens refills and has a smaller footprint than a comparable low temperature superconducting (LTS) magnet, with stability as the unknown factor of this technology. Our evaluation of its performance was successful.

10:45 AM - 12:35 PM, MONDAY
MOF: The Dynamics of Life!

Gosia Marjanska (University of Minnesota) presiding
Woodlands

MOF 10:45-11:10

Direct imaging of neuronal activity (DIANA) with high temporal and spatial resolution

Presenter: Jang-Yeon Park (Sungkyunkwan University)

All Authors: Jang-Yeon Park (Sungkyunkwan University)

There has been a longstanding demand for noninvasive neuroimaging with high spatiotemporal resolution. Recently, a novel approach was proposed that enables direct imaging of neuronal activity (DIANA) with millisecond temporal resolution, demonstrated through in vivo mouse brain imaging at 9.4 T. DIANA showed high correlation with neuronal spiking, capturing the propagation of neuronal activity along neural circuits. DIANA contrast mechanism may be due to T2 changes induced by neuronal activation. From a biophysical perspective, these T2 changes may be caused by cell swelling and changes in hydrated water molecules. Finally, because DIANA is directly related to neural activity, including spontaneous activity as well as response to stimulation, it may require different considerations in data acquisition and analysis than BOLD-fMRI.

MOF 11:10-11:30

Steady State T1rho-weighted Breast MRI at Ultra-low Field (6.5 mT)

Presenter: Sheng Shen (1. A. A. Martinos Center for Biomedical Imaging, Department of Radiology, MGH, 2.Harvard Medical School)

All Authors: 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); Mansi A. Saksena (Massachusetts General Hospital); Stephen E. Ogier (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 Harvard University)

High-risk breast cancer screening conventionally employs x-ray imaging, raising concerns about ionizing radiation. This study explores ultra-low field (ULF) MRI, specifically T1rho-weighted imaging, as a radiation-free alternative. Despite inherent signal-to-noise ratio (SNR) challenges in ULF settings, a novel steady-state (SS) T1rho-weighted sequence with enhanced SNR is proposed. Phantom imaging illustrates contrast distinctions between bSSFP and SS-T1rho scans, emphasizing T1rho contrast contributions. In-vivo breast imaging of a healthy participant confirms the efficacy of this sequence. Leveraging the balanced steady state of magnetization, the SS-T1rho sequence produces MRI images with T1rho contrast and reasonable SNR, promising advanced breast imaging in ULF settings for screening and

diagnosis. Future studies will extend the evaluation to breast cancer patients.

MOF 11:30-11:50

Correlation and Coordination during Membrane-Crossing of a Peptide-Based Magnetic Resonance Imaging Agent for Molecular Imaging Revealed by Spectroscopy, Imaging and Theoretical Calculation

Presenter: Shangwu Ding (National Sun Yat-sen University)

All Authors: Shangwu Ding (National Sun Yat-sen University)

How a molecule or an ion crosses the cellular membrane is a problem crucial for understanding many fundamental biological problems and has been studied for over one century, but there are many open questions such as the precise microscopic mechanistic picture of membrane-crossing process, the effects of transmembrane concentration difference of various ions, membrane morphology and membrane dynamics, the interaction among the membrane-crossing species etc. In this work, multi-scale spatiotemporal correlation and coordination during membrane-crossing of intracellular MRI contrast agent are observed, measured and discussed with NMR spectroscopy, multi-scale quantum mechanics/molecular mechanics (QM/MM) calculation as well as molecular dynamics simulation, showing both complexity and regularity of the dynamic process of a contrast agent crossing the membrane.

MOF 11:50-12:10

Investigating The Impact Of Diet, Gender And Genetics On Liver Metabolism Using Hyperpolarized MRSI

Presenter: Irene Marco-Rius (Institute for Bioengineering of Catalonia (IBEC))

All Authors: Alba Herrero Gomez (Institute for Bioengineering of Catalonia); Vicent Ribas (Institut d'Investigacions Biomèdiques August Pi i Sunyer (FRCB-IDIBAPS), CIBERDEM); David Gomez Cabeza (Institute for Bioengineering of Catalonia); Samantha Moron (Institut d'Investigacions Biomèdiques August Pi i Sunyer (FRCB-IDIBAPS), CIBERDEM); Joan-Marc Servitja (Institut d'Investigacions Biomèdiques August Pi i Sunyer (FRCB-IDIBAPS), CIBERDEM); Irene Marco-Rius (Institute for Bioengineering of Catalonia)

We propose to use hyperpolarization-enhanced magnetic resonance spectroscopic imaging as a tool to identify early-stage metabolic dysfunction-associated fatty liver disease (MAFLD). Our choice of experimental groups accounts for MAFLD risk factors like age, gender, and genetic predisposition. By examining the livers of wildtype mice that have been fed standard or high-fat diets, and genetically obese mice fed a standard diet, we aim to uncover the metabolic fingerprint of MAFLD.

MOF 12:10-12:35

The Role of Clinical Proton MR Spectroscopy of the Central Nervous System

Presenter: Eva-Maria Ratai (Massachusetts General Hospital/Harvard Medical School)

All Authors: Eva-Maria Ratai (Massachusetts General Hospital/Harvard Medical School)

Proton magnetic resonance spectroscopy (1H MRS) offers the unique ability to measure brain metabolite levels in a non-invasive manner. 1H MRS has evolved from a research tool into a clinical neuroimaging modality and can easily be added to clinical MRI scans without the need of extra hardware. 1H MRS has significantly impacted patient management in terms of diagnosis, prognosis and monitoring treatments. Herein, we will discuss the usefulness of clinical 1H MRS in the evaluation of neoplasms vs. MRI mimics, tumor recurrence vs. treatment effect, demyelinating disorders, infectious brain lesions and pediatric disorders (e.g. inherited metabolic diseases). The growing list of disorders for which MRS may additionally contribute to patient management extends to epilepsy, hypoxia and neurodegenerative diseases.

4:00 PM - 5:50 PM, MONDAY
MOG: Biomolecular Solutions I
Rieko Ishima (University of Pittsburgh) presiding
Merrill Hall

MOG 04:00-04:25

Rewiring Allosteric Crosstalk and Specificity in CRISPR-Cas9

Presenter: George Lisi (Brown University)

All Authors: George Lisi (Brown University)

CRISPR-Cas9 is an RNA-guided DNA endonuclease with exciting genome editing applications. At its core is an allosteric mechanism that functionally couples the catalytic and nucleic acid recognition domains. However, the intricate conformational changes occurring during DNA binding and the allosteric mechanism of nuclease function is unknown. Further, the means by which mutations improve the specificity of Cas9 is also unclear. Using synergistic NMR and MD studies of multiple Cas9 domains, we report that specificity-enhancing mutations rewire allostery via site-specific changes in multi-timescale dynamics. Despite disparate mutation regions, sites of specificity-enhancement induce a consistent structural pattern that increases flexibility of the nucleic acids. This change propagates across the entire protein, altering dynamic pathways of information transfer connecting the catalytic domains.

MOG 04:25-04:45

Visualizing a Two-State Conformational Ensemble in Stem-Loop 3 of the Transcriptional Regulator 7SK RNA

Presenter: Catherine Eichhorn (University of Nebraska Lincoln)

All Authors: Catherine Eichhorn (University of Nebraska Lincoln)

Structural plasticity is integral to RNA function. Here, we integrate solution NMR spectroscopy and chemical probing approaches to visualize a two-state conformational ensemble for the central stem-loop 3 (SL3) of 7SK RNA, a critical element for 7SK RNA function in transcription regulation. We find that the SL3 distal end undergoes slow exchange between two equally populated yet structurally distinct states. We rationally designed constructs that lock SL3 into a single state and demonstrate that both NMR and chemical probing data fit to a linear combination of the two states. These results provide new insights into RNA structural dynamics and demonstrate the utility of integrating NMR spectroscopy with chemical probing to gain quantitative insights into RNA conformational ensembles.

MOG 04:45-05:05

Mechanistic roles of enzyme tails through the lens of semi-synthesis and NMR

Presenter: Thibault Viennet (Aarhus University)

All Authors: Thibault Viennet (Aarhus University); Daniel R. Dempsey (Boston University); Nam Chu (Ohio State University); Hwan Bae (Harvard Medical School); Jiazhi Li (Harvard Medical School); Richard I. Gregory (Harvard Medical School); Philip A. Cole (Harvard Medical School); Haribabu Arthanari (Harvard Medical School/Dana Farber Cancer Institute)

Many cell cycle enzymes contain so-called tails, short terminal disordered regions that are often overlooked because their high flexibility impedes X-ray crystallography or cryo-electron microscopy studies. However, they have important mechanistic roles regulated by post-translational modifications such as phosphorylation.

Obtaining site-specific and stoichiometric phosphorylated samples for NMR studies necessitates to make use of the chemical biology toolbox. Here, we used protein semi-synthesis and intein-mediated ligations to perform segmental labeling of phospho-proteins.

We successfully investigated the regulation mechanisms of a range of enzymes including PTEN, AKT and METTL1. Interestingly, despite their common regulation by tail phosphorylation, these enzymes rely on very different mechanisms of activation/inhibition.

MOG 05:05-05:25

Structure of LARP7 protein p65–telomerase RNA complex in telomerase revealed by cryo-EM and NMR

Presenter: Yaqiang Wang (University of California Los Angeles)

All Authors: Yaqiang Wang (University of California Los Angeles)

La-related protein 7 (LARP7) are a family of RNA chaperones that protect the 3'-end of RNA. In Tetrahymena thermophila telomerase, LARP7 protein p65 together with telomerase reverse transcriptase and telomerase RNA (TER) form the core RNP. To date, only part of p65 and their interactions with TER have been structurally characterized.

We investigated the structure, dynamics and function of full-length p65 by using NMR and electron microscopy. Three previously unknown helices are identified, one in the intrinsically disordered N terminus that binds La module, one that extends La module, and another stabilizes p65–TER interactions. The structure of full-length p65 also sheds light on the biological roles of genuine La and LARP7 proteins as RNA chaperones and core RNP components.

MOG 05:25-05:50

Combining Methyl-TROSY and NMR Relaxation Experiments to Probe the Mechanisms of Chaperone Dysfunction in Disease

Presenter: Rina Rosenzweig (Weizmann Institute of Sceince)

All Authors: Rina Rosenzweig (Weizmann Institute of Sceince)

Molecular chaperones are complex protein machineries that protect our cells from misfolding and aggregation, and their mutations are implicated in multiple human disorders, including neurodegeneration, myopathies, and cancer. Due to their large size and dynamic nature, though, little is known regarding how such mutations affect the chaperones' function, leading to disease.

Here we demonstrate how a combination of methyl-TROSY NMR experiments and dynamic CPMG-RD and relaxation measurements can unveil the mechanism of function of chaperones, deciphering the core of their malfunction in disease. For example, using Methyl-TROSY NMR we show that pathogenic mutants of the 1MDa DNAJB6 differ structurally from the WT chaperone, allowing the unregulated recruitment and hyperactivation of Hsp70, depleting its levels in cells and causing the disease.

4:00 PM - 5:45 PM, MONDAY
MOH: Instrumentation I

Rachel Martin (University of California, Irvine) presiding

Chapel

MOH 04:00-04:25

Mechanical Modulation of DC Magnetic Fields: Instrumentation and Biological Applications

Presenter: Shoujun Xu (University of Houston)

All Authors: Shoujun Xu (University of Houston)

There are various modulations of magnetic fields that lead to different techniques. For example, magnetic resonance spectroscopy relies on chemical modulation of the magnetic fields, and imaging is achieved via spatial modulation. Intrinsically, dc magnetic fields do not contain chemical signatures. However, by introducing mechanical modulation on the magnetic labels of biological molecules, we can precisely reveal the strength of intermolecular interactions and monitor the motion of biological complexes. We will describe detailed instrumentation of integrating ultrasound generation and atomic magnetometers. Several biological applications will be demonstrated. In addition, we will compare magnetic readout with optical imaging and fluorescent detection in terms of molecular resolution and detection efficiency.

MOH 04:25-04:45

SSNMR Spinning Module Optimization for Minimimal Perturbance of the Applied Magnetic Field achieving Sub-Hertz Resolution on 13C Adamantane

Presenter: Jasmin Schoenzart (Colorado School of Mines)

All Authors: Jasmin Schoenzart (Colorado School of Mines); Ruixian Han (University of Wisconsin - Madison); Thomas Gennett (Colorado School of Mines); Chad Rienstra (University of Wisconsin-Madison); John Stringer (PhoenixNMR, LLC)

Magic-angle spinning (MAS) solid-state NMR methods are crucial in many areas of biology and materials science. Conventional probe designs have often been specified with 0.1 ppm magnetic field resolution, which is a limitation for many of these modern scientific applications. Here we describe a novel 5 mm MAS module design that significantly improves the linewidth and line shape for solid samples by an improved understanding of the magnetic susceptibility of probe materials and geometrical symmetry considerations, optimized to minimize the overall perturbation to the applied magnetic field (B0). The improved spinning module will only require 1st and 2nd order shimming adjustments to achieve a sub-hertz resolution of 13C resonances of adamantane and exhibiting baseline resolved J-couplings.

MOH 04:45-05:05

A High-Volume and High-Frequency Resonator for DNP-NMR

Presenter: Adam Altenhof (Los Alamos National Laboratory)

All Authors: Adam Altenhof (Los Alamos National Laboratory); Qing Yang (University of Stuttgart); Michal Kern (University of Stuttgart); Shaun G. Newman (Los Alamos National Laboratory); Jens Anders (University of Stuttgart); Michael Malone (Los Alamos National Laboratory)

Efficient generation of an applied magnetic field (B1) is crucial for implementing DNP-NMR. This can be accomplished with inductive DNP resonators. Inductors are difficult to implement at high frequencies (i.e., > 1 GHz) depending on sample volume, where the dimensions must decrease to avoid B1 inhomogeneities; resulting volumes are limited to < 100 µL. A simple low-cost and high-volume resonator based on a birdcage (BC) coil can be used for ODNP-NMR. The BC uses no tuning capacitors and relies on its geometry and parasitic capacitance to resonate around 1.6 GHz with an inner diameter of 24 mm. 45-fold enhancements are measured on several mL of TEMPO in H2O while sample heating is only +2 °C when using high-power.

MOH 05:05-05:25

Benchtop NMR Spectroscopy at Record High Fields

Presenter: John Price (University of Colorado, Boulder)

All Authors: John Price (University of Colorado, Boulder); Hilton Chan (Q Magnetics, LLC); John Frost (Q Magnetics, LLC); Rainer Malzbender (RMP Associates); Tyler Ozvat (Q Magnetics, LLC)

We describe a high-resolution permanent-magnet proton NMR spectrometer with a Larmor frequency of 125 MHz. As far we know, this is the highest field yet achieved for a non-superconducting NMR spectrometer. The magnet assembly uses radial and axial hard ferromagnets, soft ferromagnetic poles, and a soft ferromagnetic flux return. An electronic shim system corrects 16 aberrations: 3 linear, 5 quadratic, 7 cubic, and 1 quartic. Fluid samples are delivered to the center of the magnet by flow instead of by tube insertion, making the design ready for hyphenated and automated applications. Field stabilization is provided by micro-degree temperature control and full-spectrum cross-correlation for multi-scan averaging.

MOH 05:25-05:45

Novel Approaches in NanoMRI for Probing Atomic-Scale Material Structure

Presenter: Raffi Budakian (University of Waterloo)

All Authors: Raffi Budakian (University of Waterloo)

Prior to the development of MRI, NMR diffraction (NMRd) was proposed as a method to investigate the structure of crystalline materials. When realized on the atomic scale, NMRd would be a powerful tool for studying materials structure, combining the spectroscopic capabilities of NMR with spatial encoding at condensed matter's fundamental length-scale. In this talk, I will present a nanoMRI platform for achieving angstrom-scale NMRd measurements.

8:45 AM - 10:10 AM, TUESDAY
TOA: Biomolecular Solids II

Len Mueller (University of California, Riverside) presiding

Merrill Hall

TOA 08:45-09:10

Solid-State NMR Studies of DNA-Protein Complexes

Presenter: Christopher Jaroniec (The Ohio State University)

All Authors: Christopher Jaroniec (The Ohio State University)

I will discuss our recent studies of DNA-protein complexes by solid-state NMR methods aimed at characterization of: (i) histone protein structure and conformational dynamics within nucleosome arrays representative of condensed chromatin and (ii) DNA base pairing and hydrogen bonding in DNA complexes with proteins and small molecules.

TOA 09:10-09:30

Studies of Interactions Between Biological Membranes and Amyloid-Beta (Aβ) Aggregates by Solid-State NMR Spectroscopy

Presenter: June M. Kenyaga (Binghamton University)
All Authors: June Kenyaga (Binghamton University)

Cellular membrane has been shown to serve as a platform for the pathological aggregation of Aβ peptides in Alzheimer's Disease (AD). Despite the biological significance of membranes in amyloidosis of Aβ, limited work has been done to address both the early stage Aβ-membrane interactions and probe these interactions in their native environments. Using ssNMR spectroscopic techniques, we a) determined the early-stage residue-specific contacts between Aβ40 and lipids phosphates headgroups and within the Aβ40 strands and proposed a schematic mechanism of Aβ40-lipid interactions that drive the nucleation process for fibril formation. b) explored preliminary applications of in-cell ssNMR on live mouse neuroblastoma cells and demonstrated the feasibility of MAS-ssNMR spectroscopy in probing Aβ-membrane interactions in their native cellular environments.

TOA 09:30-09:50

Modulation of Protein Dynamics by Protein-Protein Interactions Through an Intermolecular Dynamic Network Observed by Solid-State NMR.

Presenter: Sara Medina Gomez (TU Dortmund University)
All Authors: Sara Medina Gomez (TU Dortmund University); Suresh K. Vasa (TU Dortmund University); Rasmus Linser (TU Dortmund University)

The dynamical properties of individual amino acids are modulated by their chemical environment, this can be influenced by intramolecular networks. We use a combination of solution and solid-state NMR to decipher the effect of mutagenesis of the R21 residue in the monomer compared to its effect in the presence of protein-protein interactions due to the crystal lattice. The study assesses the dynamical properties of SH3wt and mutant via Redfield relaxation (R1, R2, and heteronuclear steady-state NOE) for 15N and spectral-density mapping, in addition to 15N near-rotary-resonance relaxation dispersion (NERRD) in the solid state. The data show that intermolecular interactions can bring in individual elements that together form a dynamic network.

TOA 09:50-10:10

The KirBac1.1 inward-rectifier K+ channel is opened cooperatively by anionic lipids as observed by solid-state NMR

Presenter: Benjamin Wylie (Texas Tech University)
All Authors: Evan Jamaal van Aalst (Texas Tech University); Maryam Yekefallah (Texas Tech University); Roy van Beekveld (Utecht University); Isaac Eason (Texas Tech University); Borcik Collin (Univeristy of Wisconsin); Reza Amani (Massachusetts Institute of Technology); Markus Weingarth (Utrecht University)

The prokaryotic channel KirBac1.1 shares its fold and regulatory mechanisms with homologous human channels. Using solid-state NMR (SSNMR), SSNMR-driven simulated annealing structure calculations, and coarse-grain molecular dynamics (CG-MD) we resolved an intricate allosteric network between the inner gate, outer gate, and intracellular Kir domain. This network is governed by positive anionic lipid-loading allostery. We present a SSNMR titration of 13C-phosphadityl glycerol (PG) in the presence of KirBac1.1 which reveals sigmoidal lipid binding. Next, we identify the locus of PG binding and the activated channel structure. Solvent accessibility measurements and three-dimensional 13C-13C-13C dipole-assisted rotational resonance (DARR) experiments were used to solve the structure of KirBac1.1 in both the activated and inactivated states.

8:45 AM - 10:10 AM, TUESDAY
TOB: Materials I

Yusuke Nishiyama (Riken-JEOL, JEOL Resonance) presiding
Chapel

TOB 08:45-09:10

Probing Oxygen Exchange and Water Stability of Metal-Organic Frameworks using 17O solid-state NMR

Presenter: Frédérique Pourpoint (UCCS - Centrale Lille)
All Authors: Frédérique Pourpoint (UCCS - Centrale Lille); Florian Venel (UCCS - Centrale Lille); Christophe Volkringer (UCSS-Centrale Lille); Jessica Špačková (ICG - Université Montpellier); Sébastien Mittelette (ICG - Université de Montpellier); Thomas-Xavier Métro (ICG - Université de Montpellier); Danielle Laurencin (CNRS); Olivier Lafon (UCCS - Université de Lille)

MOFs are built from the three-dimensional association of metal clusters and organic ligands. Better understanding their stability in the presence of water is critical for their use in industrial processes. Solid-state NMR is a promising tool to probe the structure of defects in presence of water. Nevertheless, the observation of oxygen atoms is often prevented by the low natural abundance of 17O isotope. We explore different approaches for 17O enrichment of UiO-66 (H217O or 17O-enriched ligand from mechanochemistry). We observe exchange of O atoms between the different O sites. NMR evidences the lability of the Zr-O bonds, even when the long-range structure is preserved. Characterizing this lability is crucial to understand the degradation of MOFs in the presence of water.

TOB 09:10-09:30

SMARTER Crystallography of Porous Functional Materials – Strategies to Reveal Supramolecular Assemblies in Host Pore Systems

Presenter: Eric Breynaert (NMRCoRe, KU Leuven)
All Authors: Eric Breynaert (NMRCoRe, KU Leuven); Christine E.A. Kirschhock (COK-kat - KU Leuven); Alysson Morais (Katholieke Universiteit Leuven); Vinod C. Chandran (NMRCoRe - KU Leuven); Sambhu Radhakrishnan (NMRCoRe - KU Leuven)

Industrial-scale chemical processes heavily depend on intricate interactions among metal cations, anions, water, organics, and framework atoms, residing within porous functional materials. Understanding these processes at a molecular level is crucial for advancing material synthesis and applications. Investigating molecular compound interactions in pore systems however remains challenging, often requiring a combination of characterization techniques.

SMARTER Crystallography is a convergence research approach to unveil structure and properties of complex materials. Integrating solid-state NMR with other spectroscopies, diffraction, scattering, and modeling, unprecedented insights into guest structures within functional porous material pores can be achieved. This contribution will mostly use zeolites as examples to outline general approaches to characterizing porous functional host-guest assemblies, resolving pore content and supramolecular organization of occluded molecular species.

TOB 09:30-09:50

In Situ Chemical Shift Imaging Investigation of ZIF-67/Activated Carbon Electrochemical Supercapacitor Cell

Presenter: Mark Bovee (US Naval Research Laboratory)
All Authors: Mark Bovee (US Naval Research Laboratory); Carlos M. Hangarter (US Naval Research Laboratory); Matthew Laskoski (US Naval Research Laboratory); Christopher Klug (US Naval Research Laboratory)

To improve supercapacitor performance, it's thought metal-organic frameworks (MOFs) can be incorporated as electrode materials due to their high porosity and redox-active metal centers and functionalized organic linkers; however, low conductivity and chemical stability have reduced their applicability. Here, we utilize in situ chemical shift imaging to investigate charge storage mechanisms and drawbacks of a zeolitic imidazolate framework 67 (ZIF-67) electrode in an electrochemical cell with 1M KOH electrolyte. We notice dramatic changes in the electrolyte's chemical shift most notable near the ZIF-67 electrode that are exacerbated by applying voltage and attribute this to ZIF-67's degradation under basic conditions. This provides us with insight on this MOF's behavior in device-like configurations and helps guide us in choosing MOF electrode materials.

TOB 09:50-10:10

Revealing Molecular Mechanisms in Hierarchical Nanoporous Carbon via Nuclear Magnetic Resonance

Presenter: Haiyan Mao (Stanford University)

All Authors: Haiyan Mao (Stanford University); David Halat (UC Berkeley & LBNL); Alexander Pines (Uni); Yi Cui (Stanford University); Jeffrey Allen Reimer (University of California, Berkeley)

Hierarchical nanoporous carbons (HNC) have been proven to be an effective adsorbent for CO2, although questions remain regarding the hierarchical structure regulation, the adsorption mechanisms of adsorbate uptake and interactions within the HNC. We synthesize HNC from wood, using a microwave-induced heating method incorporating K2CO3 activation. NMR chemical shifts are consistent with ring current effects from the adsorbent. Our NMR technique provides a convenient way to quantitate adsorption of adsorbate in HNC. VOCs vapor adsorption results show NMR chemical shift changes with time, suggesting initial adsorption into mesopores, followed by diffusion into micropores. Schroeder's Paradox is demonstrated by differences in observed shifts for adsorbed liquid vis-à-vis vapor phase in these HNC. These HNC show high CO2 adsorption capacity.

8:45 AM - 10:10 AM, TUESDAY
TOC: Adding Contrast

Laura Walkup (Cincinnati Children's Hospital) presiding

Woodlands

TOC 08:45-09:10

Simple hyperpolarization chemistry for more substrates, new NMR physics, and first in-vivo metabolic MRI

Presenter: Thomas Theis (North Carolina State University)

All Authors: Thomas Theis (North Carolina State University)

Parahydrogen Induced Polarization (PHIP) is an attractive hyperpolarization modality because of its experimental ease and simplicity. In particular, the reversible-exchange variant known as SABRE (Signal Amplification By Reversible Exchange) is attractive because of its continuous repeatability. Here we describe recent progress with a) expanding the substrate scope of SABRE, b) explorations of parahydrogen induced RASERs (Radiofrequency Amplification of Stimulated Emission of Radiation) that emerge when very large degrees of hyperpolarization are created and placed in a resonant NMR circuit, and c) our first experiences with deploying SABRE polarized substrates in vivo for metabolic imaging. The described progress is also used for combinations of simple PHIP approaches with low-field NMR and MRI to deliver scalable chemical analysis and medical imaging techniques.

TOC 09:10-09:30

Whole Abdominopelvic Variable Resolution DNP 13C MRI Imaging for Advanced Prostate Cancer Patients

Presenter: Tanner Nickles (UCSF)

All Authors: Tanner Nickles (UCSF); Hsin-Yu Chen (UCSF); Yaewon Kim (University of California, San Francisco); Phillip M. Lee (UCSF); Daniel Gebrezgiabhier (UCSF); Robert Bok (UCSF); Ivan de Kouchkovsky (UCSF); Michael Ohliger (UCSF); Zhen Wang (UCSF); Peder Larson (UCSF); John Kurhanewicz (UCSF); Rahul Aggarwal (UCSF); Jeremy Gordon (UCSF); Daniel Vigneron (UCSF)

Monitoring the progression or response of advanced prostate metastases is a current clinical unmet need that is not reliably delineated with current CT and PET. Here, we developed a high-resolution whole abdominopelvic [1-13C]pyruvate hyperpolarized (HP) MRI approach for the metabolic biomarker characterization of metastases in prostate cancer patients. A variable-resolution imaging approach was used to provide high-resolution [1-13C]pyruvate, robust spatiotemporal denoising and B1+ variation correction methods were used to quantify the rate-constant for the conversion of [1-13C]pyruvate to lactate, kPL. Improved conspicuity of [1-13C]pyruvate distribution and kPL conversion maps of metastatic prostate lesions were achieved with the new approach.

TOC 09:30-09:50

A Family of Novel DNP Probes to Non-invasively Detect 2-Hydroxyglutarate by in vivo Magnetic Resonance Spectroscopy

Presenter: Norikazu Koyasu (National Institutes of Health / National Cancer Institute)

All Authors: Norikazu Koyasu (National Institutes of Health / National Cancer Institute); Chandrasekhar Mushti (National Institutes of Health); Rolf E. Swenson (National Institutes of Health); Murali Cherukuri Krishna (National Institutes of Health / National Cancer Institute); Kazutoshi Yamamoto (National Institutes of Health / National Cancer Institute)

Gliomas are the most common malignant primary brain tumors with fatal prognosis. While their therapies can be often combinational, and/or involve some period of active suveillance. Developing non-invasive imaging methods to monitor the tumor progressions and the therapeutic responses at earlier stages are desperately needed to improve individual survival rates and their quality of lives significantly. Most of WHO grade 2 and 3 gliomas in adults possess mutations in isocitrate dehydrogenase to produce the 2-hydroxyglutarate (2HG) from α-ketoglutarate (aKG) metabolically. Here, we have been developing the family of newly synthesized hyperpolarized aKG probes which have enahnced sensitivities, resolutions, selectivity, and membrane permeability, which enable us to visualize αKG metabolism in vivo utilizing hyperopolarized-13C-MRS in cancer tissues non-invasively.

TOC 09:50-10:10

High-Sensitivity Glutamate Quantification with CEST, Water-Resonant Spin-Locking, and MR Fingerprinting

Presenter: David Korenchan (Athinoula A. Martinos Center for Biomedical Imaging)

All Authors: David Korenchan (Athinoula A. Martinos Center for Biomedical Imaging); Or Perlman (Department of Bio-Medical Engineering, Tel Aviv University); Christian T. Farrar (Athinoula A. Martinos Center for Biomedical Imaging)

Glutamate mapping in vivo using high-sensitivity CEST MRI would greatly benefit cancer characterization and treatment monitoring. However, the amine protons of glutamate exchange rapidly, requiring MRI scanners above 3 T to detect them sensitively. We investigated a new imaging method of quantifying glutamate concentration and proton exchange rate that combines CEST, water spin-locking, and magnetic resonance fingerprinting. We found that incorporating spin-locking elements into a fingerprinting schedule at 9.4 T enhances chemical exchange-dependent contrast and improves parameter map accuracy. Initial data acquired on a 4.7 T scanner also suggest that spin-locking provides a greater benefit to quantification at lower field strengths. These technical developments may facilitate studies of how cellular glutamate changes in the context of many diseases.

10:45 AM - 12:30 PM, TUESDAY
TOD: Biomolecular Solutions II

Lucia Banci (University of Florence-CERM) presiding

Merrill Hall

TOD 10:45-11:10

NMR spectroscopy of Biomolecular Condensates

Presenter: Markus Zweckstetter (German Center for Neurodegenerative Diseases)

All Authors: Markus Zweckstetter (German Center for Neurodegenerative Diseases)

Liquid-Liquid phase separation has emerged as fundamental process underlying the formation of biomolecular condensates. Insights into the composition and structure of biomolecular condensates is, however, complicated by their molecular complexity and dynamics. In this presentation I will illustrate how NMR spectroscopy can be used to obtain unique insights into the physicochemical composition of biomolecular condensates.

TOD 11:10-11:30

Biomolecular Condensate Remodels the Conformational Equilibria of SOD1 Towards Aggregation-Prone States

Presenter: Rashik Ahmed (Hospital for Sick Children)

All Authors: Rashik Ahmed (Hospital for Sick Children); Rhea Hudson (Hospital for Sick Children); Mingyang Liang (University of Toronto); Julie D. Forman-Kay (University of Toronto, Hospital for

Sick Children); Lewis E. Kay (University of Toronto, Hospital for Sick Children)

Biomolecular condensates formed via phase separation can change the local concentration of biomolecules to enhance (inhibit) enzymatic activity and modulate specificity. The emergent properties of condensates can also alter protein function by remodeling their conformational free-energy landscapes. However, site-specific information on the conformation of folded proteins in condensates remains enigmatic owing to spectroscopic limitations. Here, we highlight the application of the delayed decoupling HMQC sequence, which affords significant enhancements in sensitivity for fast-relaxing signals, to probe the conformational equilibria of SOD1 inside condensates. Notably, we show that the condensed phase solvent environment dramatically biases the conformation of ALS-associated SOD1 towards unfolded states that are prone to aggregation. This observation underscores a mechanism by which phase separation can accelerate neurodegenerative disorders.

TOD 11:30-11:50

Using CS-ROSETTA to Probe Assembly of Intrinsically Disordered Spider Silk Proteins in Solution

Presenter: David Onofrei (San Diego State University)

All Authors: David Onofrei (San Diego State University); Julian Aldana (San Diego State University); Gregory P. Holland (San Diego State University)

Spider silk proteins represent a subclass of intrinsically disordered proteins (IDPs) that are challenging to study because they are highly repetitive, extremely large (300-350 kDa), and contain a central region that lacks a single native structure. These difficulties diminish the usefulness of traditional techniques such as nuclear magnetic resonance (NMR) which suffer from low resolution and spectral overlap. We used a combination of 13C-detected NMR experiments and CS-ROSETTA to simulate fragments of the major ampullate spidroin (MaSp1) silk protein. These fragments allowed us to build a model showing how recurrent geometric motifs throughout the IDP ensemble explain the NMR data. These NMR results are being combined with MD simulations to build a more comprehensive model of spider silk protein structure.

TOD 11:50-12:10

In-situ electric field in NMR to study charge, ligand binding and orientation of molecules

Presenter: Ulrich Scheler (Leibniz-Institut für Polymerforschung Dresden e.V.)

All Authors: Ulrich Scheler (Leibniz-Institut für Polymerforschung Dresden e.V.)

To investigate the electrostatic interaction affecting ligand binding and complex formation experiments with an electric field applied in-situ have been perfomed. Carged molecules move in the direction of the electric field while molecules exhibiting a dipole moment will partially orient. From the electrophoretic mobility the effective charge of molecules and complexes is dtermined and thus weak binding directly quantified without the need of a titration series. A robust data acquisition scheme and resolution enhancement by linear prediction are discussed.

In a J-resolved experiment weak orientation is determined with electric field applied in the indirect dimension. This allows measuring weak orientaion in one dimension while retaing full resolution in the other. The weak orientation scales with the electric field.

TOD 12:10-12:30

Structure Determination of RNA Tetraloop Ensembles with Integrated NMR/Molecular Dynamics Based Methods

Presenter: David Leopold (Johann Wolfgang Goethe-University Frankfurt)

All Authors: David Leopold (Johann Wolfgang Goethe-University Frankfurt); Andreas Oxenfarth (Johann Wolfgang Goethe-University Frankfurt); Felix Kümmerer (University of Copenhagen); Anna Wacker (Johann Wolfgang Goethe-University Frankfurt); Hendrik R. A. Jonker (Johann Wolfgang Goethe-University Frankfurt); Christian Richter (Johann Wolfgang Goethe-University Frankfurt); Harald Schwalbe (Johann Wolfgang Goethe-University Frankfurt); Kresten Lindorff-Larsen (University of Copenhagen)

For an optimization of the computational tools, used for structural and dynamical analysis of RNA a set of reference systems under physiological conditions is needed. Currently the best known tetraloop is the UUCG as the available information's of the UUCG are plentifully. But other naturally occurring tetraloops like the GNRA family are less extensively studied. We determined the structural ensemble of 14mer RNA tetraloops. Therefore, cross correlated relaxation rates, J couplings and residual dipolar couplings were incorporated for the NOE based structure determination. A refinement of the CUUG with molecular dynamics, which used the experimental data for the reevaluation of its output was performed and is planned for the other tetraloops.

10:45 AM - 12:35 PM, TUESDAY
TOE: Instrumentation II

Thorsten Maly (Bridge 12 Technologies, Inc.) presiding
Chapel Hall

TOE 10:45-11:10

A Semiconductor-chip Based Miniature Magnetic Field Sensor (MMFS) aimed at MRI Image Calibration

Presenter: Guang Yang (Harvard University)

All Authors: Guang Yang (Harvard University); Jie Wang (Harvard University); Juncheng Xu (Harvard University); Daniel Krügera (Harvard University); Aoyang Zhang (Harvard University); Ross W. Mair (Harvard University); Yi-qiao Song (Harvard University); Donhee Harvard (Harvard University)

This presentation addresses MRI challenges arising from magnetic field perturbations associated with gradient, causing distortion and errors. Introducing a Miniature Magnetic Field Sensor (MMFS) based on an NMR chip aims to enhance image quality by sensing gradient fields during MRI experiments. Data (SNR 36 dB, 68 ms T2*) collected from a 3T MRI scanner validated the MMFS prototype. Testing the 2D Echo-planar Imaging sequence revealed an aligned EPI gradient waveform with readout and phase-encoding blip gradients. However, imperfections in the waveform suggest potential gradient perturbations or noise. Future efforts will focus on identifying and correcting these issues, with plans to extend the spectrometer for addressing high-order field perturbations in MRI images.

TOE 11:10-11:30

From Low-Cost, Homebuilt Laboratory Prototypes to Industrial Magnetic Resonance Systems for Online Monitoring at Mine-Sites

Presenter: Thai Ly (CSIRO)

All Authors: Thai Ly (CSIRO); Richard Yong (CSIRO); Bojan Lovirc (CSIRO); Dragoslav Milinkovic (CSIRO); Giffard Roberts (CSIRO); Andrew Curtain (CSIRO); Matthew Crowther (CSIRO); Peter J. Coghill (CSIRO); David G. Miljak (CSIRO)

Antiferromagnetic Zero-field Nuclear Magnetic Resonance (ZFNMR) allows for low-cost, homebuilt laboratory systems that are scalable to industrial applications. ZFNMR does not require a DC applied field, enhancing mobility that is reduced by cryogenic cooling. Furthermore, quantification NMR eliminates the need for cumbersome gradient coils. Thus, a ZFNMR instrument can be scaled up for real-time, non-destructive, industrial monitoring.

The presentation will describe how laboratory prototypes were successfully translated to ZFNMR conveyor analysers for the quantification of chalcopyrite in 2800 tonnes of ore per hour that is implemented at a copper mine. The journey from the technology readiness level (TRL) 4 prototypes in an ideal environment to the "field" trials and tribulations of TRL 9 MR analysers will be showcased.

TOE 11:30-11:50

47 Tesla Handheld Magnet and a Path Towards Widely Available NMR >2 GHz

Presenter: Alexander Barnes (ETH Zurich)

All Authors: Pin-Hui Chen (ETH Zurich); Chukun Gao (ETH Zurich); Nicholas Alaniva (ETH-Zürich); Snaedis Bjorgvinsdottir (ETH Zurich);

Ioannis Gr. Pagonakis (ETH Zurich); Machael A. Urban (ETH Zurich); Alexander Daepp (ETH Zurich); Ronny Gunzenhauser (ETH Zurich); Edward P. Saliba (ETH Zurich); Lea Marti (ETH Zurich); Yanhui Hu (ETH Zurich); Jasmin Schoenzart (Colorado School of Mines); Roland Riek (ETH Zurich); Alexander Barnes (ETH Zurich)

Leveraging greatly simplified small bore (3.1 mm) magnets wound with high performance high temperature superconducting (HTS) tape, we have achieved a 47 Tesla magnetic field. The 47 Tesla magnet is a cylindrical double pancake coil only 6 cm in diameter and 3 cm high; small enough to fit in the palm of your hand. We implement a simplification and miniaturization magnet fabrication strategy which greatly accelerates prototyping, development iterations, and magnet energization. We will present challenges, opportunities, and strategies for implementation of improved small yet powerful magnets for NMR and DNP. We aim to improve the magnetic field

homogeneity and stability in stages.

TOE 11:50-12:10

Extreme Line Narrowing with Matched Decoupling

Presenter: Matthew Augustine (UC Davis)

All Authors: Javier Aztiazarain (UC Davis); Matthew Augustine (UC Davis)

Carefully matching the rf field amplitude of the 1H decoupler w1 to the scalar J coupling for heteronuclear coupled spin systems in unshimmed, inhomogeneous magnets produces extremely narrow peaks in the NMR spectrum of the heteronucleus. Both average Hamiltonian theory and matrix algebra suggest that the origin of the matching condition, w1 = (gH/gS)(J/2) including a ratio of gyromagnetic ratios, is an interference between the inhomogeneous static field and the scalar J coupling. Experimental 13C spectral linewidth reductions of nearly thirty – fold are readily observed. The theoretical upper limit of this resolution improvement is related to the J coupling and heteronucleus relaxation time T1, a value in excess of 2JT1 ~ 2(200 Hz)(5 s) = 2,000 for 13C.

TOE 12:10-12:35

Frugal NMR Spectroscopy

Presenter: Aldrik Velders (Wageningen University)

All Authors: Aldrik Velders (Wageningen University); Sander Baas (Wageningen University)

Commercial NMR spectrometers, and the major subcomponents they consist of, are complex and expensive pieces of technology; moreover, often used by a selected group of users with specific needs. All this presents a big hurdle for researchers to start playing around with NMR instrument hardware, and it is also inhibiting a more wide-spread use of this important analytical tool. We have been designing and fabricating in-house various microfluidic NMR chips and microcoil-probes. More recently we have started building our own permanent magnet set ups with which we manage to execute advanced 1D and 2D high -resolution NMR experiments. In this presentation we will go into the why as well as the challenges and opportunities of home-built and DIY NMR.

10:45 AM - 12:35 PM, TUESDAY
TOF: Spin Behavior and Sequence Optimization

Sharon Ashbrook (University at St. Andrews) presiding

Woodlands

TOF 10:45-11:10

Is it time to revise the NMR Signal Processing?

Presenter: Vladislav Orekhov (University of Gothenburg)

All Authors: Vladislav Orekhov (University of Gothenburg); Amir Jahangiri (University of Gothenburg)

As the rapid development of Artificial Intelligence methods reshapes nearly every research field, what can we expect for NMR signal processing? We will present new developments in spectrum processing using deep learning and artificial neural networks (ANN). For traditional tasks, such as the reconstruction of spectra from Non-Uniform Sampling or homonuclear decoupling, a properly trained artificial neural network (ANN) can match or even outperform the best algorithmic methods in spectral quality and calculation speed. Although this will be useful in many practical applications, the main benefits may appear beyond the traditional signal processing tasks established decades ago. We present several examples where ANNs solve new problems that have not been addressed by traditional NMR signal processing algorithms.

TOF 11:10-11:30

Pulsed Dynamic Nuclear Polarization Sequence Design via Effective Hamiltonian Optimization

Presenter: José P. Carvalho (Interdisciplinary Nanoscience Center (iNANO) and Department of Chemistry, Aarhus University)

All Authors: Jose Carvalho (Interdisciplinary Nanoscience Center (iNANO) and Department of Chemistry, Aarhus University); David L. Goodwin (Interdisciplinary Nanoscience Center (iNANO) and Department of Chemistry, Aarhus University); Nino Wili (Aarhus University); Anders B. Nielsen (Interdisciplinary Nanoscience Center (iNANO) and Department of Chemistry, Aarhus University); Niels Chr. Nielsen (Interdisciplinary Nanoscience Center (iNANO) and Department of Chemistry, Aarhus University)

Over the past decade, DNP has become an established approach to enhancing sensitivity of many NMR experiments. More recently, pulsed wave DNP has emerged as a viable approach to further enhancement through increased robustness and versatility, when compared to continuous wave counterparts. Full exploitation of arbitrary waveform generators and adherence to experimental constraints may be established with "state of the art" optimal control methods. In work presented here, optimal control of the effective Hamiltonian for polarization transfer produces sequences with exceptional performance. Optimization of a basic and repeated pulse sequence element shows physically intuitive control of DNP experiments while also addressing some of the limitations of prevailing optimal control methods.

TOF 11:30-11:50

Optimal control pulses for improving filtered NOESY experiments

Presenter: David Joseph (Max Planck Institute for Multidisciplinary Sciences, Göttingen, Germany)

All Authors: David Joseph (Max Planck Institute for Multidisciplinary Sciences, Göttingen, Germany); Christian Griesinger (Max Planck Institute for Multidisciplinary Sciences, Göttingen, Germany)

The study of interactions between biological macromolecules requires the set of filtered/edited NOESY experiments to obtain intermolecular NOEs. The reliability of structural models of these complexes thus depends crucially on our ability to record filtered-NOESY spectra without spurious NOE peaks. This depends on the purging capability of the filtering scheme. Here, we design J-coupling compensated optimal control (OC) pulses with a large compensation range of 10-300 Hz to improve the filtering. We show that the OC based scheme can suppress undesired spin states with more than 99% efficiency and can cover 60 kHz at 1.2GHz. These pulses are 3-4 times shorter than the adiabatic pulses and does not rely on matched sweeping. We observe, improved filtering with the Calmodulin/Munc13-1 complex.

TOF 11:50-12:10

Characterizing Internal Dynamics Using Partially Averaged Anisotropic Interactions

Presenter: Kathrin Aebischer (ETH Zurich)

All Authors: Kathrin Aebischer (ETH Zurich); Lea Marie Becker (Institute of Science and Technology Austria); Paul Schanda (Institute of Science and Technology Austria); Matthias Ernst (ETH Zurich)

This study explores solid-state NMR spectroscopy's capacity to characterize internal molecular dynamics through measurement of partially averaged anisotropic interactions. Using numerical simulations based on the stochastic Liouville equation, we investigate the impact of motional timescales on different recoupling techniques for heteronuclear dipolar couplings (e.g. CP, REDOR), chemical-shift anisotropy tensors, and quadrupolar couplings. Three distinct dynamical regions emerge: slow motion retaining the full anisotropic interaction, fast motion yielding the scaled interaction, and an intermediate transition region. The timescales of these three regimes

depend strongly on the interaction strength but show negligible influence from the MAS frequency and recoupling technique. Overall, combining measurements of large, intermediate, and small anisotropic interactions appears most promising for characterizing motional amplitudes in different time windows.

TOF 12:10-12:35

Solid-state NMR Tools in Crystal Engineering

Presenter: Michele R. Chierotti (University of Torino)

All Authors: Michele Chierotti (University of Torino)

"Crystal engineering" is the rational design of molecular crystalline materials that relies upon self-assembly via noncovalent interactions. In crystal engineering, the structural characterization plays a key role because the resulting properties of the crystal depend on the 3D arrangement of the molecules. In recent decades, solid-state NMR has developed into an indispensable tool in crystal engineering for investigating the structure of supramolecular adducts. Its ability to locate hydrogen atoms makes it unique for characterizing weak interactions. Here we present some applications in the characterization of hydrogen bonds in terms of evaluating the proton transfer and the tautomeric and zwitterionic character of molecules within crystals. The complementarity of solid-state NMR with X-Ray diffraction and computational tools will be highlighted.

4:00 PM - 5:45 PM, TUESDAY
TOG: Hyperpolarization I

Laura Walkup (Cincinnati Children's Hospital) presiding

Merrill Hall

TOG 04:00-04:25

Recent advances in magnetic resonance imaging for chemical and bioprocess engineering

Presenter: Alexander Penn (Hamburg University of Technology)

All Authors: Alexander Penn (Hamburg University of Technology)

NMR is the gold standard for a range of analyses in the chemical and biochemical sciences. In contrast, magnetic resonance imaging (MRI), so far could only gain widespread use in medical and neuroscientific applications. However, the remarkable diversity of contrasts achievable through MRI holds significant promise for its expanded integration into the fields of chemical and bioprocess engineering. This presentation aims to illustrate how clinical MRI technology can be adapted, extended, and effectively employed to assess phase distributions, fluid flow dynamics, and thermal behavior within various multiphase flow systems. Additionally, I will introduce a novel large-bore, vertically oriented MRI system, located at Hamburg University of Technology, purposefully designed to accommodate tall columnar reactors, a common feature in chemical engineering applications.

TOG 04:25-04:45

Using Computationally Optimized Three-Dimensional Field Sequences to Improve SABRE Hyperpolarization

Presenter: Shannon Eriksson (Duke University)

All Authors: Shannon Eriksson (Duke University); Jacob Lindale (Duke University); Loren L. Smith (Duke University); Luke M. Everhart (Duke University); Warren S. Warren (Duke University)

X-SABRE is a rapidly developing hyperpolarization strategy under which microtesla magnetic fields can be used to transfer polarization to heteronuclear targets. Because the necessary fields are small, rapidly changing the magnetic field in 3 dimensions is trivial and represents a degree of freedom that has not been fully explored. Here, we introduce a multi-dimensional pulse sequence, generated using a simple evolutionary strategy and physically accurate numerical simulations, to improve polarization by an order of magnitude. We also expand that pulse sequence design strategy to target systems with nearby quadrupolar sinks and systems where the dominant coupling is between the hydrides rather than the target nucleus, as in many interesting alpha-keto acid targets.

TOG 04:45-05:05

First Hyperpolarized [1-13C]alpha-ketoglutarate MR Spectroscopy of the Human Brain

Presenter: Yaewon Kim (University of California, San Francisco)

All Authors: Yaewon Kim (University of California, San Francisco); Dang Duy (University of California, San Francisco); James Slater (University of California, San Francisco); Andrew Riselli (University of California, San Francisco); Jeremy W. Gordon (University of California, San Francisco); Susan M. Chang (University of California, San Francisco); Yan Li (University of California, San Francisco); Adam W. Autry (University of California, San Francisco); Marisa Lafontaine (University of California, San Francisco); Evelyn Escobar (University of California, San Francisco); Hsin-Yu Chen (University of California, San Francisco); Chou T. Tan (MilliporeSigma, Merck KGaA); Chris Suszczynski (MilliporeSigma, Merck KGaA); Robert A. Bok (University of California, San Francisco); Daniel B. Vigneron (University of California, San Francisco)

Isocitrate dehydrogenase (IDH) mutational status is crucial for accurate diagnosis and prognosis of gliomas. However, the current clinical assessment of IDH mutation requires an invasive brain biopsy for pathological testing. MR molecular imaging with hyperpolarized [1-13C]alpha-ketoglutarate can provide novel measurements of aKG metabolism and investigate glioma IDH mutational status by detecting glutamate or 2-hydroxyglutarate.We aimed to perform first in-human studies using hyperpolarized [1-13C]aKG as a new probe of IDH mutational status via cancer metabolic reprogramming, along with cerebral bioenergetics. We acquired 13C-MRS data from healthy brain volunteers (N=6) and glioma patients (N=6) who received hyperpolarized aKG. Feasibility and safety were demonstrated in these 12 studies, with signals observed from [1-13C]aKG and its metabolite glutamate in the obtained 13C-MRS data.

TOG 05:05-05:25

Solution-State NMR signal enhancement of small molecules via intermolecular cross relaxation from PHIP hyperpolarized source molecules

Presenter: Ilai Schwartz (NVision Imaging Technologies GmbH)

All Authors: Bogdan Rodin (NVision Imaging Technologies GmbH); Zumrud Ahmadova (NVision Imaging Technologies GmbH); John W. Blanchard (NVision Imaging Technologies GmbH); Laurynas Dagys (NVision Imaging Technologies GmbH); Federico De Biasi (École Polytechnique Fédérale de Lausanne); Tim R. Eichhorn (NVision Imaging Technologies GmbH); Lyndon Emsley (École Polytechnique Fédérale de Lausanne); Felix Josten (NVision Imaging Technologies GmbH); Stephan Knecht (NVision Imaging Technologies GmbH); Martin Korzeczek (Institute of Theoretical Physics & IQST, Ulm University); Salvatore Mamone (NVision Imaging Technologies GmbH); Pinelopi Moutzouri (École Polytechnique Fédérale de Lausanne); Anna J. Parker (NVision Imaging Technologies GmbH); Martin B. Plenio (Institute of Theoretical Physics & IQST, Ulm University); Usman Qureshi (NVision Imaging Technologies GmbH); Jochen Scheuer (NVision Imaging Technologies GmbH); Ran Wei (École Polytechnique Fédérale de Lausanne); Ilai Schwartz (NVision Imaging Technologies GmbH)

Nuclear spin hyperpolarization addresses NMR spectroscopy sensitivity limits. Here, we present a comprehensive parahydrogen-induced polarization protocol to amplify signals of small molecules in high-resolution NMR via intermolecular cross-relaxation.

A concentrated (1-13C,d6)-dimethyl maleate solution is produced by hydrogenation and the 1H nuclear singlet spin order is transferred into magnetization by tailored pulse sequences at low-field. We demonstrate that over 30-fold enhancements at 9.4 T can be obtained in mixtures, and over two orders of magnitude in benchtop 1.9 T spectrometers. High-quality NMR spectra are recorded using a tailored sequence that suppresses the otherwise overwhelming source signal.

Our findings extend hyperpolarized solution-state NMR, opening the path towards scalable and accessible applications such as high-throughput ligand screening, metabolite mixture analysis, and reaction monitoring.

TOG 05:25-05:45

Live Magnetic Observation of Parahydrogen Hyperpolarization Dynamics

Presenter: Michael Tayler (ICFO - The Institute of Photonic Sciences)

All Authors: James Eills (IBEC - Bioengineering Institute of Catalonia); Morgan W. Mitchell (ICFO - The Institute of Photonic Sciences); Irene Marco Rius (IBEC - Bioengineering Institute of Catalonia); Michael Tayler (ICFO - The Institute of Photonic Sciences)

Parahydrogen-induced hyperpolarization is a key route to synthesizing extreme 1H and 13C spin ordering. Although widely used in the production of MRI spin-tracers or polarized physics targets, the dynamics of these processes is not studied `live', usually due to the extremely low (Hz) frequencies, or detector saturation by the driving fields involved.

Here we demonstrate a simple but capable remedy: a near-DC atomic magnetometer. When placed next to a parahydrogen-enriched liquid, one obtains a high-resolution and real-time view of the magnetization dynamics, leakage mechanisms and dipolar shifts from genesis through to decay. We study the following examples: (1) magnetization build-up during adiabatic uT-field singlet-to-magnetization conversion sweeps in [1-13C]-fumaric acid; (2) SABRE-SHEATH hyperpolarization of [1-13C]-pyruvate.

4:00 PM - 5:50 PM, TUESDAY
TOH: Metabolomics I

David Rovnyak (Bucknell University) presiding
Chapel

TOH 04:00-04:25

New Approaches for Studying Known and Unknown Complex Metabolomics Mixtures by NMR

Presenter: Rafael Bruschweiler (The Ohio State University)

All Authors: Dawei Li (The Ohio State University); Nicholas Rigel (The Ohio State University); Rodrigo Cabrera Allpas (The Ohio State University); Munki Choo (The Ohio State University); Claire Gething (The Ohio State University); Alexandar Hansen (The Ohio State University); Lei Bruschweiler-Li (The Ohio State University); Rafael Bruschweiler (The Ohio State University)

The characterization of complex chemical mixtures by NMR encountered in metabolomics poses a wide range of opportunities and challenges in terms of metabolite coverage, identification accuracy, reproducibility, quantitation, speed, and automation. I will discuss new approaches toward these goals that include NOAH supersequences, machine-learning methods for automated spectral processing and analysis, new metabolite data bases, a new chemical shift predictor for resonance assignment and the characterization of unknown metabolites. These methods will be demonstrated for periprosthetic joint infection studies caused by opportunistic pathogens such as Pseudomonas aeruginosa and Staphylococcus aureus that tend to form biofilms with high antibiotic resistance.

TOH 04:25-04:45

NMR Metabolomics Best Practices: What We Are Doing, What We Should Be Doing, and Some Recent Advancements

Presenter: Robert Powers (University of Nebraska-Lincoln)

All Authors: Robert Powers (University of Nebraska-Lincoln)

NMR-based metabolomics has benefited a variety of fields from agriculture and environmental studies to drug discovery and disease diagnosis. Despite appearances, metabolomics is technically very challenging and requires expertise in a diversity of areas that range from analytical chemistry and cell biology to advanced statistical analysis and deep learning techniques. Accordingly, several challenges and problems exist that need to be overcome for the field to continue to succeed, which may be addressed by the adoption of a standard set of best practices by the community. This presentation will highlight common issues with sample preparation and handling, data collection and analysis, and statistical modeling and interpretation. Recent advancements in NMR-based metabolomics techniques and recommendations for best practices will also be presented.

TOH 04:45-05:05

Unraveling Human CD8+ T-Cell Metabolism: A 13C NMR Perspective on Targeting Glycolysis for Autoimmune Disease Intervention

Presenter: Gaurav Sharma (UNIVERSITY OF FLORIDA)

All Authors: GAURAV SHARMA (UNIVERSITY OF FLORIDA); Ram Khattri (University of Florida); Mario C. Chang (University of Florida); Scott E. stimpson (University of Florida); Jing Chen (University of Florida); Brittney N. Newby (University of Florida); Harold D. Chapman (The Jackson Laboratory); Thomas E. Angelin (University of Florida); David V. Serreze (The Jackson Laboratory); Clayton E. Mathews (University of Florida); Matthew Merritt (University of Florida)

Metabolic Flux Analysis (MFA) has emerged as a pivotal tool for understanding energy utilization in biological systems. Over the past decade, the integration of stable isotopes, particularly 13C, has elevated MFA to unprecedented levels, enabling real-time measurements of metabolic reactions in systems biology.

This study harnesses 13C-based MFA to explore human CD8+ T-cell metabolism, unveiling critical details of macronutrient utilization. Inhibiting glycolysis or mitochondrial respiration during CD8+ T-cell activation impedes proliferation, with post-activation shifts revealing glycolysis as crucial for cytotoxic T-lymphocyte activity. In Type 1 diabetes models, glycolysis inhibition delays T1D onset, preserving β-cell mass. These findings underscore glycolysis and mitochondrial ATP production's vital roles in T-cell activation, proposing glycolysis as a promising target for preventing T-cell-mediated autoimmunity.

TOH 05:05-05:25

Impact of Exposure to Environmental Pollutants on Placental Metabolism

Presenter: Lindsay Cahill (Memorial University of Newfoundland)

All Authors: Haley Adams (Memorial University of Newfoundland); Jenna Hanrahan (Memorial University of Newfoundland); Sophie Kiefte (Memorial University of Newfoundland); Thomas O'Brien (Memorial University of Newfoundland); Grace Mercer (Memorial University of Newfoundland); Celine Schneider (Memorial University of Newfoundland); Karl Jobst (Memorial University of Newfoundland); Lindsay Cahill (Memorial University of Newfoundland)

For a healthy pregnancy, the placenta needs to meet the metabolic demands of the fetus. This study evaluates the effects of exposure to persistent organic pollutants, legacy and novel per- and polyfluoroalkyl substances (PFAS), on placental metabolism.

Placental samples were collected from pregnant CD-1 mice exposed to perfluorooctanoic acid and fluorotelomer ethoxylates throughout gestation. Metabolite profiles were determined using 1H magic angle NMR on a Bruker 600 MHz spectrometer with a 3.2 mm MAS solid-state NMR probe.

The relative concentration of several metabolites that are essential nutrients for fetal development were found to be significantly altered in the PFAS-exposed groups. This study adds to the growing literature that has demonstrated the significant impact of environmental pollutants on placental function.

TOH 05:25-05:50

Ultra-High Resolution NMR: a Robust Method for Determining Quantitative Metabolic Profiles in Biofluids

Presenter: Nicolas Giraud (Université Paris Cité)

All Authors: Nicolas Giraud (Université Paris Cité)

We will present methodological developments targeting the acquisition of Pure Shift 1H NMR spectra with efficient suppression of the water signal. We will present for the first time a workflow for quantifying metabolites in various biofluids based on Pure Shift data. We will show how this approach can be used to detect in an hour all the metabolites at concentration as low as 10 µM in extra-cellular media, yielding a very good separation of their NMR signals. We will describe how the statistical analysis of the metabolic profiles determined from these ultrahigh resolution data allows for getting a unique insight into the metabolic pathways that are key to Lymphoma cells and understand the mechanism of action of antimetabolic drugs.

8:45 AM - 10:15 AM, WEDNESDAY
WOA: Theory and Computation for Structural and Dynamic Aspects
Len Mueller (University of California, Riverside) presiding
Merrill Hall

WOA 08:45-09:10
Transformative Acceleration of Putative Structure Generation
Presenter: Chris Pickard (University of Cambridge)
All Authors: Chris Pickard (University of Cambridge)
The Gauge Including Projector Augmented wave (GIPAW) theory for the computation of NMR properties provided a link between putative atomistic structures of extended systems and experimentally measured quantities. First principles structure prediction, for example Ab initio Random Structure Searching (AIRSS) can be a source of these structures. I will describe machine learning, or data derived, interatomic potentials that introduce a transformative acceleration of structure generation, by up to five orders of magnitude. Ephemeral Data Derived Potentials (EDDPs) were designed for structure search and can be used straightforwardly within an AIRSS computational workflow. As an alternative to generating many structures, the computational acceleration can be exploited to introduce temperature and disorder, through Hot AIRSS searches.

WOA 09:10-09:30
Neural Net Analysis of Strongly Coupled Spin Systems
Presenter: James Prestegard (University of Georgia)
All Authors: Jim Prestegard (University of Georgia); John N. Glushka (University of Georgia); John H. Grimes Jr. (University of Georgia); Bernd Simon (University of Connecticut Health)
Scalar couplings between proton pairs can provide useful structural information on both small and large molecules. However, when differences in chemical shifts of coupled spins in Hz become similar to coupling constants, strongly coupled spectra result and extraction of both chemical shifts and scalar couplings becomes difficult. Here we illustrate an approach based on application of a trained neural net to analysis of 2D J-resolved spectra. The network was designed using tools available in MATLAB and was trained on hundreds of thousands of spectra simulated with SPINACH software. While simulation and training required many hours on high-end computational resources, the trained nets are easily accommodated on a laptop computer and analyses are returned in seconds.

WOA 09:30-09:50
First Principles Calculations of Molecular Properties in Electrolytes from Spin and Molecular Dynamics Simulations Coupled with Experimental Relaxation Rates
Presenter: Florin Teleanu (New York University)
All Authors: Florin Teleanu (New York University); Alexej Jerschow (New York University)
Sub-picoseconds water dynamics have previously been evaluated around solvated metal ions where electric field fluctuations due to solvent collisions impact the observable relaxation rates via the quadrupolar interaction. Here, we explore more complex heteronuclear multi-spin systems characteristic to battery electrolytes that are also sensitive to surrounding water motions and develop a methodology based on spin and molecular dynamics analyses to experimentally derive absolute values of the electric field gradients.

WOA 09:50-10:15
Development of QM/DFT Computational Tools to Accelerate NMR based Molecular Structure Elucidation and Solid Form Identification
Presenter: Nina Gonnella (Boehringer Ingelheim Inc.)
All Authors: Nina Gonnella (Boehringer Ingelheim Inc.)
Chemical and crystalline structure can affect physical and chemical properties of drugs, hence, determining both chemical structure and crystalline forms is essential in the pharmaceutical industry. NMR is an excellent technology for structure determination; however, experimental approaches sometimes yield inconclusive results. To accelerate structure elucidation for both solution and solid-state NMR (ssNMR), we developed two programs HiPAS and OPTICS, respectively. HiPAS uses molecular modeling, and QM/DFT calculations with solution NMR data to improve speed and accuracy in structure elucidation. OPTICS uses QM/DFT, and Bayesian probability theory combined with 13C ssNMR data as a powerful means of polymorph identification. The presentation will describe these prediction tools and associated applications for rapid chemical structure elucidation and identification of the correct solid form.

8:45 AM - 10:15 AM, WEDNESDAY
WOB: Materials II
Robert W. Schurko (Florida State University) presiding
Chapel

WOB 08:45-09:10
Structure and Dynamics of Photochromic Rare-Earth Oxyhydrides
Presenter: Arno Kentgens (MRRC, Radboud University)
All Authors: Shrestha Banerjee (MRRC, Radboud University); Angel Y.T. Wong (MRRC, Radboud University); Bernard Dam (Department of Chemical Engineering, Delft University of Technology); Gilles A. de Wijs (Theoretical Chemistry, Radboud University); Arno Kentgens (MRRC, Radboud University)
Rare-earth oxyhydrides, with formula REOxH(3−2x), are mixed-anion compounds showing reversible darkening under UV irradiation and have a potential application as smart windows. The detailed structure and dynamics of the anions, which are key to the underlying mechanism of the photochromism, are not well understood. We use 1H, 2H, 89Y and 45Sc and 17O solid-state NMR spectroscopy and DFT calculations to unravel different local environments in YHO and ScHO films before and after illumination. The limited sample amount (1 um films) is challenging for NMR, but nonetheless the lattice structure and mobility of hydrogen species are investigated in detail. In view of sensitivity challenges, we revisit optimal acquisition methods for half-integer quadrupolar methods including (repetitive) DFS, QCPMG and steady state approaches.

WOB 09:10-09:30
Exploiting In Situ Solid-State NMR Spectroscopy to Follow Non-Traditional Zeolite Formation
Presenter: Emma A. L. Borthwick (University of St Andrews)
All Authors: Emma Borthwick (University of St Andrews); Nicole L. Kelly (University of St Andrews); Gaynor B. Lawrence (University of St Andrews); Paul S. Wheatley (University of St Andrews); Colan E. Hughes (Cardiff University); Kenneth D. M. Harris (Cardiff University); Russell E. Morris (University of St Andrews); Sharon Ashbrook (University of St Andrews)
Novel non-traditional methods for zeolite synthesis have been explored but there is little understanding of the mechanism of formation for these new materials. In this work we are following two separate zeolite conversions using in situ solid-state NMR spectroscopy, using 29Si enriched starting materials and reagents monitoring their progress using repeating single pulse NMR experiments that allows us to probe the liquid-state and solid-state components of the system simultaneously, as their 29Si NMR signals appear in different regions of the spectra. The mechanism of formation is significantly different to that seen in the crystallization of zeolites and provides evidence that such non-traditional methods can lead to new zeolitic materials that would likely not form using traditional synthesis.

WOB 09:30-09:50
Massive CQ's and Fast Cation Dynamics: 23Na, 25Mg and 11B NMR Studies of "Paddlewheel" Antiperovskite Solid Electrolytes
Presenter: David Halat (UC Berkeley & LBNL)
All Authors: David Halat (UC Berkeley & LBNL); Haoyu Liu (Argonne National Laboratory); Kwangnam Kim (University of Michigan); Xiaoling Wang (CSU East Bay); Grant C. B. Alexander (University of Illinois at Chicago); Harris Mason (Los Alamos National Laboratory); Sunil Mair (Massachusetts Institute of Technology); Ping Chun Tsai (National Taiwan University of Science and Technology); Duhan Zhang (Massachusetts Institute of Technology); Bob Jin Kwon (Argonne National Laboratory); Alex Chien (Oak Ridge National

Laboratory); Jeffrey Smith (University of Michigan); Yiliang Li (Massachusetts Institute of Technology); Liang Yin (Argonne National Laboratory); Jue Liu (Oak Ridge National Laboratory); Saul Lapidus (Argonne National Laboratory); Baris Key (Argonne National Laboratory); Donald Siegel (University of Michigan); Jordi Cabana (University of Illinois at Chicago); Yet-Ming Chiang (Massachusetts Institute of Technology); Jeffrey Reimer (UC Berkeley)

Antiperovskite solid-state fast-ion conductors are important candidates in Li-ion and beyond Li-ion (e.g., Na-ion, Mg-ion) batteries with a wide chemical tunability range. From the NMR spectroscopist's viewpoint, antiperovskite systems are of particular interest because quadrupolar coupling constants (CQ's) of the X-site cation are exceptionally large. Temperature-induced changes in quadrupolar powder patterns are therefore sensitive indicators of cation motion. We report 23Na variable-temperature NMR measurements of the "double-paddlewheel" antiperovskite Na2NH2BH4, as well as 25Mg QCPMG spectra of the recently synthesized antiperovskites Mg3AsN and Mg3SbN, which possess 25Mg CQ's of up to 22 MHz, the largest such values to our knowledge. High-field 25Mg spectra acquired at 35.2 T at the MagLab further reveal how quadrupolar-broadened powder patterns can report on antiperovskite synthesizability.

WOB 09:50-10:15

Probing Structural Subtleties in Anti-Perovskite Solid Electrolytes

Presenter: Karen Johnston (Durham University)

All Authors: Karen Johnston (Durham University); George E. Rudman (Durham University); Tavleen S. Attari (Durham University); Theodosios Famprikis (Delft University of Technology); James A. Dawson (Newcastle University); Emma McCabe (Durham University); Phoebe K. Allan (University of Birmingham)

All-solid-state lithium-ion (Li-ion) batteries are attracting considerable attention as possible alternatives to conventional liquid electrolyte-based devices, as they present a viable opportunity for increased energy density and safety. In recent years, numerous materials have been explored as possible solid electrolytes, including Li-rich anti-perovskites (LiRAPs) such as Li3−xOHxCl, which have generated considerable interest based on their reported ionic conductivities. However, their lithium and proton transport capabilities as a function of composition are not fully understood. Hence, research efforts have focused on the synthesis and structural characterisation of Li3−xOHxCl using a combination of high-resolution powder diffraction, variable-temperature multinuclear solid-state NMR spectroscopy and ab initio molecular dynamics. We will demonstrate that Li-ion transport is highly correlated with both proton and Li-ion vacancy concentrations.

8:45 AM - 10:15 AM, WEDNESDAY
WOC: Innovations in MRI Methods and Instruments

Matthew Rosen (Mass General / Martinos Ctr for Biomedical Imaging, Harvard Medical School) presiding

Woodlands

WOC 08:45-09:10

20-year odyssey of the 11.7T human MRI project

Presenter: Nicolas Boulant (University of Paris-Saclay, CEA, NeuroSpin)

All Authors: Nicolas Boulant (University of Paris-Saclay, CEA, NeuroSpin)

The promises of MRI at ultra-high field in gains of increased signal-to-noise and contrast-to-noise ratios have fueled a race towards higher magnetic fields. With its 90 cm wide bore and 11.7T field strength, the Iseult whole-body magnet currently holds the world record in magnetic energy stored in a MRI scanner. This talk will summarize the 20-year project odyssey to obtain the first in vitro images in 2021 and its culmination with in vivo images in 2023. The data shows promises towards opening a new and exciting window of observation on the human brain.

WOC 09:10-09:30

Dynamic Noise Cancellation for Unshielded, Single-Sided, Fourier and Spatiotemporally Encoded Low-Field MRI

Presenter: Kartiga Selvaganesan (Promaxo Inc.)

All Authors: Kartiga Selvaganesan (Promaxo Inc.); Meredith Sadinski (Promaxo Inc.); Riwei Jin (Promaxo Inc.); Muller Gomes (Promaxo); Scott B. King (Promaxo Inc.); William A. Grissom (Case Western Reserve University); Aleksander Nacev (Promaxo Inc.)

In this study, we have developed a dynamic active noise cancellation technique that retrospectively removes noise components from MR signal prior to image reconstruction. The method uses external noise detector probes to measure electromagnetic interference (EMI), and singular value decomposition to automatically detect and suppress the strongest noise sources present in each TR. The effectiveness of the method was demonstrated for introduced EMI at specific frequencies, different pulse sequences, linear and nonlinear encoding trajectories. The spectrum and imaging results showed significant reduction in EMI without eliminating the desired proton signal. Overall, the method provides SNR robustness and improved image quality in unshielded environments thereby making MRI portable, and accessible to provide point-of-care screening.

WOC 09:30-09:50

Multiband Spatiotemporal Encoding with minimized slab boundary artifacts

Presenter: Jaeyong Yu (Sungkyunkwan University)

All Authors: Jaeyong Yu (Sungkyunkwan University); Jang-Yeon Park (Sungkyunkwan University)

Functional MRI utilizes EPI for faster acquisition while maintaining spatial resolution, but EPI is susceptible to distortion and signal loss from B0 inhomogeneity. To address this, spatiotemporal encoding (SPEN) with multiband (MB) acquisition was proposed for higher SNR efficiency, where concerns arise about crosstalk artifacts at slab boundaries. Here we proposed an approach to minimize crosstalk in MB SPEN using a WURST pulse. Suitable parameters were determined via Bloch simulation, and an optimized WURST pulse was applied in the ERASE (Equal-TE Rapid Acquisition with Sequential Excitation) sequence. Additionally, multi-shot acquisition further minimized crosstalk artifacts. Results demonstrated that multi-shot MB ERASE using an optimized WURST pulse could provide enhanced image quality with little crosstalk artifacts at slab boundaries.

WOC 09:50-10:15

Dipolar Order and Disorder in Model Systems and In Vivo

Presenter: Scott Swanson (University of Michigan)

All Authors: Scott Swanson (University of Michigan)

Magnetization Transfer (MT) between water and immobilized components in thermodynamically heterogeneous materials such as tissues and liquid crystals allows interrogation of semisolids through the water proton resonance. The molecular arrangement and dynamics of lipids and sterols in myelin, the primary component of white matter in the central nervous system, give rise to a relatively long-lived spin dipolar order. Methods to exploit dipolar order and selectively image myelin in vivo have been called inhomogeneous magnetization transfer (ihMT). We have developed a series of biomimetic materials with controllable dipolar order, MT, and ihMT parameters. This talk will outline the mechanisms of MT and ihMT in model systems to help understand the structure and dynamics of myelin in vivo.

10:45 AM - 12:35 PM, WEDNESDAY
WOD: Biomolecular Solids-Materials

Sharon Ashbrook (University at Andrews) presiding

Merrill Hall

WOD 10:45-11:10

A close look at the surface of nanocelluloses with DNP-enhanced solid-state NMR

Presenter: Sabine Hediger (Univ. Grenoble Alpes / CEA / CNRS)

All Authors: Sabine Hediger (Univ. Grenoble Alpes / CEA / CNRS)

Cellulose nanofibrils (CNFs) offer a promising combination of renewable, biodegradable, and biocompatible qualities, coupled with high specific surface area and tunable surface chemistry. These

features make them excellent candidates for numerous applications, including drug delivery systems. To improve our understanding and maximize their potential, it is imperative to study the surface chemistry of CNFs in depth. Recent advances in high-field dynamic nuclear polarization (DNP), together with the development of improved polarizing agents, have revolutionized our ability to obtain atomic-level insights on CNF surface modifications, even at low grafting levels. For example, DNP-enhanced spectra can distinguish between adsorption and covalent grafting, or track modifications to grafted compounds throughout the synthesis and washing stages. Such information is essential for materials optimization.

WOD 11:10-11:30

Hydration water dynamics in globular villin headpiece and amyloid fibrils, a joint H-2 and O-17 NMR study

Presenter: Liliya Vugmeyster (CU Denver)

All Authors: Dmitry Ostrovsky (Department of Mathematics, CU Denver); Riqiang Fu (National High Magnetic Field Lab)

We investigated bound water in hydrated powders of globular villin headpiece protein and amyloid-beta aggregates. We combine line shapes and relaxation studies at H-2 and O-17 nuclei in the 300 to 180 K temperature range to characterize hydration layers' dynamics. We can parametrize large and small-angle fluctuations in hydration layers, differentiated by how tightly water molecules are bound to the protein interfaces. Examples of resulting parameters include diffusion coefficients and fractions of the loosely bound layer, rate constants for tetrahedral jumps and 2-site deuteron flips of water molecules bound to the interfaces, as well as small-angle motions. We can also observe the dynamical transition and determine the rate constant for the translational motion between water hydration layers.

WOD 11:30-11:50

Proton Relaxation Dispersion at fast MAS, a Tool with Important Prospects

Presenter: Rasmus Linser (TU Dortmund University)

All Authors: Rasmus Linser (TU Dortmund University); Suresh Kumar Vasa (TU University Dortmund)

Assessment of protein dynamics has largely relied on heteronuclear labels, which are not always available. Proton relaxation, by contrast, would be a splendid tool for assessing dynamics in a wide range of cases, but in particular in the solid state, it has been associated with several inherent difficulties. Here we show that under fast MAS, background effects can be separated from the information of interest, which demonstrates the prospects of the technique for a plethora of biological applications.

WOD 11:50-12:10

Novel Dipolar Recoupling Elements

Presenter: Evgeny Nimerovsky (MPI-NAT)

All Authors: Evgeny Nimerovsky (MPI-NAT); Eszter Éva Najbauer (MPI-NAT); Abel Cherian Varkey (MPI-NAT); Kumar Tekwani Movellan (MPI-NAT); Kai Xue (MPI-NAT); Myeongkyu Kim (MPI-NAT); Stefan Becker (MPI-NAT); Loren B. Andreas (MPI-NAT)

Dipolar recoupling elements serve as fundamental components in routine magic-angle spinning NMR experiments for determination of resonance assignments, dynamic processes, structure, and probing the chemical state of samples, including microcrystalline and non-crystalline preparations like membrane proteins in lipid bilayers. Here we introduce four novel dipolar recoupling elements designed for selective and not selective transfers between homonuclear and heteronuclear spin pairs. These elements include MODIST (band-selective proton-proton) and GODIST (band-selective carbon-carbon), alongside two elements grounded in the simplified preservation of equivalent paths spectroscopy (SPEPS). Homonuclear and heteronuclear SPEPS elements facilitate the simultaneous transfer of both spin pairs' transverse components. This concurrent transfer allows for a potential improvement of up to $\sqrt{2}$ in sensitivity for each dimension within a multidimensional spectrum.

WOD 12:10-12:35

Optimal Control Methods for Multidimensional Solid-State NMR of Proteins

Presenter: Zdenek Tosner (Charles university)

All Authors: Jan Blahut (Institute of Organic Chemistry and Biochemistry, Czech Academy of Science); Bernd Reif (TU Muenchen); Zdeněk Tošner (Charles university)

Solid-state NMR investigations of proteins benefit from the recent technology of high magnetic fields and ultrafast MAS. Proton-detected multidimensional methods are used to resolve spectral overlap challenges and yield connectivity between atoms. However, sensitivity remains the major obstacle to broader applicability because of the integrated loss of sensitivity through multiple magnetization transfers.

We present heteronuclear and homonuclear dipolar recoupling elements using TRansverse mixing based on Optimal-control Pulses (TROP). This concept explores the preservation of equivalent pathways principle known from solution-state NMR. Transferring both x- and y- magnetization components after indirect chemical shift evolution leads to a 1.41-fold increase in sensitivity per indirect dimension. This provides an order of magnitude time savings in just emerging 4-5D proton-detected experiments.

10:45 AM - 12:35 PM, WEDNESDAY
WOE: Eclectica
Carl Michal (University of British Columbia) presiding
Chapel

WOE 10:45-11:10

Ultra-sensitivity with radiation-detected NMR using unstable nuclei

Presenter: Magdalena Kowalska (CERN/UNIGE)

All Authors: Magdalena Kowalska (CERN/UNIGE)

Radiation-detected (RD) NMR allows recording resonances on a million nuclei. This ultra-high sensitivity is due to combination of hyperpolarization with resonance detection via radiation emitted by unstable probe nuclei.

Using our setup at CERN, which combines optical pumping with beta-decay detection, we have established a new way of referencing RD-NMR spectra, connecting them to conventional NMR and to quantum chemistry calculations. We have also used it to study the structure of unstable nuclei and looked at the binding sites of Na and K in ionic liquids.

In parallel, with our partners, we explore using long-lived Xe isotopes for MRI imaging and look into hyperpolarizing nuclear medicine isotopes, e.g 13N and 11C, using combined RD-ZULF-NMR.

WOE 11:10-11:30

Towards Ultra-high-quality-factor Wearable RASER MR Sensing Using Parametric Pumping

Presenter: Eduard Chekmenev (Wayne State University)

All Authors: Isaiah Adelabu (Wayne State University); Shiraz Nantogma (Wayne State University); Mustapha Abdulmojeed (North Carolina State University); Henri De Maissin (University of Freiburg); Andreas Schmidt (University of Freiburg); Stephan Appelt (RWTH Aachen & FZ-Juelich); Sören Lehmkuhl (KIT); Matt Rosen (MGH/Martinos Center); Chunqi Qian (Michigan State University); Thomas Theis (North Carolina State University); Eduard Chekmenev (Wayne State University)

It has been recently shown that a bolus of hyperpolarized nuclear spins can yield stimulated emission similar in nature to that of maser and laser. The stimulated NMR signal emissions lasting for minutes don't require radio-frequency excitation, offering unprecedented advantages compared to conventional MR sensing. However, creating nuclear stimulated emission is challenging in practice due to stringent fundamental requirements. We demonstrate the utility of a wireless NMR maser detector at 300 MHz, the quality factor (Q) of which can be enhanced by 20-fold (to up to nearly 1,700) via parametric pumping. Greater gains to Q of 1,000,000 are potentially feasible, paving the way to new NMR applications, including in vivo RF Amplification by Stimulated Emission of Radiation (RASER) MRI.

WOE 11:30-11:50

Moving MRI (mMRI): Imaging a Moving Body with Synchronized Magnet Movement

Presenter: Jingting Yao (Massachusetts General Hospital/Harvard Medical School)

All Authors: Jingting Yao (Massachusetts General Hospital/Harvard Medical School); Artan Kaso (Massachusetts General Hospital/Harvard Medical School); Nikhil Patel (Massachusetts General Hospital); Yin-Ching Iris Chen (Massachusetts General Hospital/Harvard Medical School); Yi-Fen Yen (Massachusetts General Hospital/Harvard Medical School); André J.W. van der Kouwe (Massachusetts General Hospital/Harvard Medical School); Daniel M. Merfeld (The Ohio State University College of Medicine); Jerome Ackerman (Massachusetts General Hospital/Havard Medical School)

MRI is largely limited to scenarios involving small-scale bodily movements to minimize artifacts and field-induced physiological effects. We are developing a moving MRI system where the magnet and subject's head remain stationary with respect to each other during large-scale motion. Utilizing a compact, cryogen-free 1.5 T magnet, we built an apparatus that tilted the entire magnet assembly, including the cold head, gradient/shim/RF coils, and the subject, up and down during scanning. We demonstrated the ability to scan phantoms and live animals while the magnet is in motion and to correct for imaging artifacts caused by tilting the magnet. The outcome of this work may advance studies in vestibular research, traumatic brain injury, and brain-behavior interactions, among other areas.

WOE 11:50-12:10

Deconstructing Porous Media: Magnetic Resonance Insights into the Heterogeneous, Fractal-like Kinetics of Chemically Upcycled Polymers

Presenter: Sophia Fricke (University of California, Berkeley)

All Authors: Sophia Fricke (University of California, Berkeley); Shira Haber (Lawrence Berkeley National Laboratory); Mia Salgado (University of California, Berkeley); Mutian Hua (Lawrence Berkeley National Laboratory); Brett A. Helms (Lawrence Berkeley National Laboratory); Jeffrey A. Reimer (University of California, Berkeley)

Chemical upcycling of polymers is a critical component of a lossless, waste-free plastics economy. However, tracking polymer deconstruction through hydrolysis is fraught with the difficulty of disentangling concurrent swelling, diffusion, and time-dependent reaction kinetics. It is necessary to develop strategies to track this process in situ and adequately treat its nonlinearity. Here, time-resolved magnetic resonance imaging, relaxometry, and diffusometry are used to observe the kinetics of poly(diketoenamine) hydrolysis. Quantification of polymer cross-sectional area over time yields rate law information about the diffusion-mediated deconstruction process. Fractal-based mathematics succinctly captures the time-dependence of both the rate coefficient and the reaction order, providing streamlined methods to investigate polymer hydrolysis kinetics that will ultimately assist in implementing chemical upcycling efforts on the industrial scale.

WOE 12:10-12:35

Probing singlet states in frustrated magnets using muons as a magnetic resonance probe

Presenter: Stephen Blundell (University of Oxford)

All Authors: Stephen Blundell (University of Oxford)

The interaction between an implanted muon and neighbouring spins can be used to probe entanglement and decoherence, as well as characterise the muon site in many different materials with high accuracy. Muons can be used to study magnetically frustrated systems that exhibit persistent spin dynamics so that as the temperature is lowered, the muon-spin relaxation rate rises (as would be expected for the slowing-down of spin fluctuations) but this rate then saturates at low temperature. To explain this phenomenon, I will describe how muons can couple to singlet states and how this can be extended to understand the way muons couple to a variety of systems exhibiting highly frustrated magnetism.

10:45 AM - 12:30 PM, WEDNESDAY
WOF: Small Molecules II

Yusuke Nishiyama (Riken-JEOL, JEOL Resonance) presiding
Woodlands

WOF 10:45-11:10

Protein dynamics from relaxation and paramagnetic NMR

Presenter: Christian Griesinger (MPINAT)

All Authors: Christian Griesinger (MPINAT); Supriya Pratihar (Columbia University); Dwaipayan Mukhopadhyay (Columbia University); Felipe Alvarado (Columbia University); Niels Karschin (Bruker Biospin)

Relaxation dispersion allows to measure rates of interconversion of conformational states of proteins. Since the fastest rates accessible by relaxation dispersion are limited by the radiofrequency power one digit microsecond rates or slightly faster are accessible. Yet, motion in the range between the correlation time and the fastest rates from relaxation dispersion are difficult to detect. New methodology will address this question. In addition, domain motion of proteins can be studied with paramagnetic NMR spectroscopy where one domain is paramagnetically labelled while the other domain experiences the ordering of the labelled one due to alignment. The conversion of the respective data to an ensemble will be discussed. Finally optimal control pulses for 1.2 GHz NMR will be presented.

WOF 11:10-11:30

Free Ligand Solution Conformations Guided Design of Potent and Bioavailable Drug Candidates

Presenter: Amber Balazs (AstraZeneca)

All Authors: Amber Balazs (AstraZeneca); Nichola L. Davies (AstraZeneca); Jennifer Kingston (AstraZeneca); David Longmire (AstraZeneca); Martin J. Packer (AstraZeneca); Kevin J. Robbins (AstraZeneca); Markus Schade (AstraZeneca); James S. Scott (AstraZeneca); Bryony Smith (AstraZeneca); David Wilson (AstraZeneca); Elisabetta Chiarparin (Merck KGaA)

'Rule-of-five' (Ro5) are guidelines for designing passive permeability of drugs. These empirical thresholds triage design by relating molecular structure and physical chemistry properties. Multiple approvals of oral drugs violating Ro5 guidelines question Ro5 models. We demonstrated structural insights for optimal pharmacological activity dubbed 'SAR by 1D NMR'. We next explored physicochemical properties for oral drug candidates. Structure-Property Relationships (SPR) of ~ 100 clinical candidates and marketed drugs were explored, including full conformational and dynamical details for a subset of 15 oral drug Ro5 violators. We observed potent oral drugs preferentially preorganized into a bioactive conformation and highly populated conformations conducive to polarity masking. Our key learnings revisit guideline thresholds to predict the likelihood of oral exposure for beyond Ro5 drugs.

WOF 11:30-11:50

The Field and Temperature Dependence of 1H and 13C Relaxation Rates in Rapidly-Rotating Methyl Groups and the Effect of Spin Rotation

Presenter: Kelsey Marr (New York University)

All Authors: Kelsey Marr (New York University); Mohamed Sabba (University of Southampton); Harry Harbor-Collins (University of Southampton); Malcolm H. Levitt (University of Southampton); Alexej Jerschow (New York University)

Rapidly rotating methyl groups in liquids have attracted interest due to the potential to support exotic phenomena such as quantum rotor-induced polarization and long-lived nuclear spin order. Consequences of this rapid methyl rotation may be seen in nuclear magnetic resonance spectra and characterized by NMR relaxation studies. The aim of this work is the systematic measurement of both 1H and 13C nuclei relaxation rates of compounds such as 4-methylpyridine and a structural analogue, 2,5-dimethylanisole, as a function of magnetic field strengths and temperatures.These experiments and further computational work aim to produce a solid foundation for studies of methyl rotor dynamics work both experimentally and computationally.

WOF 11:50-12:10

Slice Through the Water – Exploring the Fundamental Challenge of Water Suppression for Benchtop NMR Systems

Presenter: Ronald Soong (University of Toronto Scarborough)

All Authors: Ronald Soong (University of Toronto Scarborough); Andre Simpson (University of Toronto); Katelyn Downey (University of Toronto); Rajshree Ghosh Biswas (University of Toronto); Flavio Kock (University of Toronto Scarborough); Katrina Steiner (University of Toronto); Myrna Simpson (Simpson); Benjamin Goreling (Bruker); Agnes Haber (Bruker); Colin Elliott (Bruker); Venita Busse (Bruker); Falko Busse (Bruker); Jacob Pellizzari (University of Toronto Scarborough)

Benchtop NMR provides improved accessibility in terms of cost, space, and technical expertise. In turn, this encourages new users into the field of NMR spectroscopy. Unfortunately, many interesting samples, from beer to whole blood, contain significant amounts of water that require suppression in 1H NMR in order to recover sample information. Therefore, simply translating solvent suppression experiments intended for high-field NMR instruments to benchtop NMR systems without careful consideration can be problematic. In this study, the effectiveness of several popular water suppression schemes was evaluated for benchtop NMR applications. Emphasis is placed on pulse sequences with no, or few, adjustable parameters making them easy to implement and further illustrate the potential of these approaches in education and research.

WOF 12:10-12:30

Parahydrogen Polarization of Biological Molecules in Homogeneous and Heterogeneous Media

Presenter: Christian Hilty (Texas A&M University)

All Authors: Pierce Pham (Texas A&M University); Oindrila Biswas (Texas A&M University); Ratnamala Mandal (Texas A&M University); Christian Hilty (Texas A&M University)

Nuclear spin hyperpolarization using nonhydrogenative parahydrogen polarization methods, including signal amplification by reversible exchange (SABRE), of biological molecules is demonstrated. Enhanced signals are used for the measurement of macromolecular interactions and molecular dynamics in two modalities. First, a purpose designed reporter ligand is hyperpolarized in an organic solvent. After dilution with a buffer, it is used to measure the interaction of ligands of interest with a target protein. The compatibility with high- or low-field NMR extends the range of dynamic processes that can be studied. Second, a nano-scale phase separated medium comprising reversed micelles solubilizing a polarization complex in the organic phase and protein in the aqueous phase provides a means to measure biological interactions.

4:00 PM - 5:50 PM, WEDNESDAY
WOG: Biomolecular Solutions III

Rasmus Linser (TU Dortmund University) presiding

Merrill Hall

WOG 04:00-04:25

Understanding Periplasmic Protein Quality Control at Atomic Level in Live Cells

Presenter: Alejandro Vila (IBR (CONICET, UNR))

All Authors: Lisandro J. Gonzalez (IBR (CONICET - UNR)); Letizia Pontoriero (CERM, University of Florence); Francisco J. Hita (IBR (CONICET - UNR)); Roberta Pierattelli (CERM, University of Florence); Andres Binolfi (IBR (CONICET-IBR)); Alejandro J. Vila (IBR (CONICET-UNR))

Metabolic processes in the bacterial periplasm are intimately connected to highly variable extracellular stimuli such as the concentration of essential cell nutrients and the presence of antibiotics targeting bacterial survival. Here we study the degradation of the New Delhi metallo-β-lactamase (NDM-1) in the bacterial periplasm of E. coli. The host native immune system response limits the availability of the Zn(II) ions at the infection sites, leading to accumulation of the non-metalated (apo) NDM-1 variant in the periplasm, that is degraded by the proteases Prc and DegP. We identified the cleavage sites of each protease and their concerted mechanism of action providing new insights about the molecular recognition events in living E. coli cells.

WOG 04:25-04:45

In-cell NMR: a Powerful Approach for Drug Discovery

Presenter: Lucia Banci (University of Florence)

All Authors: Lucia Banci (University of Florence)

In-cell NMR, i.e. high resolution NMR spectra of biomolecules in intact, living cells, represents one of the highest impact applications of magnetic resonance.

These experiments allow to obtain information on the conformational and functional properties of biomolecules at atomic resolution in conditions as close as possible to the physiological ones. In-cell NMR allows also to monitor protein-protein interactions and to follow functional processes. A striking application is drug screening in real time at cellular level, in human living cells.

WOG 04:45-05:05

Cell-free synthesis of trifluoromethionine labeled proteins for 19F NMR studies

Presenter: Wenkai Zhu (University of Pittsburgh)

All Authors: Wenkai Zhu (University of Pittsburgh); Christina Monnie (Department of Structural Biology and Pittsburgh Center for HIV Protein Interactions); Kristīne Kitoka (Laboratory of Physical Organic Chemistry, Latvian Institute of Organic Synthesis, Riga, Latvia); Angela Gronenborn (University of Pittsburgh School of Medicine)

In this study, we demonstrate that trifluoromethyl-methionine (tfmM) can be incorporated into Cyclophilin A with high efficiency by cell-free expression, circumventing the toxicity of tfmM encountered by biosynthetic incorporation using E. coli Met auxotrophs. tfmM CypA retains its binding affinity to the N-terminal domain of its native substrate HIV-1 capsid protein (HIV-1 NTD), as evidenced by Isothermal Titration Calorimetry (ITC) and 19F NMR titration experiments. Unambiguous assignments for five 19F peaks were obtained by 19F solvent paramagnetic relaxation enhancement measurements and measuring D2O solvent isotope-induced 19F chemical shifts, both of which provide the solvent accessibility of 19F probes in the protein. Therefore, tfmM modified proteins by cell-free expression provide novel ways for studying large proteins by 19F NMR spectroscopy.

WOG 05:05-05:25

NMR Observation of Membrane-Associated H-Ras in the Native Cellular Environment

Presenter: Takanori Kigawa (RIKEN Center for Biosystems Dynamics Research)

All Authors: Takanori Kigawa (RIKEN Center for Biosystems Dynamics Research)

Ras acts as a molecular switch to control intracellular signalling on the plasma membrane (PM). Elucidating how Ras associates with PM in the native cellular environment is crucial for understanding its control mechanism. Herein, we explored the structural features of membrane-associated H-Ras using in-cell NMR spectroscopy. To overcome the low sensitivity of the in-cell NMR spectra, we used the site-specific 19F-labeled H-Ras to simplify the NMR spectra and a Bayesian spectral deconvolution to ensure the objectivity of our interpretation. We demonstrated that the exogenous H-Ras was assimilated by endogenous membrane trafficking and adopted nucleotide-dependent multiple conformations relative to PM in the cell. Our study may be helpful in elucidating the atomic-scale picture of membrane-associated proteins in living cells.

WOG 05:25-05:50

19F NMR spectroscopy: a powerful approach for studying biological systems in vitro and in cells

Presenter: Conggang Li (Chinese Academy of Sciences)

All Authors: Conggang Li (Chinese Academy of Sciences)

19F NMR spectroscopy has become a valuable tool in the study of complex biological systems that cannot be easily analyzed using traditional spectroscopic techniques. Recent advancements in 19F labeling efficiency, sensitivity, and application have further enhanced its capabilities. In this presentation, we will discuss new probes containing 19F and their distinctive NMR spectroscopic features. We will also explore the various applications of these probes in biological

systems, such as aromatic ring flip-flop dynamics, protein dimerization in cells and in mitochondria, and the use of chemical shift fingerprinting with 19F NMR to detect the topology of human telomere overhang with native length, both in vitro and in cells.

4:00 PM - 5:45 PM, WEDNESDAY
WOH: Hyperpolarization II
Daniel Raftery (University of Washington) presiding
Chapel

WOH 04:00-04:25
High-Field Dynamic Nuclear Polarization from the Electron Spin Perspective
Presenter: Ilia Kaminker (Tel-Aviv University)
All Authors: Ilia Kaminker (Tel-Aviv University)
Dynamic Nuclear Polarization (DNP) is the most versatile hyperpolarization technique for solid-state NMR (ssNMR). In the core of the DNP lies the polarization transfer from the electron spins to nuclear spins. Because of the resolution requirements, the majority of ssNMR experiments are performed under magic angle spinning (MAS) and at high (> 7 T) magnetic fields. Despite extensive theoretical research of the electron-nuclear polarization transfer under these conditions, until recently, electron spins were never observed directly under the MAS-DNP conditions. We have recently demonstrated the first pulsed electron paramagnetic resonance experiments that allow for experimental observation of the electron spin dynamics at 7 T under MAS. In this presentation, I will discuss our recent results, methodology, and instrumentation for MAS-EPR.

WOH 04:25-04:45
First Example of Nitrogen-14 Hyperpolarization
Presenter: Roman Shchepin (South Dakota School of Mines & Technology)
All Authors: Oleg G. Salnikov (International Tomography Center SB RAS); Zachary T. Bender (South Dakota School of Mines & Technology); Ivan A. Trofimov (International Tomography Center SB RAS); Alexandra I. Trepakova (International Tomography Center SB RAS); Connor W. Ullrich (South Dakota School of Mines & Technology); Garrett L. Wibbels (South Dakota School of Mines & Technology); Roman Shchepin (South Dakota School of Mines & Technology); Igor V. Koptyug (International Tomography Center SB RAS); Danila Barskiy (Johannes Gutenberg University Mainz)
Here, we present first of its kind proof-of-principle study, which demonstrate feasibility of hyperpolarization for quadrupolar 14N nuclei in solution using PHIP technique. Unsaturated quaternary ammonium salts were chosen as substrates for pairwise parahydrogen addition and PH-INEPT and PH-INEPT+ pulse sequences were used for polarization transfer from 1H to 14N. Dependence of 14N NMR signal on inter-pulse delays was simulated and perfectly reproduced experimentally. Up to 0.33% 14N polarization was obtained for the hydrogenated salts with 14N T1 of ca. 2–3 s at 7 T.

WOH 04:45-05:05
Frequency-Chirped MAS DNP and Combination with Electron Decoupling
Presenter: Nicholas Alaniva (ETH-Zürich)
All Authors: Nicholas Alaniva (ETH-Zürich); Snaedis Björgvinsdottir (ETH-Zürich); Marthe Millen (ETH-Zürich); Alexander Barnes (ETH Zurich)
Electron decoupling and frequency-chirped dynamic nuclear polarization (DNP) are methods that have been shown to improve sensitivity in magic-angle spinning (and static) DNP NMR. Here, these two methods are combined in MAS DNP experiments, resulting in additive improvements in DNP-enhanced NMR signal. This combination improves the enhancement by 40% with Finland trityl at 4 kHz, while frequency-chirped DNP alone results in 22% improvement using TEMTriPol, spinning at 8 kHz. Frequency-chirped MAS DNP is investigated with TEMTriPol, Finland trityl, and AMUPol, and the microwave frequency chirps from the frequency-agile gyrotron are directly observed during the experiment using a "GHz-detection" circuit.

WOH 05:05-05:25
Feasibility of HyperCEST Spectroscopy With Caged Xenon Under Enhanced Relaxation Conditions
Presenter: Hannah Gerbeth (German Cancer Research Centre (DKFZ))
All Authors: Hannah Gerbeth (German Cancer Research Centre (DKFZ)); Leif Schroeder (Deutsches Krebsforschungszentrum (DKFZ))
Xenon-129 HyperCEST, promising for molecular diagnostics with NMR/MRI, requires further research for the translation to in vivo applications due to altered conditions in physiological environments. An in vitro phantom solution developed for sequence optimization prior to animal testing reduced the transversal and longitudinal relaxation times of hyperpolarized Xenon-129 from 40 s (in water) to 30 ms and from 110 s to 11 s, respectively, which is close to physiological values. Testing the feasibility of HyperCEST acquisitions using CrA-ma under these enhanced relaxation conditions yielded a relative HyperCEST contrast of 30% with promising spectral resolution, confirming phantom's suitability for sequence testing and optimization. Further research will investigate the effects of other optimization parameters on HyperCEST signals in this physiologically representative setup.

WOH 05:25-05:45
Optically Enhanced Solid-State 1H NMR Spectroscopy
Presenter: Lyndon Emsley (EPFL)
All Authors: Federico De Biasi (EPFL); Michael Hope (University of Warwick); Ganesan Karthikeyan (Aix-Marseille University); Gilles Casano (Aix-Marseille University); Moreno Lelli (CERM-University of Florence); Olivier Ouari (Aix-Marseille University); Lyndon Emsley (EPFL)
There is high current interest in optically induced hyperpolarization methods, since they can in principle yield very high levels of polarization, even at room temperature. Notably, for solid-state NMR, photo-CIDNP has been demonstrated for 13C and 15N nuclei, but their low natural abundance usually traps the hyperpolarization near the polarizing agent, impeding the development of bulk hyperpolarization. Polarization of 1H nuclei would allow for efficient spin diffusion. Here, we report optically enhanced solid-state 1H NMR spectroscopy via direct 1H photo-CIDNP,1 yielding a bulk signal enhancement under continuous laser irradiation at 450 nm. Our findings enable a novel strategy for hyperpolarized NMR beyond the limits of microwave-driven DNP.

[1] F. De Biasi, et al., J. Am. Chem. Soc. 2023, 10.1021/jacs.3c03937

8:45 AM - 10:10 AM, THURSDAY
ThOA: Biomolecular Solutions IV
Jeffrey Peng (University of Notre Dame) presiding
Merrill Hall

ThOA 08:45-09:10
A Complete Set of Cross-Correlated Relaxation Experiments for Structural Studies of Intrinsically Disordered Proteins
Presenter: Anna Zawadzka-Kazimierczuk (University of Warsaw)
All Authors: Anna Zawadzka-Kazimierczuk (University of Warsaw); Paulina Bartosińska-Marzec (University of Warsaw); Bartłomiej Banaś (University of Warsaw); Clemens Kauffmann (University of Vienna); Daniel Braun (University of Vienna); Irene Ceccolini (University of Vienna); Krzysztof Kazimierczuk (University of Warsaw); Julian Holzinger (University of Vienna); Thomas Schwarz (University of Vienna); Georg Kontaxis (University of Vienna); Wiktor Koźmiński (University of Warsaw); Robert Konrat (University of Vienna)
Intrinsically disordered proteins (IDPs) play a key role in many physiological processes, particulary in the context of signaling and regulation. This makes IDPs an important target of research. The complex interplay of structure and dynamics in IDPs suggests NMR as the technique of choice for their investigation, providing information at atomic resolution. Due to the transient nature of structural elements in IDPs, they are arguably best described via phi and psi backbone angle distributions, which can be extracted from a set of cross-

correlated relaxation (CCR) rates. Here we present such a set of IDPs-tailored 4D experiments and demonstrate their application.

ThOA 09:10-09:30

Disentangling NOE from 1H relaxation measurements reveal a third state in Watson-Crick-Franklin to Hoogsteen DNA base pair dynamics.

Presenter: Rubin Dasgupta (Uppsala University)

All Authors: Rubin Dasgupta (Uppsala University); Christian Steinmetzger (Uppsala University); Julian Ilgen (University Regensburg); Katja Petzold (Uppsala University)

Watson-Crick-Franklin (WCF) to Hoogsteen (HG) base pair dynamics is studied using 1H R1ρ relaxation dispersion experiment in a model DNA duplex with 12 base pairs (A2 DNA). Typical NOE contributions to 1H relaxation was shown to be non-problematic and therefore revealed a previously not observed third state in the WCF – HG dynamics. This state persists even in the A2 DNA with various chemical modifications of the purine base that stabilises either the WCF or the HG conformation. The chemical shift and temperature dependent exchange parameters suggests that this state is more compact and helical. This study provides the first experimental evidence of a possible intermediate state in WCF – HG dynamics.

ThOA 09:30-09:50

New applications of TOCSY NMR for pursuing the structure and dynamics of protein sidechains

Presenter: Peter Hwang (Departments of Medicine, Biochemistry, University of Alberta)

All Authors: Peter Hwang (Departments of Medicine, Biochemistry, University of Alberta)

Total correlation spectroscopy (TOCSY) is used for chemical shift assignment of protein sidechains. We have developed three novel applications of TOCSY:

1) By appending TOCSY elements to the beginning of any NMR pulse sequence starting on 1H, we can measure initial 1H relaxation rates that are inversely proportional to protein sidechain dynamics, independent of variable J-coupling and cross-relaxation effects.

2) Others have created "pure" TOCSY elements by increasing the time spent along the z-axis so that cross-relaxation effects (NOE, ROE) cancel out. We use these elements to measure J-couplings in protein sidechains and determine chi1 dihedral angles.

3) TROSY-TOCSY with maximized NOE effect can be used for chemical shift assignment of protein sidechains in high molecular weight systems.

ThOA 09:50-10:10

Enhanced Sensitivity and Resolution CEST NMR by the Extended Hadamard Scheme

Presenter: Jihyun Kim (Weizmann Institute of Science)

All Authors: Jihyun Kim (Weizmann Institute of Science); Eriks Kupce (Bruker UK Ltd); Micael Silva (Weizmann Institute of Science); Sundaresan Jayanthi (Indian Institute of Space Science and Technology); Adonis Lupulescu (Weizmann Institute of Science); Rina Rosenzweig (Weizmann Institute of Science); Lucio Frydman (Weizmann Institute)

Although ¹⁵N Chemical Exchange Saturation Transfer (CEST) experiments are useful for studying conformational exchanges, measuring CEST-based profiles with adequate sensitivity and resolution is typically time-consuming. We introduce a novel ¹⁵N CEST method that alleviates these penalties by utilizing extended Hadamard multiplexing. By employing Hadamardized shaped pulses/gradient blocks for the ¹⁵N frequency saturation, clearer encoding profiles than those obtained using Hadamardized CW irradiation are achieved. Additionally, artifacts generated by the conventional Hadamard scheme due to cross-talk among various exchanging sites, are effectively eliminated by our recently-introduced extended Hadamard scheme. The enhanced performance of Hadamard CEST is demonstrated through profiles observed on drkN SH3, which enable the identification of minor conformers and tackling low-concentration samples where conventional CEST would typically fail.

8:45 AM - 10:10 AM, THURSDAY
ThOB: Metabolomics II

Daniel Raftery (University of Washington) presiding
Chapel

ThOB 08:45-09:10

Development and Applications of a Novel 13C High-Temperature Superconducting Probe at 21.1 T

Presenter: Art Edison (University of Georgia)

All Authors: Omid Sanati (University of Georgia); Ilya M. Litvak (NHMFL); William Brey (Florida State University); John Glushka (University of Georgia); Larry Hornak (University of Georgia); Jeremy Thomas (University of Florida); Vijay Ramaswamy (Bruker Switzerland AG); Nicolas Freytag (Bruker Switzerland AG); Arthur Edison (University of Georgia)

We have designed and fabricated a cryogenic 13C HTS probe that operates at 21.1 T. The probe can accommodate either a custom flattened sample tube with outer dimensions 3 mm x 6.2 mm or a 3 mm conventional cylindrical tube. The probe was fabricated in a standard Bruker CryoProbe body and is cooled by a Bruker CryoPlatform. 13C was optimized, resulting in an ASTM value of 5700:1 in 425 uL. The experimental B1 homogeneity measured by 810/90 pulse width values is nearly 70% and in agreement with simulations. The probe has good lineshape and can be shimmed using the standard Bruker TopShim routine. The high sensitivity afforded by this probe greatly improves 13C detection experiments such as INADEQUATE and HETCOR.

ThOB 09:10-09:30

Developing HRMAS 13C NMR Reactor for Monitoring Real-Time Live Cell Metabolomic Reactions

Presenter: Rajshree Ghosh Biswas (MGH Harvard Medical School)

All Authors: Ella J. Zhang (Massachusetts General Hospital); Aidan Pavao (Brigham and Women's Hospital, Harvard Medical School); Rajshree Ghosh Biswas (Massachusetts General Hospital); Lynn Bry (Brigham and Women's Hospital); Leo Cheng (MGH Harvard Medical School)

Cells are the fundamental units where all biological activity occur. Real-time biochemical changes, within live cells, can be monitored and quantified via NMR to gain critical insights into pathophysiological processes. This study monitors the cellular metabolism of pathogenic Clostridioides difficile (C. diff., 100,000 cells) in 13C-media using 13C HRMAS NMR, for 48 hours. Due to the small sample volume, the sensitivity of such studies is limited. This proof-of-concept-study utilizes higher NMR sensitive nuclei (1H) to quantify less sensitive nuclei (13C) via 1D 1H HSQC through 13C editing (1H[13Ced]). A 13.9X improvement in S/N by 1H[13Ced], compared to standard 13C NMR, was observed, corresponding to a timesaving factor of 194. This makes monitoring in vivo processes of mass limited samples feasible.

ThOB 09:30-09:50

Advancing towards monitoring bacteria metabolism through SABRE

Presenter: Julia Schulte-Hermann (Karlruhe Institute of Technology)

All Authors: Julia Schulte-Hermann (Karlruhe Institute of Technology); Jing Yang (Karlsruher Institute of Technology); Hagen Rießland (Karlsruhe Institute of Technology); Camilla Stolle (Karlsruhe Institute of Technology); Sören Lehmkuhl (KIT); Kersten Rabe (Karlsruhe Institute of Technology); Jan Korvink (Karlsruher Institute of Technology); Neil MacKinnon (Karlsruhe Institute of Technology)

Monitoring and detecting metabolism of bacteria is essential to understanding and analyzing bacteria function and for drug development. In this work, we employed Signal Amplification by Reversible Exchange (SABRE) to detect bacteria metabolites. Three challenges were tackled to evaluate the suitability of SABRE: toxicity of the SABRE components for bacteria, hyperpolarization in aqueous environments and SABRE compatibility with metabolites. The toxicity evaluation and the hyperpolarization limit detection in aqueous

environments show promising results towards in vivo SABRE for bacteria metabolites. Additionally, we showed SABRE hyperpolarization of metabolites of bacteria. The next step is the identification of those metabolites. We expect to apply SABRE on living bacteria directly in-vivo without cell death with the determined compound concentrations through the toxicity assays.

ThOB 09:50-10:10

From Conventional to High-Power-Laser-Driven Irradiation: Timely Detection of Magnetic Resonance Biomarkers in Cells Cultures

Presenter: Ioana Fidel (Extreme Light Infrastructure Nuclear-Physics)

All Authors: Ioana Fidel (Extreme Light Infrastructure Nuclear-Physics); Diana Serafin (Extreme Light Infrastructure Nuclear-Physics); Gabriel Petrisor Bleotu (Extreme Light Infrastructure Nuclear-Physics); Georgiana Giubega (Extreme Light Infrastructure Nuclear-Physics); Liviu Neagu (Extreme Light Infrastructure Nuclear-Physics); Mihai Iovea (Accent Pro 2000); Cezara Zagrean Tuza (University Babes Bolyai); Anamaria Hanganu (University of Bucharest); Silvana Vasilca (Extreme Light Infrastructure Nuclear-Physics); Daniel Negut ("Horia Hulubei" National Institute for Physics and Nuclear Engineering (IFIN-HH)); Mihai Suditu (Amethyst Radiotherapy Center); Roxana Popescu ("Horia Hulubei" National Institute for Physics and Nuclear Engineering (IFIN-HH)); Mihaela Bacalum ("Horia Hulubei" National Institute for Physics and Nuclear Engineering (IFIN-HH)); Madalin Rosu (Extreme Light Infrastructure - Nuclear Physics); Ovidiu Tesileanu (Extreme Light Infrastructure - Nuclear Physics); Adrian Mihai Voda (Extreme Light Infrastructure - Nuclear Physics); Aude Sadet (Extreme Light Infrastructure - Nuclear Physics); Ion Chiricuta (Amethyst Radiotheraphy Center); Paul Vasos (Extreme Light Infrastructure - Nuclear Physics)

Understanding the biomolecular effects of high dose-rate radiation is a challenge with implications for clinical translation1. Biological systems are impacted differently by various types of radiation.

Our aim is to detect via magnetic resonance spectroscopy differences in metabolic profiles of cells irradiated with conventional (Gy/min) and high-intensity-laser dose rates (Gy/ns).

We have developed a magnetic-resonance profiling method for radiation effects in cells. Changes in metabolites concentrations were observed after irradiation.

We showed for the first time that Magnetic Resonance biomarkers can be used for timely detection of radiation dose-rate effects. The initial attempts to investigate the effects of high dose-rate radiation on cells are presented, focusing on utilizing the radiation generated by accelerated secondary sources through a high-intensity laser.

8:45 AM - 10:15 AM, THURSDAY
ThOC: Computation for SS NMR

Robert Schurko (Florida State University) presiding

Woodlands

ThOC 08:45-09:10

Modulation of Structure and Dynamics in Solids via Directional Non-Covalent Interactions. The Roles of NMR Experiments and Theory

Presenter: David Bryce (University of Ottawa)

All Authors: David Bryce (University of Ottawa)

Non-covalent element-based electrophilic interactions, including halogen bonds, chalcogen bonds, and more recently elucidated classes such as matere bonds, play critical roles in governing the structure and dynamics of a wide range of materials, catalysts, and biomolecules. We report here on our recent multinuclear solid-state magnetic resonance and quadrupole resonance studies of the structure and dynamics of engineered cocrystals featuring a range of non-covalent interactions. Our work relies on a strong interplay between experimental and computational efforts. Two examples include (i) the use of deuterium NMR to understand the role of halogen bonds in modulating rotational dynamics in solids and (ii) the use of 185/187Re NMR and NQR to probe matere-bonded solids.

ThOC 09:10-09:30

Biexponential I = 3/2 Spin-Lattice Relaxation in the Solid State: Multiple-Quantum 7Li NMR as a Probe of Fast Ion Dynamics

Presenter: Stephen Wimperis (Department of Chemistry, Lancaster University)

All Authors: Stephen Wimperis (Department of Chemistry, Lancaster University); George E. Rudman (Department of Chemistry, Durham University); Karen E. Johnston (Department of Chemistry, Durham University)

Spin-lattice relaxation is used in 7Li NMR studies of materials of potential use in solid-state batteries as a probe of ion mobility. The analysis often assumes exponential behaviour or, equivalently, a single T1 time constant. However, the spin-lattice relaxation of spin I = 3/2 nuclei, such as 7Li, is in general biexponential. Here, we show that triple-quantum filtered NMR experiments, as previously exploited in NMR of liquids, can be used to observe and quantify biexponential 7Li spin-lattice relaxation in solids exhibiting ion mobility. The results are less straightforward to interpret than in liquids as a consequence of direct triple-quantum excitation by the finite-power pulses, but we show that this unwanted excitation can be modelled theoretically and accommodated within the analysis.

ThOC 09:30-09:50

An Exploration of Dipolar Order in Wideline Solid State NMR

Presenter: James Kimball (Florida State University)

All Authors: James Kimball (Florida State University); Lucio Frydman (Weizmann Institute); Robert Schurko (FSU and NHMFL)

Dipolar order can be used as a source of polarization transfer in SSNMR experiments. However, the short lifetimes of dipolar ordered states (T1D) have curbed a more widespread exploitation of this phenomenon. Herein, we explore the relationship between T1D and the signal enhancement afforded by the dipolar reservoir. Novel variants of the Progessive Saturation of the Proton Reservoir (PROSPR) sequence are presented based on both adiabatic and pulsed approaches, and used for the application of wideline SSNMR powder patterns of low-γ nuclei such as 109Ag, 99Ru, and 103Rh.

ThOC 09:50-10:15

Cryogenic Solid-State NMR and MAS DNP Investigation of Metastable Species Formed along Crystallization Processes

Presenter: Giulia MOLLICA (CNRS, ICR)

All Authors: Giulia MOLLICA (CNRS, ICR)

In the attempt of better understanding the mechanisms underlying crystallization, we recently introduced cryogenic solid-state NMR and MAS DNP strategies enabling time-resolved investigation of crystallization of polymorphic organic solids from bulk and confined solutions. Here, I will discuss how these strategies enable the stabilization and characterization of metastable forms produced along crystallization pathways. I will show how the increased NMR contrast based on transport of DNP-enhanced polarization can be exploited for revealing and quantifying the formation of a small amount of a polymorph within a distinct polymorph, yielding unique quantitative information on the spatial distribution of two chemically identical phases. Then, I will discuss how NMR can help revise the structure of a recently discovered metastable form of calcium carbonate.

10:45 AM - 12:35 PM, THURSDAY
ThOD: Biomolecular Solids III

Rachel Martin (University of California, Irvine) presiding

Merrill Hall

ThOD 10:45-11:10

DNP-enhanced solid-state NMR in mechanistic Membrane Protein Research

Presenter: Clemens Glaubitz (Goethe University, BMRZ)

All Authors: Jiafei Mao (Goethe University Frankfurt); Xiao He (East China Normal University); Johanna Becker-Baldus (Goethe University Frankfurt); Jagdeep Kaur (Goethe University Frankfurt); Anne Mayer (Goethe University Frankfurt); Samuel Seidl (Goethe University Frankfurt); Clemens Glaubitz (Goethe University, BMRZ)

We will demonstrate the use of cross-effect-based dynamic nuclear polarization (DNP) for solid-state NMR studies of membrane-based

photoreceptors. We will present our approach for light-induced and cryo-captured photointermediates of outward and inward proton pumps from the microbial rhodopsin family. In particular, we will show how the combination of DNP-generated solid-state NMR parameters for the chemical shift in conjunction with linear scaling QM/MM approaches can be used to improve 3D structural models to obtain accurate predictions of the optical properties of these proteins. Based on this approach, we are able to explain the green/blue color change in the widespread proteorhodopsin family. A perspective for the general use of solid-state NMR to study light-induced biochemical processes in membranes is discussed.

ThOD 11:10-11:30

Solid-State and Solution NMR Comparison of GPCR Energy Landscapes in Membrane Mimetics

Presenter: Matthew Eddy (University of Florida)

All Authors: Arka Prabha Ray (University of Florida); Beining Jin (University of Florida); Naveen Thakur (University of Florida); Nessa P. Afsharian (University of Florida); Matthew Eddy (University of Florida)

G protein-coupled receptors (GPCRs) are sensory membrane proteins comprising the largest class of "druggable" targets. GPCR function is closely linked with their conformational equilibria, which is controlled by interactions with ligands and by the presence of specific lipids. Utilizing both 19F solid-state magic angle spinning (MAS) NMR and 19F-NMR with aqueous solutions, we systematically compared the conformational equilibria of the human A2A adenosine receptor (A2AAR) in three distinct membrane mimetics: detergent micelles, lipid nanodiscs, and unilamellar vesicles. For A2AAR complexes with inhibitors, 19F spectra were surprisingly consistent across three different membrane mimetics. However, spectra of A2AAR complexes with activating ligands revealed intriguing differences among all three membrane mimetics, underscoring the significant influence of membrane mimetics on receptor structure and function.

ThOD 11:30-11:50

Mapping the energy landscape, stages, and domains of membrane protein unfolding using solid-state NMR spectroscopy

Presenter: Vlad Ladizhansky (University of Guelph)

All Authors: Peng Xiao (University of Guelph); Philip Drewniak (University of Guelph); Dylan Dingwell (University of Toronto); Leonid S. Brown (University of Guelph); Vlad Ladizhansky (University of Guelph)

Understanding how the amino acid sequence dictates protein structure is a fundamental problem in molecular biology. It is especially challenging for membrane proteins because they reside in the complex environment of a lipid bilayer. We obtain an atomic-level picture of the thermally induced unfolding of a membrane-embedded alpha-helical protein human aquaporin 1 using MAS NMR. The protein unfolds in two steps beginning with an extracellular loop, and proceeds to an intermediate state with a native-like helical packing. In the second step, the transmembrane domain unravels as a whole, resulting in a heterogeneous misfolded state. We show that folding of loops kinetically stabilize membrane protein structure, in support of the notion of the third stage in the membrane protein folding model.

ThOD 11:50-12:10

A Targeted DNP Approach to Study the Activity of the Membrane Protein Ail in Whole Bacterial Cells

Presenter: Nesreen Elathram (UC San Diego)

All Authors: Nesreen Elathram (UC San Diego); Melinda R. Serrato (UC San Diego); Drake Jimenez (UC San Diego); Francesca M. Marassi (Medical College of Wisconsin); Galia Debelouchina (UC San Diego)

Ail (attachment invasion locus) is an outer membrane protein that plays a crucial role in adhesion, invasion, virulence, and, most importantly, human serum resistance of the deadly Yersinia Pestis, a plague-causing bacterium. In this study, we use DNP-enhanced solid-state NMR spectroscopy to examine the extramembranous activity of Ail in whole bacterial cells. We specifically label the extracellular loop of the protein with an unnatural amino acid (UAA) that subsequently gets targeted with a tetrazine-based DNP agent, with the goal to efficiently enhance the NMR signals of the region of interest of the protein against the cellular background. This will enable us to selectively probe Ail's structure and interactions with the surrounding membrane and proteins found in human blood serum.

ThOD 12:10-12:35

Adventures in Biomolecular MAS DNP

Presenter: Joanna Long (University of Florida)

All Authors: Maria Luiza Caldas Nogueira (The University of Florida); Diana Tymochko (Oberlin College); Nhi Tran (University of Florida); Faith Scott (National High Magnetic Field Laboratory); Frederic Mentink-Vigier (National High Magnetic Field Laboratory, Florida State University); Rittik Ghosh (Department of Biochemistry, University of California - Riverside, CA 92521); gwladys Riviere (DZNE); Len Mueller (University of California Riverside); Joanna Long (University of Florida)

NMR can provide unique insights into biomolecular structure, dynamics, and chemistry with sufficient sensitivity and resolution. With recently developed biradicals, we reliably see 10- to 100-fold gains in sensitivity at 14.1 T using MAS DNP for microcrystalline enzymes and membrane-active peptides. Notably, we capture similar spectral resolution, relaxation times, and protein activity under MAS DNP conditions that we observe via conventional NMR. The improved sensitivity enables us to pursue multidimensional experiments in hours that previously required days/weeks of NMR time or were simply not possible. A cross-section of our recent results will be presented.

10:45 AM - 12:35 PM, THURSDAY
ThOE: Hyperpolarization III
Thorsten Maly (Bridge 12 Technologies, Inc.) presiding
Chapel

ThOE 10:45-11:10

Synchronizing a High-Rep Rate Tunable Pulsed Laser with MAS NMR Experiments for the Development of Optically Pumped NMR Methods

Presenter: Claudia Avalos (New York University)

All Authors: Claudia Avalos (New York University)

Optically pumped electron spins may be used to enhance the sensitivity of NMR experiments both in the liquid and solid state. Small molecules such as chromophore-radical and donor-bridge-acceptor systems exhibit high transient electron spin polarization upon laser irradiation at low magnetic fields and have so far remained relatively unexplored for Optical DNP NMR applications. These species are excited at different wavelengths and often require high pulse energies in order to produce a high enough concentration of polarized species to be observed in EPR. In this work we present our progress towards synchronizing a high rep-rate tunable pulsed laser system with high-field MAS NMR experiments with the aim of exploring a variety of systems for optical DNP.

ThOE 11:10-11:30

Scaling Up Hyperpolarization of 117Sn and 1H for Neutron Optics-Based Time-Reversal Symmetry Investigations.

Presenter: Abubakar Abdurraheem (Wayne State University)

All Authors: Abubakar Abdurraheem (Wayne State University); Shahabuddin Alam (Southern Illinois University); Anthony F. Petrilla (Southan Illinois University); Boyd Goodson (Southern Illinois University); Roman Shchepin (South Dakota School of Mines & Technology); Michael Snow Williams (Indiana University); Eduard Chekmenev (Wayne State University)

Heavy atoms were identified to violate fundamental asymmetries. Recent measurement of angular asymmetry of 117Sn indicates that 117Sn is one of the best candidates for TRIV searches. The proposed neutron-optics experiment for this search requires a large volume of 117Sn nuclei with high magnetization. We report our progress in creating sustainable HP 117Sn states in two tin-functionalized imidazole substrates using SABRE technique. Moreover, for other TRIV experiments that involve high-energy neutron resonance, highly polarized protons in large volume and magnetization are desired. We are reporting on our progress to create SABRE-hyperpolarized

samples of up to one liter in size, where polarization is confirmed using low-field NMR spectrometer detecting at 41 kHz.

ThOE 11:30-11:50

Magnetic-Field Dependence of LC-Photo-CIDNP Nuclear-Spin Hyperpolarization via a Rapid-Shuttling Device

Presenter: Silvia Cavagnero (University of Wisconsin-Madison)

All Authors: Silvia Cavagnero (University of Wisconsin-Madison); Siyu Li (University of Wisconsin-Madison); Shibani Bhattacharya (New York Structural Biology Center); Ching-Yu Chou (Field Cycling Technology LTD); Shu-Cheng Chou (Field Cycling Technology LTD); Marco Tonelli (University of Wisconsin-Madison); Michael Goger (New York Structural Biology Center); Hanming Yang (University of Wisconsin-Madison); Arthur G. Palmer (New York Structural Biology Center and Columbia University)

Liquid-state low-concentration photochemically induced dynamic nuclear polarization (LC-photo-CIDNP) is an emerging technology tailored to enhance NMR sensitivity via LED-mediated optical irradiation. LC-photo-CIDNP enables the detection of solvent-exposed aromatic residues in isolation or within polypeptides and proteins. This study investigates the magnetic-field dependence of the LC-photo-CIDNP of a Trp isotopolog bearing a H-C quasi-isolated spin pair (QISP). We employed a new rapid-shuttling side-illumination field-cycling device that enables ultra-fast vertical movements of NMR samples within the bore of a superconducting magnet. Resonance lineshapes were excellent, and the effect of several fields (1.18-7.08 T) on hyperpolarization efficiency were explored. LC-photo-CIDNP enhancements up to 1,200 were obtained at 50 MHz (1.18 T), suggesting exciting avenues to hypersensitive LED-enhanced NMR in liquids at low field.

ThOE 11:50-12:10

Optimizing Signal Amplification by Reversible Exchange: Recyclable Perfluorinated Iridium Catalysts for Enhanced [1-13C]Pyruvate Hyperpolarization in Fluorinated Solvents and its Metal-Free Aqueous Delivery

Presenter: Jess Benjamini-Ettedgui (NHLBI/NIH)

All Authors: Jess Ettedgui (NHLBI/NIH); Burchelle Blackman (NHLBI/NIH); Natarajan Raju (NHLBI/NIH); Eduard Chekmenev (Wayne State University); Boyd Goodson (Southern Illinois University); Murali Krishna (NCI/NIH); Rolf Swenson (NHLBI/NIH)

Signal Amplification By Reversible Exchange in SHield Enabled Alignment Transfer (SABRE-SHEATH) can rapidly hyperpolarize [1-13C]pyruvate, however its clinical use is constrained by the inherent toxicity of the Iridium catalyst. Addressing this limitation, we present a novel hydrophobic SABRE catalyst featuring bis(polyfluoroalkylated) tails. This perfluorinated catalyst effectively sustains polarization levels in methanol and reaches increased levels (up to 16%) in a methanol-hydrofluoroether (HFE) blend. Its removal from HP [1-13C]-pyruvate aqueous solution is facilitated through ReD-SABRE or biphasic extraction, resulting in 177 ppb and 370 ppb residual Ir respectively. Additionally, the perfluorinated catalyst showcases recyclability and sustained activity over multiple [1-13C]pyruvate hyperpolarization cycles, positioning SABRE-SHEATH as an economically viable substitute for the formulation of biocompatible HP [1-13C]pyruvate in advanced molecular imaging applications.

ThOE 12:10-12:35

Continuous-wave NMR for Nuclear Physics Polarized Scattering Experiments

Presenter: James Maxwell (Jefferson Lab)

All Authors: James Maxwell (Jefferson Lab); Hai Dong (Jefferson Lab); Chris Keith (Jefferson Lab)

Dynamically nuclear polarized targets for accelerator-based scattering experiments have relied upon continuous-wave NMR polarization measurements since their first uses in pion--proton scattering in the early 1960's. The Liverpool Q-meter system was conceived in the late 1970's to improve CW-NMR measurements using phase-sensitive demodulation, and it became the workhorse device for target polarization measurements throughout the fields of high-energy and nuclear physics. As the aging components of these Q-meters have become difficult to service, we have developed a new series of Q-meters for our high-luminosity electron scattering experiments at Jefferson Lab. We will discuss this new system, which hews closely to the original Liverpool electronic design, with modern components and a more modular, flexible architecture.

10:45 AM - 12:35 PM, THURSDAY
ThOF: Materials III
Gaël De Paëpe (CEA / Univ. Grenoble Alpes) presiding
Woodlands

ThOF 10:45-11:10

Advanced Solid-State NMR Spectroscopy of Materials using Fast Magic Angle Spinning and Dynamic Nuclear Polarization

Presenter: Amrit Venkatesh (National High Magnetic Field Laboratory, Florida State University)

All Authors: Amrit Venkatesh (National High Magnetic Field Laboratory, Florida State University); Rishi Verma (Department of Chemical Sciences, Tata Institute of Fundamental Research (TIFR)); Lucas Urbano José (Department of Physics, Chemistry and Pharmacy, University of Southern Denmark); Lyndon Emsley (EPFL); Ulla Gro Nielsen (Department of Physics, Chemistry and Pharmacy, University of Southern Denmark); Vivek Polshettiwar (Department of Chemical Sciences, Tata Institute of Fundamental Research (TIFR)); Julien Trébosc (Univ. Lille, CNRS, Centrale Lille, ENSCL, Univ. Artois, UMR 8181, UCCS, Unité de Catalyse et Chimie du Solide); Aaron J. Rossini (Department of Chemistry, Iowa State University)

Solid-state NMR spectroscopy provides vital atomic-level structure information, but its low intrinsic sensitivity frequently inhibits the application of advanced multidimensional experiments. Here, we utilize fast magic angle spinning (MAS), indirect detection, high magnetic fields and dynamic nuclear polarization (DNP) to enhance solid-state NMR sensitivity. Fast MAS allows proton detection of very low-gyromagnetic ratio quadrupolar nuclei including 17O, 25Mg, 35Cl, 39K, 47/49Ti and 91Zr at 9.4 T. The efficacy of proton detection of 35Cl at 18.8 T and 28.2 T is also compared. Applications of high field solid-state NMR on defected aluminosilicates will be demonstrated. Finally, we demonstrate the application of MAS DNP to MgAl layered double hydroxides, which reveals the mechanism of phosphate removal by these materials.

ThOF 11:10-11:30

Local structure and diffusion of sodium in a hybrid glass from high-temperature 23Na MAS NMR at 20 T

Presenter: Dominik Kubicki (University of Birmingham)

All Authors: Pascal Kolodzeiski (Inorganic Chemistry, Department of Chemistry and Chemical Biology, TU Dortmund); Benjamin Gallant (School of Chemistry, University of Birmingham); Dominik Kubicki (University of Birmingham); Sebastian Henke (Inorganic Chemistry, Department of Chemistry and Chemical Biology, TU Dortmund)

The glassy state in coordination polymers, such as MOFs, is relatively rare. One of the main practical challenges is the missing understanding of their structure-property relationships that would enable design and tuning of the glassy behavior and glass transition temperature to enable easier processability.

We report an Na-containing additive that enables unprecedented tunability of the glass transition temperature in a hybrid Zn-based MOF glass. I will discuss high-temperature (300-700 K) 23Na MAS NMR experiments which enabled us to determine the speciation of the dopant, quantify its dynamics and impact on the glassy behavior across a range of compositions.

ThOF 11:30-11:50

Surface Sensitive Solid-State NMR Spectroscopy Reveals Local Structural Features of an Industrial Catalyst

Presenter: Sonja Egert (University of St Andrews)

All Authors: Sonja Egert (University of St Andrews); Paul B. Webb (University of St Andrews); Jeremy J. Titman (University of Nottingham); Sharon Ashbrook (University of St Andrews)

Methyl methacrylate (MMA), used in the production of acrylics, is an industrially important monomer. Its production via Mitsubishi Chemical's Alpha Process is a growing technology based on the use

of silica-based heterogeneous catalysts with zirconium oxide and caesium hydroxide surface species. In-depth characterisation of the disordered surface structure is carried out to establish links between the atomic structure, composition, and catalytic performance, ultimately working towards the smart design of next-generation catalysts. To this end, a library of model compositions with systematically varied surface loadings is analysed using Si-29, Cs-133, and O-17 solid-state NMR spectroscopy supplemented by DNP and isotopic enrichment. The results are complemented by ab initio calculations as well as XAFS experiments.

ThOF 11:50-12:10

Unifying finally all NMR/DNP data related to substituted hydroxyapatite by extensive NMR/DNP crystallography

Presenter: Christian Bonhomme (Sorbonne University)

All Authors: christian bonhomme (Sorbonne University); Melinda Duer (University of Cambridge); Danielle Laurencin (ICGM Montpellier University); Christel Gervais (Sorbonne University)

Bone mineral has a component similar to hydroxyapatite (Ca10(PO4)6(OH)2), HAp, but with a number of important substitutions such as by carbonate and silicate anions. For decades, the structure of substituted HAp has remained a mystery due to the potential disorder present. Our recent contribution (based on all experimental and numerical data currently available in the literature) tackles four spectroscopic challenges related to substituted HAp structures leading for the first time to a true quantitative and coherent interpretation of all types of solid-state NMR data. Extensive modeling at DFT level has been performed (35 models) leading to a statistically acceptable representation of numerous charge compensation mechanisms involved in HAp derived structures.

ThOF 12:10-12:35

Magnetic Resonance for Li-ion Batteries: From NMR to EPR Imaging

Presenter: Bingwen Hu (East China Normal University)

All Authors: Bingwen Hu (East China Normal University); Fushan Geng (East China Normal University); Chao Li (East China Normal University)

(1) EPR Imaging: Copper and aluminum current collectors are difficult to penetrate by light or electrons; therefore, most characterization techniques observe lithium deposition in side view. We have developed in-situ high-resolution x-band EPR imaging which can observe lithium deposition in front view. EPR imaging was also developed for all-solid-state batteries. We also developed a 1D quantitative EPR imaging techniques with DPPH correction.

(2) EPR&NMR: In the Li2RuxSn1-xO3 system, EPR was confirmed to be able to detect trapped O2 in the cathode, while the popular RIXS method may not be able to distinguish between O2 and Ru-O2(δ-). We propose possible evolution paths for trapped O2. EPR and NMR were employed to study P2-Na0.66[Li0.22Mn0.78]O2, proving the existence of trapped O2 and O2n-.

POSTERS

Odd numbers present Monday & Wednesday. Even numbers present Tuesday & Thursday

Biomolecular Solids NMR (Posters 001-018)

POSTER 001

Longitudinal relaxation time in rotating frame (T1rho) approach to evaluate polymorphism of active pharmaceutical ingredients

Presenter: Luisa Almeida (Sao Paulo University)

All Authors: Luisa Almeida (Sao Paulo University); Rodrigo H. S. Garcia (Sao Paulo University); Julian Ticona (Federal University of Sao Paulo); Silvia L. Cuffini (Federal University of Sao Paulo); Eduardo de Azevedo (Sao Paulo University); Luiz Alberto Colnago (Embrapa)

Polymorphism in Active Pharmaceutical Ingredients (API) solid-dosages may influence their bioavailability. Relaxation times measurements in Time Domain Nuclear Magnetic Resonance (TD-NMR) instruments, recently, have been used to characterize API polymorphism. This work presents the measurements of longitudinal relaxation time in a rotating frame (T1rho) for API polymorphism characterization. We evaluated T1rho measurements of Mebendazole (MBZ) API polymorphs in solid mixtures (form C and A). Uni and multivariate statistics were calculated from the collected data. Linear regression coefficient (r) = 0.9199, std dev = 0.47, and Limit of Detection (LOD) = 36.07. The multivariate calibration RMSEP = 10.69, std dev = 11.93, and r = 0,9716. Therefore, T1rho measurements offer a complementary approach for solid-state API polymorphic characterization.

POSTER 002

Studying Sidechain Interactions in Dragline Spider Silk Fibers Using MAS Solid-State NMR and DNP

Presenter: Kevin Chalek (San Diego State University)

All Authors: Kevin Chalek (San Diego State University); Nesreen Elathram (UC San Diego); Galia Debelouchina (UC San Diego); Gregory P. Holland (San Diego State University)

Major ampullate (Ma) silk is used by spiders in web construction and is the strongest type of spider silk. Recently, our lab has found evidence of potential CH/π interactions taking place in major ampullate spidroin 2 (MaSp2) between Pro and Tyr sidechains using a new selective 1D 13C-13C spin-diffusion magic angle spinning solid-state NMR (MAS ssNMR) experiment. To further study sidechain interactions in dragline spider silk fibers, we have been conducting Dynamic Nuclear Polarization (DNP) MAS ssNMR experiments to improve sensitivity and enable multi-dimensional experiments. These experiments reveal potential cation/π interactions between the positively charged Arg sidechain and the aromatic Tyr ring in MaSp1, allowing us to build more comprehensive models of spider silk structure.

POSTER 003

Investigating the Conformational Dynamics of the Human A2A Adenosine Receptor in Lipid Vesicles by 19F MAS Solid-State NMR

Presenter: Beining Jin (University of Florida)

All Authors: Beining Jin (University of Florida); Arka Prabha Ray (University of Florida); Matthew Eddy (University of Florida)

G protein-coupled receptors (GPCRs) are sensory integral membrane proteins involved in a wide range of physiological processes. With the aim of studying GPCRs in physiological conditions, we present optimized protocols for preparing samples of functional 19F-labeled human A2A adenosine receptor (A2AAR), a class A GPCR, incorporated into lipid vesicles composed of binary and ternary lipid mixtures. A2AAR vesicle preparations yielded 19F magic angle spinning (MAS) solid-state NMR spectra of similar spectral resolution and dispersion as preparations with lipid nanodiscs in aqueous solution. The similar resolution and dispersion facilitated direct comparisons between vesicle and nanodisc preparations, which enabled new insights into the impact of bulk membrane properties, such as curvature, on A2AAR structure and function.

POSTER 004

Determining nitrogen protonation state using experimentally determined tensor values and a novel spectral editing technique.

Presenter: Riley Nickles (Brigham Young University)

All Authors: Riley Nickles (Brigham Young University); James Harper (Brigham Young University)

Spectral editing is a technique used to simplify NMR spectra and distinguish parts of a molecule. Past spectral editing techniques include linearly dependent and independent metrics that are valuable in 13C NMR but inadequate with 15N. Based on the linearly independent metrics we propose a new metric using tensor values that distinguishes between the 15-N protonation states NH2 and NH3+. Nitrogen's multiple protonation states make distinction difficult, so to find a way to unambiguously separate the amine species is crucial. This distinction is invaluable in development and characterization of amine-based products such as pharmaceuticals because the charged NH3+ can do acid-base chemistry. Application of this technique will provide insight into amine behavior that will revolutionize our characterizations of amines.

POSTER 005

Activated State of a Wild-Type Potassium Channel

Presenter: Isaac Eason (Texas Tech University)

All Authors: Isaac Eason (Texas Tech University); Benjamin J. Wylie (Texas Tech University)

nward Rectifier K+ Kir Channels are tetrameric, ligand gated ion channels. In their native bilayers, these channels have an affinity for anionic lipids, which are often the activating ligands. In response to association with these ligands, these channels conduct an inward K+ current, regulating the resting membrane potential in excitable cells. Noted channelopathies include forms of heart disease, psychiatric disorders, and alcoholism. The prokaryotic channel KirBac1.1 shares its fold and regulatory mechanisms with homologous human channels. Recently, Solid State NMR (SSNMR) derived water accessibility restraints were used to refine the closed/inactive state, however, the open/active remained heretofore elusive. Here, SSNMR driven simulated calculations have given the first glimpse of the open/active state of a wild-type Kir channel.

POSTER 006

Structural Characterizations of IP6-Stabilized HIV-1 Capsid Protein Assemblies by Magic-Angle Spinning NMR

Presenter: Changmiao Guo (University of Delaware)

All Authors: Changmiao Guo (University of Delaware); Somayeh Zeinalilathori (University of Delaware); Kumar Tekwani Movellan (University of Delaware); Roman Zadorozhnyi (University of Delaware); Barbie K. Ganser-Pornillos (University of Utah); Angela Gronenborn (University of Pittsburgh School of Medicine); Tatyana Polenova (University of Delaware)

We characterized HIV-1 capsid (CA) assemblies of different morphologies using magic-angle spinning (MAS) NMR. We examined the structure of IP6-induced CA spherical, conical-like and tubular assemblies by 19F fast MAS NMR and 13C-detected experiments. High-resolution 1D and 2D 19F-detected spectra of CA assemblies of different morphologies were acquired in the presence of 1H decoupling. Both 19F-detected and 13C-13C MAS NMR spectra reveal discernable differences in the chemical shifts and line widths for IP6-induced tubular, conical and spherical CA assemblies, suggesting local structural variations in CA belonging to pentameric and hexameric units. Our work also demonstrates 19F fast MAS NMR as a sensitive reporter of conformational ensembles in HIV-1 CA assemblies possessing high degree of conformational heterogeneity.

POSTER 007

Solid-state NMR in native bacterial samples: characterization of the host-pathogen interactions

Presenter: Kyungsoo Shin (Medical College of Wisconsin)

All Authors: Kyungsoo Shin (Medical College of Wisconsin); James E. Kent (Sanford Burnham Prebys Medical Discovery Institute); Gopinath Tata (Medical College of Wisconsin); Bryce E. Ackermann (University of California San Diego); Alyssa Kraft (Medical College of Wisconsin); Rajlaxmi Panigrahi (Medical College of Wisconsin); Nicholas Wood (Medical College of Wisconsin); Galia Debelouchina (UC San Diego); Francesca M. Marassi (Medical College of Wisconsin)

In structural biology, NMR spectroscopy is the ideal tool for characterizing proteins and their interactions in their native environment due to its high sensitivity and sample type versatility. Yersinia pestis pathogenesis involves key membrane proteins, Adhesion Invasion Locus (Ail) and Plasminogen Activator (Pla), recruiting serum factors for bacterial survival. Through in situ NMR characterization, we reveal high-resolution insights into Ail and Pla in the bacterial outer membrane. Additionally, we produce their serum binding partners, Vitronectin and Plasminogen Activator Inhibitor 1, uncovering how bacterial membrane proteins recruit soluble counterparts. This NMR-focused approach identifies key residues and structural changes, validated through cell-based assays, unveiling previously unknown binding mechanisms in Y. pestis pathogenesis.

POSTER 008

Novel magnetically aligned peptoid macrodiscs and new triple resonance experiments for structure determination of membrane proteins by solid-state NMR

Presenter: Alexander Nevzorov (North Carolina State University)

All Authors: Alexander Nevzorov (North Carolina State University); Azamat Galiakhmetov (North Carolina State University); Adit Shah (North Carolina State University)

We present unprecedented spectroscopic resolution in highly aligned peptoid-based macrodiscs. Sub-ppm 15N NMR linewidths have been obtained for Pf1 coat-protein reconstituted in DMPC/DMPG macrodiscs composed of short (9-15 mer) synthetic peptoid belts consisting of alternating phenyl-ethyl and carboxyl-ethyl side chains. Moreover, new triple-resonance experiments suitable for (13C, 15N) labeled membrane proteins have been developed, which allow for both spectroscopic assignment and de-novo structure determination by combining the chiral 13Ca-1Ha dipolar couplings with 15N CSA and 1H-15N dipolar interactions. Finally, we present a computer-assisted approach for generating pulse sequences for high-resolution separated local-field experiments termed ROULETTE (Random Optimization Using the Liouville Equation Tailored To the Experiment). The resulting linewidths are superior to those obtainable by previous PISEMA-type experiments.

POSTER 009

19F Dynamic Nuclear Polarization with Fast Magic Angle Spinning: A Powerful Tool for Protein Structural Biology in Mammalian Cells

Presenter: Kumar Tekwani Movellan (University of Delaware)

All Authors: Kumar Tekwani Movellan (University of Delaware); Wenkai Zhu (University of Pittsburgh); Daniel Banks (Bruker Biospin Corporation); Brent Runge (University of Delaware); James Kempf (Burker Biospin Corporation); Angela Gronenborn (University of Pittsburgh School of Medicine); Tatyana Polenova (University of Delaware)

19F dynamic nuclear polarization (DNP) combined with fast magic angle spinning (MAS) NMR spectroscopy is a powerful technique to study proteins in their native cellular environments. We present DNP signal enhancements of over 100-fold in high signal-to-noise ratio MAS NMR spectra on nanomole-quantities of SARS-CoV-2 5F-Trp-NNTD protein electroporated into mammalian cells. These spectra required only minutes of measurement time. 2D 19F-19F dipolar correlation spectra have 19F line widths as narrow as ~2 ppm and exhibit 19F-19F intramolecular cross peaks between fluorine atoms as far as 9 Å apart. This work paves the way for 19F DNP-enhanced MAS NMR applications to cellular structural biology and it will permit to probe cellular events at the atomic level.

POSTER 010

MAS Solid State NMR versus Cryogenic Electron Microscopy: A Structural Biology Case Study on Protein Fibrils

Presenter: Blake Fonda (UC Davis)

All Authors: Blake Fonda (UC Davis); Masato Kato (UT Southwestern Medical Center); Yang Li (UT Southwestern Medical Center); Dylan Murray (UC Davis)

Cryogenic electron microscopy, cryo-EM, and solid state NMR are both excellent tools to investigate the structure of protein cross-β fibril assemblies. In this work, the same fibrils are investigated with both techniques. Cryo-EM analysis robustly determined a high-resolution structure of the rigid core of the fibrils and also effectively described the overall packing of protein molecules. Solid-state NMR secondary structure analysis agrees with the cryo-EM structure and additionally determined a flanking β-strand region not observed in the cryo-EM data. Refocused INEPT based NMR experiments showed that the remaining flanking residues are not heterogeneously rigidified but rather remain dynamically disordered with motion on the time scale of μs or faster. These experiments demonstrate the complementary nature of cryo-EM and solid-state NMR.

POSTER 011

Solid-State NMR of Suberin and Soil Carbon

Presenter: Anna De Angelis (Salk Institute for Biological Studies)

All Authors: Anna De Angelis (Salk Institute for Biological Studies); Leonardo R. Andrade (Salk Institute for Biological Studies); Thach V. Can (Salk Institute for Biological Studies); Riqiang Fu (National High Magnetic Field Laboratory); Shrikaar Kambhampati (Salk Institute for Biological Studies); James J. La Clair (University of California San Diego); Vladimir A. Maslivetch (Salk Institute for Biological Studies); Justin R. Pacheco (Salk Institute for Biological Studies); Shane Hunt (Salk Institute for Biological Studies); Nolan Mitschke (Salk Institute for Biological Studies); Nesreen Elathram (UC San Diego); Galia Debelouchina (UC San Diego); Klaus Schmidt-Rohr (Brandeis University); Joseph P. Noel (Salk Institute for Biological Studies)

Suberin, the main component of cork, is a plant biopolymer found in the outer bark of trees, roots, seeds, abscission zones, fruit skins, leaf bundle sheaths, and more. Suberin has been proposed as one of the key plant components for long-term carbon storage in soil. Our solid-state NMR studies aim at elucidating the molecular and supramolecular structure of Suberin, understanding its biological variability and its role in carbon accumulation in soil. Complex plant and soil samples at natural isotopic abundance containing this chemically recalcitrant polymer present unique challenges, calling for a multidisciplinary approach that integrates solid state MAS NMR spectroscopy with other techniques, including EPR and Mass Spectrometry.

POSTER 012

Solid-state NMR Characterization of Biosynthesized Melanin—Insights from 13C Labeling

Presenter: Christopher Klug (US Naval Research Laboratory)

All Authors: Christopher Klug (US Naval Research Laboratory); Mark Bovee (US Naval Research Laboratory); Nishani K. Jayakodi (US

Naval Research Laboratory); Matthew Laskoski (US Naval Research Laboratory); Aaron D. Smith (US Naval Research Laboratory); Tiffany M. Hennessa (US Naval Research Laboratory); Zheng Wang (US Naval Research Laboratory)

Our laboratory has successfully engineered a V. natriegens strain to rapidly and economically produce melanin from tyrosinase with the ultimate goal of controlling melanin structures at the nanoscale for a wide range of military and civilian applications. Insights into relative purities of the materials was obtained from CPMAS 13C and 15N NMR spectra for a series of melanins, a pure melanin, a commercial biosynthesized melanin, a fungal 1,8-dihydroxynaphthalene (DHN)-melanin prepared via E. viscosa, and several forms of melanin prepared via V. natriegens, biomass, heat-treated biomass, and supernatant. Insights into the metabolic pathways were obtained via a series of biosynthetically labeled melanins via the incorporation of 13C- and 15N-enriched tyrosines into the cell culture media.

POSTER 013

High-resolution structural studies of human A117V and M129V huPrP23-144 amyloids using solid-state NMR and Cryo-EM.

Presenter: Vidhyalakshmi Sridharan (The Ohio State University)
All Authors: Vidhyalakshmi Sridharan (The Ohio State University); Sean Smrt (The Ohio State University); Hanh Dao (The Ohio State University); Justin Thomas (The Ohio State University); Wenjun Sun (The Ohio State University); Daniel Conroy (The Ohio State University); Witold Surewicz (Case Western Reserve University); Christopher Jaroniec (The Ohio State University)

The C-terminal truncated (PrP23-144) is associated with familial amyloid cerebral angiopathy in humans and serves as an amenable invitro model for structural studies by ssNMR. Here, we investigate the structure of amyloid fibrils formed by Gerstmann-Straussler-Scheinker disease related mutants A117V and M129V of huPrP23-144 by magic-angle spinning ssNMR and Cryo-EM.2D15N-1H solid state NMR fingerprint spectra of 2H,13C,15N labeled fibrils indicate differences in the structures of the A117V and M129V mutants. This is corroborated by the cryo-EM density maps of these fibrils that show two distinct structures containing two protofilaments each.3D RFDR experiments (H)NNH and (H)CNH also reveal multiple long-distance structural restraints which we seek to incorporate into a high-resolution structural model reinforced by the density maps from Cryo-EM.

POSTER 014

Investigating the Structure and Maintenance of the GammaS Crystallin Hydrogel Interactome.

Presenter: Jaewon Suk (University of California, Irvine)
All Authors: Jaewon Suk (University of California, Irvine); Collin Sroge (University of California Irvine); Ravi Shankar Palani (Postdoc, MIT); Robert Griffin (Massachusetts Institute of Technology); Rachel Martin (UC Irvine)

1) Gamma-crystallins are structural proteins in the eye lens that increase the refractive index with respect to its immediate concentration. These proteins show impressive solubility at high concentrations, and reversibly form transparent hydrogels in vitro without the addition of any crosslinkers or additives. Using MAS-NMR, we have performed RFDR and TEDOR experiments on gammaS-crystallin gel samples to show the capability of solid-state NMR to obtain high-resolution spectra on semi-solid materials.

2) AlphaA crystallin are small heat-shock proteins that bind structurally impaired lens proteins to mitigate precipitation. Truncated alphaA crystallin retain said chaperone activity, but the mode and specificity of binding is unknown. We have performed 1H-15N-HSQC experiments to illuminate chemical-shift perturbations of amino acids that interact with said chaperone.

POSTER 015

Solid-State 13C NMR Evaluation of Lignin in Lignocellulosic Waste Materials

Presenter: Luiz Silvino Chinelatto Jr. (PETROBRAS)
All Authors: Daniel F. Cipriano (Federal University of Espírito Santo (UFES)); Luiz Chinelatto Jr (PETROBRAS); Hercilio de A. Honorato (PETROBRAS); Jair C. C. Freitas (Federal University of Espírito Santo (UFES))

In this work, ssNMR was used to study the lignin contents and structural changes of a set of lignocellulosic materials derived from sugarcane and various Brazilian plant species. Various physical and chemical processes can be used to extract lignin content and structural information of biomass, but an adequate characterization of the lignocellulosic raw material is not always easily feasible. This work aims to apply newly developed methodologies, based on ssNMR spectra recorded with CP and MAS, to determine the contents and the structural composition of lignin present in some lignocellulosic residues.

POSTER 016

HR-MAS 1H NMR Characterization of Extracellular Matrix and Metabolic Markers in Tendinopathy

Presenter: Maia Baumbach (Georgia Institute of Technology)
All Authors: Maia Baumbach (Georgia Institute of Technology); Johannes E. Leisen (Georgia Institute of Technology); Ryan Miller (Georgia Institute of Technology); Joe Pearson (Georgia Institute of Technology); Daniel M. Dinakarapandian (Georgia Institute of Technology); Anant K. Paravastu (Georgia Institute of Technology); Rishin Kadakia (Emory Musculoskeletal Institute); Johnna S. Temenoff (Georgia Institute of Technology); David A. Reiter (Emory University)

Quantitative magnetic resonance imaging (MRI) has the potential to detect microstructural changes associated with tendinopathy, a condition resulting in pain, restricted mobility, and weakness in the extremities. High resolution magic angle spinning proton nuclear magnetic resonance (HR-MAS 1H NMR) can aid in examining off-resonance signals attributed to extracellular matrix (ECM) damage, providing context to less-specific in vivo ultrashort echo time (UTE) MRI. This study aims to examine human and rat tendon imaging markers of ECM damage and rat tendon cellular metabolic response to load-stimulation. Results confirm lactate concentrations in human tendinopathic Achilles tendon and the ability to quantify lactate peaks using COSY spectra. Future work will examine loaded lactate differences in rat-rotator cuff tendon.

POSTER 017

Structural Characterization of E22G Aβ1-42 Fibrils via 1H detected MAS NMR

Presenter: Robert Griffin (Massachusetts Institute of Technology)
All Authors: Robert Griffin (Massachusetts Institute of Technology)

Amyloid fibrils have been implicated in the pathogenesis of several neurodegenerative diseases, the most prevalent example being Alzheimer's disease (AD). This has motivated our studies of fibril structures, of the familial Arctic mutant of Aβ1-42, E22G-Aβ1-42. Since the high surface activity of E22G-Aβ1-42 makes it difficult to obtain more than sparse quantities of fibrils, we employed 1H detected magic angle spinning (MAS) nuclear magnetic resonance (NMR) experiments to characterize the protein. The MAS NMR spectra indicate that the biosynthetic samples of E22G-Aβ1-42 fibrils comprise a single conformation with 13C chemical shifts that are very similar to those of wild type Aβ1-42 fibrils. These results suggest that E22G-Aβ1-42 fibrils have a structure similar to that of wild type Aβ1-42.

POSTER 018

Studies of seeded Full-Length Tau Filaments by 19F NMR spectroscopy

Presenter: Kristine Kitoka (Latvian Institute of Organic Synthesis)
All Authors: Kristine Kitoka (Latvian Institute of Organic Synthesis); Christina Monnie (Department of Structural Biology and Pittsburgh Center for HIV Protein Interactions, University of Pittsburgh School of Medicine); Angela Gronenborn (Department of Structural Biology, University of Pittsburgh School of Medicine); Kristaps Jaudzems (Latvian Institute of Organic Synthesis)

The microtubule-associated protein tau forms disease-specific filamentous aggregates across several neurodegenerative diseases. Recently, cryo-EM studies revealed that a truncated tau fragment dGAE (I297-E391) can form AD and CTE filaments in vitro. Even

though dGAE provides a more biologically relevant route to generate disease-relevant tau filaments in vitro, tau deposits present in the brain are mostly formed by the full-length protein.

Here, we use tau fragment dGAE to seed the formation of full-length tau filaments. To investigate the rigid core and the fuzzy outer coat interactions, we incorporated 19F labeled amino acids into full–length tau with high efficiency by cell-free protein synthesis. Obtained filaments will be studied by 19F MAS NMR spectroscopy and other complementary biophysical methods.

Biomolecular Solution NMR (019-064)

POSTER 019

19F-qNMR-Assisted Structural Elucidation of Ligand-Free GPCR-G Protein Intermediate Complex

Presenter: Libin Ye (University of South Florida)
All Authors: Libin Ye (University of South Florida); Maxine Bi (UCSF); Xudong Wang (University of South Florida); Jinan Wang (UNC); Wenkai Sun (University of South Florida); Natasha Jaiswal (University of South Florida); Yinglong Miao (UNC); Victor Ayo Adediwura (UNC); Yifan Cheng (UCSF)

Despite the cryo-EM revolution leading to the resolution of over 500 GPCR-Gαβγ complex structures, these snapshots primarily capture the fully activated end-state. Consequently, a comprehensive understanding of the conformational transitions during GPCR activation and the roles of intermediates in signal transduction remains elusive. Herein, creation of an intermediate-state-trapped mutant, guided by 19F quantitative NMR (19F-qNMR) and Molecular Dynamics (MD) simulations1, facilitated sampling of a homogeneous intermediate state in the adenosine A2A receptor (A2AR). This approach yielded a high-resolution cryo-EM structure of intermediate ligand-free (apo) GPCR-Gαβγ complex, via the strategy of blocking conformational transition to the fully activated state. This advancement fills critical structural gap of intermediate complex in the course of GPCR signaling.

POSTER 020

Quantitative NMR analysis of the mechanism and kinetics of chaperone Hsp104 action on amyloid-β42 aggregation and fibril formation

Presenter: Shreya Ghosh (National Institutes of Health)
All Authors: Shreya Ghosh (National Institutes of Health); Vitali Tugarinov (National Institutes of Health); G. Marius Clore (National Institutes of Health)

Hsp104 is a unique chaperone prevents protein aggregation as well as reorders aggregated proteins like fibrils. While the ability of Hsp104 to prevent fibrilization is common knowledge, yet the pathway, mechanism, and kinetics associated with the activity remains unanswered. To this end, I have used a combination of NMR along with imaging techniques of EM and AFM to show how Hsp104 prevents fibrilization of amyloid beta-42, the main constituent of amyloid plaques found in the brains of individuals with Alzheimer's disease. We observed that Hsp104 binds reversibly to sparsely populated AB-42 seeds that are present in nanomolar concentrations with very high affinity, thereby completely inhibiting the on-pathway fibrilization at substoichiometric ratios of Hsp104 to AB-42 monomers.

POSTER 021

Elucidating the Biophysical Mechanisms of Pharmaceutically Relevant Excipient-Fab Interactions

Presenter: Anupreet Kaur (Institute for Bioscience and Biotechnology Research, University of Maryland and National Institute of Standards and Technology, Rockville, MD 20850)
All Authors: ANUPREET KAUR (Institute for Bioscience and Biotechnology Research, University of Maryland and National Institute of Standards and Technology, Rockville, MD 20850); Frank Delaglio (Institute for Bioscience and Biotechnology Research, University of Maryland and National Institute of Standards and Technology, Rockville, MD 20850); John P. Marino (Institute for Bioscience and Biotechnology Research,University of Maryland and National Institute of Standards and Technology, Rockville, MD 20850); Robert Brinson (Institute for Bioscience and Biotechnology Research,University of Maryland and National Institute of Standards and Technology, Rockville, MD 20850)

Monoclonal antibodies (mAb) are the primary biologic platform for drug development due to their high clinical target specificity and developability. Formulation of mAbs involves an extensive excipient selection process based on various trials and biophysical experiments. While these trials often successfully identify storage conditions that confer greater drug substance stability, there remains a lack of in-depth understanding of how excipients exert their stabilizing affects. Here, using the Fab domain derived from the NISTmAb, we report relaxation measurements to characterize the conformational changes of NIST-Fab in various pharmaceutically relevant formulations. Our investigation elucidates the biophysical mechanisms of mAb/excipient interactions. Understanding these underlying mechanisms will enable the streamlining of formulation development and manufacturing, helping to ensure a stable, efficacious, and safe therapeutic.

POSTER 022

Solving and Targeting 5_SL5 - the Translational Start Site of SARS-CoV-2

Presenter: Klara Mertinkus (Goethe University-Frankfurt)
All Authors: Klara Mertinkus (Goethe University-Frankfurt); Andreas Oxenfarth (Goethe University-Frankfurt); Christian Richter (Center for Biomolecular Magnetic Resonance (BMRZ)); Andreas Schlundt (Goethe University-Frankfurt); Anna Wacker (Goethe University-Frankfurt); Julia Wirmer-Bartoschek (Goethe University-Frankfurt); Harald Schwalbe (Goethe University-Frankfurt)

When the Covid-19-pandemic hit humanity, researchers eagerly got to work on the investigation of the different tools used by the virus. Structural assessments of those can lead to opportunities to impact or suppress the viruses' functions. In this work we wrap up the active studies about the structure of the large RNA construct 5_SL5 by solution NMR.

SARS-CoV-2 is from the Beta-Coronavirus family, which have a large, linear, single-stranded RNA genome. The UTRs at the genomic 5'- and 3'-ends provide function by often forming conserved elements. They are of high interest to characterize, as they are potentially involved in RNA-based regulatory functions as well as protein-RNA or RNA-RNA interactions for other cellular or viral purposes.

POSTER 023

Membrane Mimetic Systems Modulate GPCR Energy Landscapes

Presenter: Arka Prabha Ray (University of Florida)
All Authors: Arka Prabha Ray (University of Florida); Naveen Thakur (University of Florida); Beining Jin (University of Florida); Nessa P. Afsharian (University of Florida); Zhan-Guo Gao (NIDDK); Kenneth A. Jacobson (NIDDK); Matthew Eddy (University of Florida)

Utilizing variable-temperature 19F-NMR, we investigate how different membrane mimetics impact the conformational dynamics of the human A2A Adenosine Receptor (A2AAR), a representative G protein-coupled receptor (GPCR). We show that while population of active A2AAR increases with increasing temperature in lipid nanodiscs, strikingly different responses are observed in detergent micelle preparations. Our data also show that A2AAR complexes with partial agonists exhibit a unique conformational state in lipid nanodiscs. Quantification of relative conformer populations show NMR data recorded for A2AAR in lipid nanodisc preparations closely correlates with cell signaling data. The presented data provide clear evidence of the influence of the membrane environment on GPCR conformational dynamics, underscoring the significant influence of the properties of membrane environments on receptor function.

POSTER 024

RAPID Acquisition of NMR Experiments at High and Ultra-High Magnetic Fields Using AI Designed RF Pulses

Presenter: Manu Veliparambil Subrahmanian (Department of Biochemistry, Molecular Biology & Biophysics)

All Authors: Gianluigi Veglia (Department of Biochemistry, Molecular Biology & Biophysics, and Department of Chemistry); Manu Veliparambil Subrahmanian (Department of Biochemistry, Molecular Biology & Biophysics)

The Longitudinal Relaxation Enhanced (LRE) experiments, crucial for rapid NMR data acquisition, face a challenge with the limited bandwidth of SOFAST/BEST techniques, leading to uneven excitation in proteins at high magnetic fields. We addressed this by using GENETICS-AI software to design novel band-selective RF pulses, enhancing bandwidth and sensitivity, especially in HMQC and TROSY-HSQC experiments. Demonstrated on a 22 kDa Raf Kinase Inhibitory Protein (RKIP) with a 900 MHz spectrometer, our RApid PulsIng broadband (RAPID) approach achieved clear visibility of peaks near water signals (5.5 ppm) and in the 11-12 ppm range, indicating superior peak resolution across a wide chemical shift spectrum.

POSTER 025

Solving the NMR solution structures of two elements 5_SL5a and 5_SL5b as part of the translational start site of SARS-CoV-2

Presenter: Andreas Oxenfarth (Goethe University-Frankfurt)
All Authors: Andreas Oxenfarth (Goethe University-Frankfurt); Klara Mertinkus (Goethe University-Frankfurt); Christian Richter (Center for Biomolecular Magnetic Resonance (BMRZ)); Anna Wacker (Goethe University-Frankfurt); Julia Wirmer-Bartoschek (Goethe University-Frankfurt); Harald Schwalbe (Goethe University-Frankfurt)

The Covid-19-pandemic, which slowed done the world drastically for more than two years, showed the need for understanding the function of RNA viruses. SInce structure and function are often interlinked in nature, the need for RNA structures arose. Here we show the structures of two subconsturcts of the largest RNA element in the 5'-UTR namely 5_SL5a and 5_SL5b. The overall 4-way-junction RNA 5_SL5 is of great interest since it is the largest RNA in the 5'-UTR and contains the AUG start codon of ORF1a/b. In addition, we analyzed the two most often occuring mutations of VoCs for both constructs. To achieve this we used a combination of NOESYs, direct detection of carbon and nitrogen experiments as well as RDCs.

POSTER 026

NMR Relaxation Reveals Changes in Tau Dynamics in Biomolecular Condensates in Link with its Aggregation

Presenter: Anton Abyzov (German Center for Neurodegenerative Diseases)
All Authors: Anton Abyzov (German Center for Neurodegenerative Diseases); Christian F. Pantoja (German Center for Neurodegenerative Diseases); Maria-Sol Cima-Omori (German Center for Neurodegenerative Diseases); Markus Zweckstetter (German Center for Neurodegenerative Diseases)

Intrinsically disordered protein Tau plays important roles in human neuronal cells, including assembly and stabilization of the microtubules. Tau is involved in multiple neurodegenerative diseases, including Alzheimer's disease, where it undergoes irreversible aggregation. As many IDPs, Tau forms biomolecular condensates in living cells through the liquid-liquid phase separation (LLPS). The LLPS of Tau is believed to be important for its physiological activities and its pathological aggregation. In biomolecular condensates, increased crowding and presence of intermolecular contracts slows down IDP motions, affecting thermodynamics and kinetics of reactions involving IDPs. In this work, we measured 15N NMR relaxation to study the impact of Tau LLPS on its picosecond-to-nanosecond timescale motions, its intramolecular and intermolecular contacts and possible connection thereof to Tau aggregation.

POSTER 027

Dynamics of A-Minor Interactions in the Bacterial Ribosome

Presenter: Christian Steinmetzger (Uppsala University)
All Authors: Christian Steinmetzger (Uppsala University); Hampus Karlsson (Karolinska Institute); Carolina Fontana (Karolinska Institute); Judith Schlagnitweit (Karolinska Institute); Emilie Steiner (Karolinska Institute); Sarah Friebe Sandoz (Karolinska Institute); Natalia Galindo Riera (Karolinska Institute); Magdalena Riad (Karolinska Institute); David Kosek (Karolinska Institute); Ieva Berzina (Karolinska Institute); Chandra S. Mandava (Uppsala University); Martin Hällberg (Karolinska Institute); Suparna Sanyal (Uppsala University); Emma R. Andersson (Karolinska Institute); Katja Petzold (Uppsala University)

Conformational exchange in the apical region of E. coli ribosomal RNA helix 44 (h44) was studied using 1H, 13C and 15N R1ρ relaxation dispersion NMR. Two adenosines in the central bulge, which are normally oriented to engage in an A-minor interaction with the neighboring helix 8, were found to be sequestered into an intrahelical conformation. In silico screening based on 3D motif classification and secondary structure prediction revealed that similar structural switches of A-minor interactions are possible at various sites throughout the two subunits of the ribosome. RNA mutants that trap the switching equilibrium in a particular state were observed to interfere with bacterial growth. Cryo-EM structures of wild-type and mutant ribosome provide insight into the underlying mechanism.

POSTER 028

Deuterium Spin Relaxation of Fractionally Deuterated Ribonuclease H using Paired 475 MHz and 950 MHz NMR Spectrometers

Presenter: Arthur Palmer (Columbia University)
All Authors: Shibani Bhattacharya (New York Structural Biology Center); Kristen M. Varney (University of Maryland School of Medicine); Tassadite Dahmane (New York Structural Biology Center); Bruce A. Johnson (Advanced Science Research Center, CUNY Graduate Center); David J. Weber (2University of Maryland School of Medicine); Arthur Palmer (Columbia University)

Deuterium spin relaxation of deuterated methyl isotopomers is a powerful approach for investigation of conformational dynamics of proteins by solution-state NMR spectroscopy. Deuterium spin relaxation data recorded on NMR spectrometers with static magnetic field differing by a factor of two have the special property that the double-quantum spectral density function at the lower magnetic field is identical to the single-quantum spectral density function at the higher magnetic field. The present work takes advantage of this relationship by using deuterium relaxation rate constants recorded at static magnetic fields of 475 and 950 MHz, together with data at 700 MHz, in a model-free analysis of side-chain dynamics of fractionally deuterated and alternate carbon-13 labeled Escherichia coli ribonuclease HI.

POSTER 029

Exploiting 19F NMR in a multiplexed assay for small GTPase activity

Presenter: Fatema Bhinderwala (Univeristy of Pittsburgh)
All Authors: Fatema Bhinderwala (Univeristy of Pittsburgh); Angela Gronenborn (University of Pittsburgh School of Medicine)

Small GTPases (SmG) are a family of proteins with about 150 members divided into five subfamilies: Ras, Rho, Arf, Rab, and Ran-GTPases. We investigated 4-, 5-,6-, and 7-fluoro tryptophan containing SmGs to identify a robust nucleotide binding reporter in vitro and cellular milieu. 19F 1D spectra provide distinct resonances for GDP- or GTP-bound 19F-labeled SmGs for three different SmGs: RhoA, KRas, and Rac1. Using this position-specific tryptophan labeling strategy, we can assess their activity in a multiplexed fashion. Employing established inhibitors, we illustrate how this multiplexed system permits screening for SmG and nucleotide-specific ligands under true physiological conditions

POSTER 030

Conformational Dynamics in Enzyme Activation and Disease

Presenter: Irina Bezsonova (UCONN Health)
All Authors: Emilie Korchak (UCONN Health); Dmitry M. Korzhnev (UCONN Health); Irina Bezsonova (UCONN Health)

USP7 is a deubiquitinating enzyme overexpressed in several human malignancies. Its mutations cause a neurodevelopmental disorder Hao-Fountain syndrome. USP7 is a dynamic enzyme with a complex regulatory mechanism. Its understanding is key to the development of treatments for the disease.

Solution NMR spectroscopy provided a unique insight into the mechanism of regulation of USP7 activity. Using CPMG relaxation dispersion experiments we show that the catalytic domain of the enzyme exists in a dynamic equilibrium between inactive and low-populated active conformations. Functional analysis of Hao-Fountain syndrome-causing mutations revealed a subset of variants that result in hyperactivation of the enzyme. Remarkably, the hyperactive mutants dramatically enhanced the conformational dynamics of USP7 and provided a mechanistic link between dynamics and enzyme activity.

POSTER 032

Solution NMR of Liquid-liquid Phase Separation in Native Major Ampullate Spider Silk Proteins Indicates Arg and Tyr Influence Pre-assembly

Presenter: Hannah Johnson (San Diego State University)

All Authors: Hannah Johnson (San Diego State University); Andy T. Chau (San Diego State University); Anikin Rae Domingo (San Diego State University); Julian E. Aldana (San Diego State University); Hieu Nguyen (San Diego State University); Alexia de Loera (San Diego State University); Lado Shapakidze (San Diego State University); David Onofrei (San Diego State University); Gregory P. Holland (San Diego State University)

Liquid-liquid phase separation (LLPS) is a reversible process in which intrinsically disordered proteins concentrate together to form dense liquid droplets. Important for transient biological processes like stress response, liquid droplets sometimes aggregate and form fibrils implicated in neurodegenerative disease. During biomaterials assembly, LLPS is thought to be a mechanism for controlled protein aggregation and hierarchical organization. In spider silk, coacervation is induced by potassium phosphate and is known to improve fiber toughness; however, the role of phosphate in pre-ordering the protein is not yet understood. To investigate LLPS in native silk proteins, we collected both structural and relaxation measurements using solution NMR. The results indicate ordering of Tyr and Arg residues, though random coil secondary structure is maintained overall.

POSTER 033

Binding and Functional Folding (BFF): A Physiological Framework for Studying Biomolecular Interactions and Allostery by NMR

Presenter: David Weber (University of Maryland, School of Medicine)

All Authors: Xinhao Zhuang (University of Maryland); Brianna D. Young (University of Maryland); Brianna K. Costabile (Columbia University); Riya Samanta (University of Maryland); Spiridon Sevdalis (University of Maryland); Kristen M. Varney (University of Maryland); Filippo Mancia (Columbia University); Silvina Matysiak (University of Maryland); Eaton Lattman (University of Maryland); David Weber (University of Maryland); Owen Eby (Mount St. Mary's University)

In this presentation, mechanisms of allostery for EF-hand Ca2+-binding proteins (CBPs), are coalesced into an empirical "binding and functional folding (BFF)" physiological framework based on NMR data and computational approaches. The BFF framework is introduced with two straightforward BFF types for proteins (type 1, concerted; type 2, stepwise) and considers how homologous and nonhomologous amino acid residues of CBPs and their effector protein(s) evolved to provide allosteric tightening of Ca2+ and simultaneously determine how specific and relatively promiscuous CBP-target complexes form as both are needed for proper cellular function, including specificities of protein-protein interactions observed in cases of the type 1 S100 family members.

POSTER 034

Structural Studies of the hnRNP A18 Protein and Development of Small Molecule Inhibitors

Presenter: Kristen Varney (University of Maryland, School of Medicine)

All Authors: Kristen M. Varney (University of Maryland); Katherine Coburn (University of Maryland); Wenbo Yu (University of Maryland); Braden Roth (University of Maryland); Alexander MacKerell (University of Maryland); France Carrier (University of Maryland); David Weber (University of Maryland)

To understand how the A18 (hnRNP A18) protein regulates translation initiation a series of structure, function and inhibition studies were undertaken, including small molecule drug development. A18 (hnRNP A18) is overexpressed in several solid tumors, as compared to normal tissues, and promotes tumor growth via the coordination of mRNA transcripts associated with pro-survival genes. Towards blocking this complex in cancer, we are studying the structure and dynamic properties of hnRNP A18 and using these data together with CADD techniques to engineer inhibitors that are highly efficacious and specific for targeting A18 versus other RNPs. Additionally, NMR data for the structured and unstructured components of A18 will be illustrated as needed to begin examining the translation initiation complex and its regulation.

POSTER 035

NMR studies of calcium ion dissociation from the cell-binding component of Clostridioides difficile binary toxin induces conformational exchange necessary for membrane binding and pore formation

Presenter: Spiridon Sevdalis (University of Maryland, Baltimore)

All Authors: Spiridon Sevdalis (University of Maryland, Baltimore); Kristen Varney (University of Maryland, School of Medicine); Dinendra L. Abeyawardhane (University of Maryland School of Medicine); Mary Cook (University of Maryland School of Medicine); Kaylin A. Adipietro (University of Maryland School of Medicine); Raquel Raquel Godoy-Ruiz (University of Maryland School of Medicine); Izza F. Nawaz (University of Maryland School of Medicine); Alejandro X. Spittel (University of Maryland School of Medicine); Vitalii I. Silin (University of Maryland - Institute for Bioscience and Biotechnology Research); Amedee des Georges (Structural Biology Initiative, CUNY Advanced Science Research Center); Edvin Edwin Pozharski (University of Maryland School of Medicine); David Weber (University of Maryland School of Medicine)

Clostridioides difficile infection (CDI) is a major pathogenic threat in the United States. Of the toxins secreted by C. difficile, CDT is present in the most hypervirulent C. difficile strains. CDT consists of two proteins: CDTa, the enzymatic subunit, and CDTb, the translocase/pore forming subunit. 2D [1H, 15N]-HSQC NMR experiments revealed receptor binding domain 1 (RBD1) of CDTb folds in the presence of Ca2+. Informed by this discovery, a novel mechanism for toxin delivery will be presented. Additional NMR, structural, biophysical, and cellular studies were done to show that the dissociation of Ca2+ from RBD1 triggers CDTb pore formation in membrane bilayers during endosomal maturation, allowing for the export of CDTa into the cytoplasm and facilitating cellular.

POSTER 036

Synthesis of 13C-Methyl-Labeled Amino Acids and their Incorporation into Proteins in Mammalian Cells

Presenter: Andrew Hinck (University of Pittsburgh)

All Authors: Matteo Borgini (Augusta University); Łukasz Wieteska (Francis Crick Institute); Troy Krzysiak (University of Pittsburgh); Peter Wipf (University of Pittsburgh)

Carbon-13 methyl labeling has transformed applications of solution-based NMR spectroscopy and allowed the study of much larger and complex proteins than previously possible with 15N labeling. Methyl labeling procedures are well-established in bacteria, and more recently in yeast, but these expression systems are not suitable for production of some human proteins. To extend the benefits of methyl labeling to difficult to express human proteins, we developed versatile synthetic routes to 13C methyl-labeled amino acids and demonstrated efficient incorporation using suspension-cultured HEK293 cells. Production costs are minimal and the labeling efficiciency is near 100%. In summary, we demonstrate the cost-effective production of methyl-labeled proteins in mammalian cells by incorporation of 13C methyl-labeled amino acids generated by a versatile synthetic route.

POSTER 037

Opening-Dissociation Equilibrium of the E. coli β-Clamp Protein

Presenter: Dmitry Korzhnev (UCONN Health)
All Authors: Dmitry Korzhnev (UCONN Health)

The ring-shaped E. coli β-clamp protein is an 81 kDa head-to-tail homodimer, which serves as a processivity factor for DNA polymerases and a binding platform for DNA replication, repair, and damage response proteins. We used a combination of amide and methyl TROSY NMR, AUC, and SAXS to probe a 3-state opening-dissociation equilibrium of the β-clamp in solution, in which the clamp populates the closed-dimer, open-dimer, and monomer states. Together, our biophysical data suggest that the β-clamp transitions between the closed and monomeric states without residing in an open intermediate, pointing to an induced fit opening mechanism of the β-clamp opening by the clamp-loader complex, which promotes the opening transition of the closed clamp during its loading onto DNA.

POSTER 038

Quantitative NMR using water as internal calibrant

Presenter: Bosong Xiang (Bayer)
All Authors: Bosong Xiang (Bayer)

A new quantitative nuclear magnetic resonance (qNMR) method, called qNMRw, using water as the internal calibrant has been developed. Its principles, procedures, calculations, and test results are presented here. It is shown to avoid the difficulties created by moisture present in other reference materials. High precision and accuracy can be achieved with qNMRw. The method can be used for analyzing technical materials, herbicide formulation products, and other types of chemical samples. It can also be used to measure the purity and concentration of materials to be used as quantitation calibrants.

POSTER 039

Measuring protein 15N relaxation with pure shift HSQC experiments

Presenter: Jorge Moreira (The University of Manchester)
All Authors: Jorge Moreira (The University of Manchester); Mathias Nilsson (The University of Manchester); Gareth A. Morris (The University of Manchester); Alexander Golovanov (The University of Manchester)

The measurement of backbone 15N nuclear spin longitudinal and transverse relaxation time constants, T1 and T2, and the steady-state heteronuclear Nuclear Overhauser effect (ss-hetNOE) provides important relaxation parameters used to investigate local, residue-level, motion of proteins on the picosecond to nanosecond timescale. In conventional relaxation-encoded 1H-15N HSQC experiments, signal overlap can hinder the extraction of the signal intensities needed to measure relaxation. Here, new relaxation-encoded 1H-15N HSQC experiments are implemented with real-time band selective homonuclear decoupling (BASHD). These pure shift methods give ultra-high resolution 2D spectra at no extra cost in experiment time and with no detriment to the measured relaxation parameters, as shown for 15N-ubiquitin.

POSTER 040

PULSAR-AI, a new AI software to generate high-fidelity RF pulses for NMR spectrometers from 60 MHz to 1.2 GHz.

Presenter: Gianluigi Veglia (Department of Biochemistry, Molecular Biology & Biophysics)
All Authors: Manu Veliparambil Subrahmanian (Department of Biochemistry, Molecular Biology & Biophysics); Gianluigi Veglia (Department of Biochemistry, Molecular Biology & Biophysics, and Department of Chemistry, Minneapolis)

We introduce PULSAR-AI, a novel software leveraging an evolutionary algorithm and AI to create RF pulses for NMR, enhancing homogeneity and bandwidth across magnetic fields. Utilizing a 1-million RF pulse library from GENETICS-AI, PULSAR-AI customizes pulses for specific NMR tasks, offering universal 90° and 180° excitations and inversion pulses. Achieving ~1 MHz excitation bandwidth, it simplifies generating RF shapes for NMR systems from benchtop to ultra-high field (1.2 GHz), compatible with various commercial spectrometers.

POSTER 041

The Insights into Common Physiological Metal Ions Interacting with S100A1 versus S100B

Presenter: Xinhao Zhuang (University of Maryland)
All Authors: Xinhao Zhuang (University of Maryland); Brianna Young (University of Maryland School of Medicine); Spiridon Sevdalis (University of Maryland, Baltimore); Dylan Weber (University of Maryland School of Medicine); Zephan Melville (New York Structural Biology Center); Kristen Varney (University of Maryland, School of Medicine); David Weber (University of Maryland School of Medicine)

S100A1 and S100B, two EF-hand calcium binding proteins, play wide and important roles in functions regulated by calcium. This study investigates their interaction with common physiological ions: sodium, potassium, and magnesium. The results indicate that under resting intracellular low calcium concentrations, these three ions probably enhance calcium binding to S100A1 and S100B by stabilizing the apo form of the proteins. Therefore, the importance is highlighted for further study to investigate the interaction between S100 proteins and physiologically relevant ions prior to their target proteins.

POSTER 042

An RNA excited conformational state at atomic resolution

Presenter: Ainan Geng (Columbia University)
All Authors: Ainan Geng (Columbia University); Laura Ganser (Duke University); Rohit Roy (Columbia University); Honglue Shi (Duke University); Supriya Pratihar (Columbia University); David Case (Rutgers University); Hashim Al-Hashimi (Columbia University)

Sparse short-lived RNA conformational states are essential players in cell physiology, disease, and therapeutics, yet determining their 3D structures remains challenging. Combining mutagenesis, NMR, and computational modeling, we determined the 3D structural ensemble formed by a short-lived (~2.1 ms) lowly-populated (~0.4%) state in HIV-1 TAR. Through a strand register shift, the excited state completely remodels the 3D structure of the ground state (RMSD ~7.2 Å), forming a surprisingly more ordered ensemble rich in non-canonical mismatches. The structure impedes the formation of motifs recognized by Tat and the super elongation complex, explaining why this alternative TAR conformation cannot activate HIV-1 transcription. Our ability to determine the structures of fleeting RNA states holds great promise for understanding RNA biology and RNA-targeting therapeutics.

POSTER 043

Conformational Dynamics of NADH in Mixed Binary Solvents Studied by 31P NMR Spectroscopy

Presenter: Jiaqi Lu (NYU)
All Authors: Jiaqi Lu (NYU); Florin Teleanu (New York University); Huijing Zou (New York University); Chengtong Zhang (New York University); Alexej Jerschow (New York University)

Investigating NADH conformational dynamics via 31P NMR spectroscopy in D2O/DMSO-d6 mixtures, we observed distinct solvent-induced conformations. Temperature-sensitive chemical shifts in D2O suggested a dynamic equilibrium between folded and unfolded states, while DMSO-d6 favored an unfolded state. Relaxometry data supported these findings, revealing solvent-dependent changes in NADH's hydrodynamic radius. This study highlights the influence of solvent environment on NADH structure, with implications for its enzymatic role in energy metabolism and potential as a metabolic biomarker.

Keywords: NADH, 31P NMR, solvent effects, conformational dynamics, energy metabolism.

POSTER 044

Strong Coupling's effect on HMQC and HSQC Spectra for Determination of Conformational Motion

Presenter: Darón Freedberg (CBER/FDA)
All Authors: Jasmin Zarb (CBER/FDA); Vivekanandan Subramanian (University of Kentucky); Jeahoo Kwon (CBER/FDA); Hugo van-Ingen (Utrecht University); Darón Freedberg (CBER/FDA)

Chemical shifts determined from HSQC spectra do not match those from HMQC spectra in the presence of conformational motion. This

difference can be used to extract rate constants and thermodynamic parameters when taken at different magnetic fields. However, non-first order coupling may also distort chemical shifts. Therefore, use of HMQC-HSQC differences to study conformational processes may yield inaccurate results when used to extract kinetic and thermodynamic parameters in strongly coupled systems. Here, we examine the effects of strong coupling on HMQC-HSQC chemical shift differences and their consequences for accuracy for 1H-13C and 1H-15N spectra.

POSTER 045

Rapid Characterization of Structural and Behavioral Changes of Therapeutic Proteins by 1H-SOFAST NMR Experiments

Presenter: Xingjian Xu (Merck)
All Authors: Xingjian Xu (Merck); Guilherme Dal Poggetto (Merck); Mark McCoy (Merck); Mikhail Reibarkh (Merck); Pablo Trigo-Mourino (Merck)

Efficient evaluation of the structural and dynamic behavior of therapeutic proteins is critical in drug development. NMR methods, particularly rapid pulsing schemes like SOFAST, have emerged as versatile tools for this purpose. Here, we present modified 1H 1D-SOFAST experiments that provide a fast and reliable approach for characterizing therapeutic proteins. By incorporating specific modifications into the SOFAST pulse sequence, our proposed methods allow for quick estimation of transverse relaxation rates and diffusion coefficients. We demonstrated the versatility of these methods by evaluating a range of samples under different concentrations or buffer conditions, demonstrating their potential to be used in conjunction with other techniques, enabling efficient screening of proteins and biologics in solution.

POSTER 046

Dynamic base pair rearrangement facilitates c-di-GMP riboswitch folding

Presenter: Ji-Yeon Shin (Korea Institute of Science and Technology)
All Authors: Ji-Yeon Shin (Korea Institute of Science and Technology); Seo-Ree Choi (Korea Institute of Science and Technology); Kyeong-Mi Bang (Korea Institute of Science and Technology); Hyun Kyu Song (Korea University); Nak-Kyoon Kim (Korea Institute of Science and Technology)

This study investigates the Vc2 (Vibrio cholerae) riboswitch, focusing on the fold process of its aptamer domain, which is required for recognition of the c-di-GMP. Using NMR spectroscopy and ITC, we discovered that stable folding requires Mg^{2+}, Na^{+}, and K^{+} ions, with Mg^{2+} being essential for pre-folding and K^{+} necessary for G-C interaction and ligand pocket stability. Precise imino-proton assignments by NMR and CLEANEX-PM experiments revealed dynamic exchanges in the P2 helix, demonstrating its role in folding and ligand recognition. It contributes to our understanding of riboswitch behavior and establishes NMR as a powerful tool.

POSTER 047

Structural Investigation of Human U6 snRNA Recognition by The Spliceosome Recycling Factor SART3

Presenter: Kyeong-Mi Bang (Korea Institute of Science and Technology)
All Authors: Kyeong-Mi Bang (Korea Institute of Science and Technology); Iktae Kim (Texas A&M University); So Young An (Seoul National University); Ji-Yeon Shin (Korea Institute of Science and Technology); Hyun Kyu Song (Korea University); Jeong-Yong Suh (Seoul National University); Nak-Kyoon Kim (Korea Institute of Science and Technology)

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 SART3 RRM1 binds to two distinct specific binding interfaces on U6 snRNA bulge.

POSTER 048

Structural Insights into the DNA Binding Mechanism of the Meis1 Transcription Factor Revealed by NMR and MD Simulations

Presenter: Seo-Ree Choi (Korea Institute of Science and Technology)
All Authors: Seo-Ree Choi (Korea Institute of Science and Technology); Yeo-Jin Seo (Gyeongsang National University); Ho-seong Jin (Gyeongsang National University); Hyebin Ahn (gyeongsang national university); youyeon Go (Gyeongsang national university); Kyoung-Seok Ryu (KBSI); Joon-Hwa Lee (Gyeongsang national university); Juyong Lee (Seoul National University); Nak-kyoon Kim (Korea Institute of Science and Technology)

Transcription, the first step of gene expression, involves RNA polymerase copying DNA into mRNA, facilitated by transcription factors (TFs) binding specific DNA sequences. Myeloid ecotropic viral integration site-1 (Meis1), crucial in myeloid leukemia and ovarian cancer, contains a TALE homeobox domain (HD) binding to 5'-TGACA-3' DNA sequence. This study using NMR to analyze Meis1-HD complexes with meisDNA, comparing wild-type and variant meisDNAs via imino proton and HSQC titrations. ITC and 15N relaxation dispersion experiments were also performed to study Meis1-HD DNA binding thermodynamics and kinetics, while MD simulations investigated structural features. This study provides key structural features of structures of the Mesi1-HD-DNA complex and the information about the molecular mechanism of target DNA recognition by the Meis1 transcription factor.

POSTER 049

Interaction between a fluoroquinolone derivative KG022 and RNAs

Presenter: Rika Ichijo (Chiba Institute of Technology)
All Authors: Rika Ichijo (Chiba Institute of Technology); Gota Kawai (Chiba Institute of Technology)

RNA-targeted small molecules are a promising modality in drug discovery. Recently, we found that a fluoroquinolone derivative, KG022, can bind to RNAs with bulged C or G. To clarify the RNA specificity of KG022, we analyzed the effect of the base pair located at the 3' or 5' side of the bulged residue by NMR spectroscopy. For the stable complexes of KG022 with RNA molecules, solution structures were determined. It was found that the fluoroquinolone moiety is located between two purine bases in these complexes. This work provides an important example of the specificity of RNA-targeted small molecules.

POSTER 050

Development of quantitative and predictive thermodynamic model for DNA base pair ensemble

Presenter: Yeongjoon Lee (Columbia University)
All Authors: Yeongjoon Lee (Columbia University); Stephanie Gu (Duke University); Hashim M. Al-Hashimi (Columbia University)

In solution, biomolecules morph through numerous conformations, each with its own probability and lifespan. Cellular cues reshape this conformational landscape, favoring improbable states. The dynamic ensemble offers a statistical mechanical description of energetic penalties determining binding affinities, specificities, and the kinetics of biochemical processes. However, predicting these ensembles from sequence remains challenging, and experimental determination of conformational ensembles is time-consuming and technically demanding. This project aims to develop and test a quantitative and predictive thermodynamic model to determine the relative energetics of DNA base pair conformations. By predicting the conformational ensembles of mismatches and the impact of epigenetic modifications, the model will advance our understanding of the conformational behavior of DNA and its role in biochemical processes.

POSTER 051

Determining structure and dynamics of mini αA-crystallin
Presenter: Collin Sroge (University of California Irvine)
All Authors: Carter Butts (University of California Irvine); Sophie Padilla (University of California Irvine); Jason Zhu (University of California Irvine); Jaewon Suk (University of California, Irvine); Rachel Martin (UC Irvine)
α-crystallins are chaperone proteins that maintain stability and clarity of the eye lens by inhibiting structural β and γ crystallin aggregation. αA crystallin has been shown to maintain chaperone activity when truncated into a 19 amino acid peptide. The chaperone activity is mediated by hydrophobic interactions, yet critical amino acids involved and structural effects of loss of hydrophobic residues is still unknown. I will present solution-state NMR spectra, including TOCSY and NOESY, that will determine the structure and dynamics of mini αA crystallin (MAAC) and alanine variants. This data will then be compared to molecular dynamic simulations.

POSTER 052
Structural Dynamics of Active K-Ras-GTP and its Oncogenic Mutants at the National Gateway Ultrahigh Field NMR Facility
Presenter: Alexandar Hansen (The Ohio State University)
All Authors: Alexandar Hansen (The Ohio State University); Xinyao Xiang (The Ohio State University); Chunhua Yuan (The Ohio State University); Lei Bruschweiler-Li (The Ohio State University); Rafael Bruschweiler (The Ohio State University)
Despite the prominent role of K-Ras protein in many different types of human cancer, major gaps in atomic-level information severely limit our understanding of K-Ras function in health and disease. We report the quantitative backbone structural dynamics of K-Ras by solution NMR spectroscopy of the active state of wild-type K-Ras.GTP and two of its oncogenic P-loop mutants, G12D and G12C, by a novel nanoparticle-assisted spin relaxation method, relaxation dispersion and chemical exchange saturation transfer experiments covering the entire range of timescales from picosecond to milliseconds.

The stability and representative spectra recorded on the recently commissioned 1.2 GHz NMR spectrometer at OSU, the centerpiece of the new National Gateway Ultrahigh Field NMR Center funded by the NSF, will be presented.

POSTER 053
Structural insights into peptide substrate interactions of Src kinase
Presenter: Dan Xie (Eli Lilly and Company)
All Authors: Dan Xie (Purdue University); Yixing Sun (Purdue University); Casey J. Krusemark (Purdue University); Carol B. Post (Purdue University)
Understanding oncogenic kinase substrate recognition has become a priority for enabling drug discovery that targets mutated or overactive tyrosine kinases in cancer. In this study, we exploited solution NMR techniques and biophysical methods to investigate structural patterns of Src kinase peptide and protein substrates recognitions. Chemical shift analysis, paramagnetic relaxation approaches and molecular dynamic simulations together reveal alternative binding modes for different peptide substrates. Mechanistic understanding of substrate interaction of oncogenic protein tyrosine kinases could facilitate new approaches to designing anti-cancer drugs with fewer side effects.

POSTER 054
Investigating the Structure of Ci-βγ Crystallin
Presenter: Matthew Jimenez (University of California, Irvine)
All Authors: Matthew Jimenez (University of California, Irvine); Mina Mozafari (Graduate Student); Megan Alma Rocha (Graduate Student); Rachel Martin (UC Irvine)
The βγ-crystallin superfamily consists of refractive and structural proteins that are found in microbial, invertebrate, and vertebrate organisms. Ci-βγ-crystallins is found in an invertebrate organism, and it contains both refractive and divalent cation-binding properties; while, human lens crystallins have evolved away from these binding sites. Due to these characteristics, Ci-βγ is signified as an evolutionary standpoint between ancestral crystallins and current vertebrate crystallins. The broader goal of this project is to study the evolutionary lineage in the βγ-crystallin superfamily. This will be accomplished by comparing the biophysical properties and structure of Ci-βγ to the vertebrate βγ-Crystallin. Towards this goal, I have monitored Ci-βγ backbone rearrangement during Ca2+ binding and temperature change through 1H-15N HSQCS.

POSTER 055
NMR structure of the aggregate-targeting N1 domain of the bacterial AAA+ disaggregase ClpG
Presenter: Bernd Simon (UConn Health)
All Authors: Bernd Simon (UConn Health); Panagiotis Katikaridis (Center for Molecular Biology of Heidelberg University); Timo Jenne (Center for Molecular Biology of Heidelberg University); Janosch Hennig (Division of Biophysical Chemistry); Axel Mogk (Center for Molecular Biology of Heidelberg University)
We determined the core structure of the N1 domain of the bacterial AAA+ disaggregase ClpG that harbors a Zn2+-coordination site that is crucial for structural integrity and functionality. Structures were calculated in the ARIA1.2/CNS1.2 package with standard simulated annealing protocols using a log harmonic NOE potential for the distance restraints. The mean structure superimposes well with the structure predicted by AlphaFold2. However, the ensemble indicates dynamics that AlphaFold2 cannot predict. We found that conserved hydrophobic N1 residues located on a β-strand are crucial for aggregate targeting and disaggregation activity. Chemical shift titrations with a previously identified as N1 binder in a peptide library screen confirm a complex structure predicted by AlphaFold2 where the peptide is forming and additional antiparallel b-strand.

POSTER 056
Sequence and Context Dependence of Watson-Crick-like G•T/U as Observed by R1ρ Relaxation Dispersion and 19F NMR
Presenter: Or Szekely (Duke University)
All Authors: Or Szekely (Duke University); Qi Zhang (UNC Chapel Hill); Hashim Al-Hashimi (Columbia University)
DNA replication errors account for two thirds of mutations found in human cancers. However, we do not understand what governs the probability and sequence dependence of replicative errors. G•T/U wobble mismatches in B-DNA, A-RNA and RNA:DNA hybrids can form low-abundance Watson-Crick-like conformations through tautomerization or ionization of the bases, having implications in replication, translation and transcription errors. Here, using a combination of R1ρ relaxation dispersion experiments and high throughput 19F NMR pKa screening, we show that dynamics of the anionic conformation is highly sequence dependent, with up to 40-fold variation in propensity. Our study indicates plausible roles for Watson-Crick-like conformations in replication errors and indicates that mutagenic dynamics strongly depend on nucleic acid sequence and structural contexts.

POSTER 057
Controlling base pair conformational equilibrium in DNA hairpins.
Presenter: Serafima Guseva (Columbia University)
All Authors: Serafima Guseva (Columbia University); Or Szekely (Duke University); Akanksha Manghrani (Duke University); Stephanie Gu (Duke University); Mark A. Wilson (University of Nebraska); Hashim M. Al-Hashimi (Columbia University)
Hoogsteen base pair conformation, while transient in standard DNA duplexes, can be found as a dominant state in non-canonical DNA structures like aptamers, G-quadruplexes, and hairpins. Here, we study Hoogsteen-to-Watson-Crick conformational equilibrium in DNA hairpins using IS200/IS608 transposons terminal hairpins, vital for the transposase enzyme recognition preceding DNA cleavage. Using NMR we characterise base pair conformational exchange, with significant Hoogsteen base pair presence of up to 90%. Furthermore, our results suggest that base pair conformational equilibrium is influenced by temperature and apical loop sequence. These findings propose a potential mechanism for regulating protein-DNA interaction

and, by extension, transposase activity through base pair conformational dynamics.

POSTER 058
NMR structure of the kinase inhibitor Palbociclib bound to HIV TAR
Presenter: RAVIKANTH REDDY RAMIREDDY (University of Washington)
All Authors: Ravikanth Reddy Ramireddy (University of Washington); Bhawna Chaubey (University of Washington); Gabriele Varani (University of Washington); Thomas Pavelitz (University of Washington); Venkata Narayana Vidadala (University of Washington); Changyan Tang (University of Washington)
The present work reports NMR structure of a specific complex between Palbociclib and HIV-1 TAR RNA. Palbociclib binds to the TAR with nM affinity with specificity. Palbociclib recognizes a site spanning the UCU bulge and induces formation of a new G36-C29.C24+ base triple and a structure never observed before. The C24 base of UCU bulge is deeply inserted into the major groove of stem I and is in close contact with G36. The aromatic protons of Palbociclib depicts gigantic chemical shift changes and exhibits intermolecular NOEs with G21, A22, G26, U40 and C41 residues. Furthermore, 2′-OH and NH2 groups for all residues of Stem I are observed in NOESY and 1H-15N HSQC indicating the stabilization of the RNA structure.

POSTER 059
Chemically induced partial unfolding of the multifunctional Apurinic/apyrimidinic endonuclease 1
Presenter: Ratan Rai (Indiana University)
All Authors: Ratan K. Rai (Department of Biochemistry and Molecular Biology, Indiana University School of Medicine)
Targeting of the multifunctional enzyme apurinic/apyrimidinic endonuclease I/redox factor 1 (APE1)1 has produced small molecule inhibitors of both its endonuclease and redox activities. While one of the small molecules, the redox inhibitor APX3330, completed a Phase I clinical trial for solid tumors and a Phase II clinical trial for Diabetic Retinopathy/Diabetic Macular Edema, the mechanism of action for this drug has yet to be fully understood. Here, we demonstrate through 1H-15N HSQC NMR studies that APX3330 induces chemical shift perturbations of both surface and internal residues in a concentration-dependent manner, with a cluster of surface residues defining a small pocket on the opposite face from the endonuclease active site of APE1.

POSTER 060
HIV-1 p17 Interactions with Heparan Sulfate
Presenter: Brandon Chavez (California State University, Dominguez Hills)
All Authors: Brandon Chavez (California State University, Dominguez Hills); Rochelle Johnson (California State University, Dominguez Hills); Ana Ponce (California State University, Dominguez Hills); Kenia Vidal (California State University, Dominguez Hills); Stephanie Rauda (California State University, Dominguez Hills); Bradley Heath (California State University, Dominguez Hills); William Omenwu (California State University, Dominguez Hills); Alexander W. Sorum (California Institute of Technology); Linda C. Hsieh-Wilson (California Institute of Technology); Kari Pederson (California State University, Dominguez Hills)
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. Disrupting this interaction could help mitigate the contraction of viral infections. Heparan sulfate interactions are known for signaling growth factors, cell adhesion, and enzymatic catalysts for human cells. The HIV-1 p17 protein, the focus of this study, is one of three HIV-1 proteins known to interact with heparan sulfate proteoglycans. 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 interest for future drug development.

POSTER 061
Cataract Variants of gS-crystallin.
Presenter: Megan Rocha (UCI)
All Authors: Megan Rocha (UCI); Paaras Shah (UCI); Ellen Xu (UCI); Jessica Kelz (UC Irvine); Mina Mozafari (Postdoc); Brenna Norton-Baker (NREL); Rachel Martin (UC Irvine)
The eye lens is a transparent and refractive tissue which is maintained by highly soluble proteins called crystallins that are at high density. Crystallins are a class of extremely long-lived proteins since lens cells lack protein turnover machinery which would scatter light and impair vision. Post translational modifications (PTMs) accrued through the lifetime of the lens damage crystallin solubility and lead to cataract – the leading cause of blindness worldwide. One of the most abundant PTMs found in cataractous lenses is deamidation. Here, I investigate the biophysical effects of multiply deamidating a human lens crystallin. I show that human □S-crystallin has decreased fast dynamics with increasing deamidation while remaining largely similar in their slow dynamics.

POSTER 062
An NMR-Based Fragment Screening Approach to Identify LCAT Modulators
Presenter: Elena Hausmann (National Center for Advancing Translational Sciences)
All Authors: Elena Hausmann (National Center for Advancing Translational Sciences); Samuel A. Kotler (National Center for Advancing Translational Sciences); Savanah L. Saldana-Shumaker (National Center for Advancing Translational Sciences); Maggie G. Seidel (American University); Bianca R. Woodward (American University); Maura Fletcher (American University); Jacob W. Crater (American University); Monika I. Konaklieva (American University); Christopher A. LeClair (National Center for Advancing Translational Sciences)
Atherosclerotic cardiovascular disease (ASCVD), which involves the buildup of cholesterol-containing plaque in the arteries, is the leading cause of death in the world. Lecithin:cholesterol acyltransferase (LCAT) is an enzyme that drives maturation of HDL particles to HDL-C. Low HDL-C levels are associated with increased risk of ASCVD. Novel therapies that improve LCAT function are necessary for the treatment of cardiovascular disease and rare diseases like familial LCAT deficiency. We aim to identify new small molecules that bind LCAT and modulate its activity through NMR-based fragment screening. A curated library of rationally designed fragments was screened against LCAT. Synthetic elaboration of the hits by NMR data will afford a ligand that is, ideally, more potent and selective than the initial fragment.

POSTER 063
NMR Studies of Conformation, Dynamics, and Interaction of Histone Tail Domains in Nucleosomes
Presenter: Wenjun Sun (The Ohio State University)
All Authors: Wenjun Sun (The Ohio State University); Nicole Gonzalez Salguero (The Ohio State University); Olga O. Lebedenko (St. Petersburg State University); Mohamad Zandian (The Ohio State University); Vidhyalakshmi Sridharan (The Ohio State University); Michael Poirier (The Ohio State University); Nikolai R. Skrynnikov (St. Petersburg State University); Christopher Jaroniec (The Ohio State University)
The nucleosome, which is composed of DNA and histone octamer containing two copies each of histones H2A, H2B, H3 and H4. Dynamically disordered H3 and H4 N-terminal tails are key components in chromatin regulation and compaction. We investigate the conformational ensembles and interactions of the H4 tail in nucleosomes by NMR measurements of PREs combined with MD simulations. Collectively, these data enable improved location of the H4 tails and support the notion that H4 tails engage in a fuzzy-complex interaction with nucleosomal DNA. We further extend our studies in conformation, dynamics, and interaction of H3 and H4 histone tails in nucleosomes. Overall, the relaxation data shows that tails in nucleosomes are modulated by ionic strength, and histone protein reader binding.

POSTER 064

Uncovering Specificity hidden within the DNA Double Helix in the form of Sequence-Dependent A-T Hoogsteen Dynamics

Presenter: Akanksha Manghrani (Duke University)
All Authors: Akanksha Manghrani (Duke University); Bei Liu (University of Chicago); Atul Kaushik Rangadurai (University Of Toronto); Hashim Al-Hashimi (Columbia University)

There is growing evidence suggesting that numerous proteins bind DNA in non-canonical Hoogsteen conformation. The propensity to form Hoogsteen bps could therefore impact sequence specificity of DNA-protein interactions. Here using 1H CEST and 13C R1ρ experiments we measured the propensity to form Hoogsteen bps as a function of trinucleotide sequence in 13 out of 16 contexts. Our results suggest that both the propensity and the kinetics of Hoogsteen are modulated by the DNA sequence and sequence-dependent Hoogsteen propensities can contribute ~1.7 kcal/mol variation in specificity in DNA-protein interactions. We also showed that stacking plays an important role in determining Hoogsteen propensities. Taken together, our results highlight the rich reservoir of sequence specificity hidden in DNA dynamics.

Eclectica in Magnetic Resonance (Posters 065-073)

POSTER 065

Boosting 19F-NMR Sensitivity via Steady State Free Precession (SSFP) to Assess Perfluorinated Compounds in Complex Biological and Environmental Matrices

Presenter: Flavio Vinicius Crizostomo Kock (University of Toronto Scarborough)
All Authors: Flavio Kock (University of Toronto Scarborough); Jeremy Gauthier (University of Toronto); Katelyn Downey (University of Toronto Scarborough); Ronald Soong (University of Toronto Scarborough); Tiago Moraes (University of São Paulo); Derek Muir (Environment and Climate Change Canada); Scott Mabury (University of Toronto); Luiz Colnago (EMBRAPA Instrumentation); Andre Simpson (University of Toronto Scarborough)

Per- and polyfluorinated alkyl substances (PFAS) include many classes of fluorinated molecules which are utilized as surfactants, in firefighting foams, and as non-stick hydrophobic coatings. PFAS are routinely found in biological matrices such as urine and blood, as well as in numerous environmental matrices such as soil and water. In this work, steady state free precession (SSFP) protocols are explored to assess PFAS contaminants in solution. The results show an increase up to 20 times in signal-to-noise ratio (SNR) over the standard 19F-NMR spectrum. 19F-SSFP NMR holds promise in studies addressing the identification, quantification, and bioaccumulation of persistent contaminants in highly complex environmental and biological matrices, such as soils, blood and living organisms.

POSTER 066

Investigating a purported connection between DNP and Inertial Mass

Presenter: Mark Sokol (www.falconspace.org www.altpropulsion.com)
All Authors: mark sokol (www.falconspace.org www.altpropulsion.com); Fredrick Alzofon (deceased) (Cal Berkley, Boeing)

In 1981 Fredrick Alzofon published a paper "anti gravity with present technology" which outlines a connection between DNP and inertial mass shielding which would revolutionize science and technology.

In 1994 he ran this experiment and observed an %80 weight reduction in a 1 gram aluminum sample.

At Falcon Space we are investigating these claims with a skeptical scientific mindset using X-band pulsed DNP methodology based on the book by C.D Jeffries Dynamic Nuclear Orientation.

To date our best result recorded was 17.8% weight reduction.

Our experimental setup, methodology, sample choice etc will be demonstrated in the hopes that ENC attendees will be able to collaborate with us on this potentially groundbreaking discovery with massive industrial implications.

POSTER 067

Design of adiabatic excitation and refocusing for use with single-sided magnetic resonance relaxometers

Presenter: Sydney Sherman (MIT)
All Authors: Sydney Sherman (MIT); David Korenchan (Athinoula A. Martinos Center for Biomedical Imaging); Matt Rosen (MGH/Martinos Center); Michael Cima (Massachusetts Institute of Technology)

Highly inhomogeneous B0 and B1 fields present a challenge for the acquisition of robust and accurate signal with single-sided magnetic resonance (MR) relaxometers. We aim to design and implement linear-frequency swept adiabatic pulses for use with low-field single-sided MR relaxometers to increase both acquired signal and signal accuracy. Excitation and composite refocusing chirp pulses were designed to meet the adiabatic condition using the Spinach MATLAB library. The designed pulses were tested in a Carr-Purcell-Meiboom-Gill sequence with B0 and B1 field maps from a single-sided relaxometer as system inputs. We demonstrate a simulated increase of >430% in signal intensity and 25% decrease in percent error of T2 decay fit when compared with the performance of hard pulses.

POSTER 068

13C-1H Couplings Detected with Low-Field NMR at Natural Abundance Using a Synchronized Echo Pulse Sequence

Presenter: Stephen DeVience (Scalar Magnetics)
All Authors: Stephen DeVience (Scalar Magnetics); Matt Rosen (MGH/Martinos Center)

Synchronized Echo (SyncE) pulse sequences enable spectroscopy at low magnetic fields where there is no chemical shift resolution. To be detectable, the target molecule must have at least two coupled protons with different chemical shifts. However, while using solvents that should be "SyncE-silent," such as acetone, we noticed a series of unexpected amplitude dips. These arise from the 2.2% of molecules with a 13C isotope in one of the methyl groups, which breaks magnetic equivalence of the protons. Similar signals were measured for a series of molecules with two or more methyl groups attached to a central atom. With further refinement, this technique might be useful for studying rotational dynamics and relaxation properties of the methyl groups in symmetric molecules.

POSTER 069

Earth-Field and High-Field 1H Relaxation for Real-Time Detection of Free-Radical Formation

Presenter: Aude Sadet (ELI-NP/IFIN-HH)
All Authors: Alexandru Topor (Biophysics and Biomedical Applications Lab, ELI-NP, Extreme Light Infrastructure - Nuclear Physics, IFIN-HH); Aude Sadet (ELI-NP/IFIN-HH); Mihai Adrian Voda (Biophysics and Biomedical Applications Lab, ELI-NP, Extreme Light Infrastructure - Nuclear Physics, IFIN-HH); Arnaud Comment (Cancer Research UK Cambridge Institute, University of Cambridge); Paul Vasos (ELI-NP/IFIN-HH)

Real-time detection methods for following the course of redox reactions can be useful to understand the mechanism of therapeutic approaches. We demonstrate that water 1H relaxation time constants in Earth's magnetic field detect the formation of oxidation reaction products in real time. With our proposed approach, the generation of intermediate *OH free radicals can be detected using spin-traps. In high magnetic fields, 1H-based long-lived states (LLS) and coherences (LLC's) of endogenous antioxidants (glutathione) were used to the same effect. We demonstrate that sensitivity can be enhanced for both high-field and Earth-field experiments using dissolution-DNP. The translation of these methods to radiobiology settings to follow free-radical formation by ionizing radiation with different dose-rates will be discussed.

POSTER 070

Selective excitation of exchanging systems in magnetic resonance

Presenter: Jacob Lindale (Duke University)

All Authors: Jacob Lindale (Duke University); Warren Warren (Duke University)

Nuclear Magnetic Resonance (NMR) spectroscopy is pivotal in probing structural dynamics, offering critical spatial and temporal resolution. The analysis of intricate systems faces challenges from spectral crowding, compelling the use of time-intensive multidimensional experiments. Overcoming these limitations, we introduce exact master equations for chemical exchange in conventional approaches, boosting the efficiency of theoretical simulations. Employing these equations, novel methods are developed to selectively excite dynamic components in exchanging systems while suppressing static elements. This strategic approach retains up to half the conventional signal and accommodates exchange rates across four orders of magnitude. Guided by computational models, these innovations push the boundaries of NMR spectroscopy, enabling precise exploration of molecular dynamics in larger, intricate systems.

POSTER 071

Two surprises with hyperpolarized nuclear spins in solids

Presenter: Ashok Ajoy (UC Berkeley)
All Authors: Ashok Ajoy (UC Berkeley)

We report on two complementary experiments that reveal unexpected and non-intuitive behavior in hyperpolarized nuclear spins in solids, with applications to quantum sensing and nanoscale spin imaging. First, we show unexpected, and remarkably long transverse spin lifetimes for hyperpolarized 13C spins in diamond exceeding T2'=800s at 100K, a factor of >600,000-fold extension over the FID time (T2*=1.5ms). This opens avenues for continuously interrogated AC magnetic field sensors with these spins. Second, we generate tunable polarization spin textures in the nuclear spins surrounding a electron, and demonstrate an approach to "stabilize" the texture, immune to spin diffusion, for multiple minutes. The formed texture spans multiple nanometers and encompasses hundreds of nuclear spins.

POSTER 072

Spin 3/2 NQR Rabi Frequencies: Powder Vs. Single Crystal

Presenter: Ritik Modi (George Mason University)
All Authors: Ritik Modi (George Mason University); Karen L. Sauer (George Mason University)

NQR, done at zero-field, relies on the intrinsic electric field gradient EFG at a quadrupolar nucleus for signal. The relative experimental simplicity and minimal amount of equipment make it an attractive technique for studying internal structure and dynamics in a solid. However, for spin-3/2 nuclei, with only one detectable frequency, the full EFG cannot be determined from frequency alone. Rather this information can be teased out from spectral lineshape for a powder or goniometry measurements of a single crystal in a magnetic field; for large fields the latter technique becomes quite accurate, but also costly. We demonstrate an alternate zero-field technique in which goniometry measurements are made with respect to RF excitation, comparing powder and single crystal samples of KClO3.

POSTER 073

Extended Rotating Frame Lifetimes of 13C in NV Diamonds

Presenter: Kieren Harkins (UC Berkeley)
All Authors: Kieren Harkins (UC Berkeley); Cooper Selco (UC Berkeley); David Marchiori (UC Berkeley); Christian Bengs (UC Berkeley); Samantha Breuer (UC Berkeley); Emanuel Druga (UC Berkeley); Liang Tan (Lawrence Berkeley National Lab); Marin Bukov (Max Planck Institute); Yi-qiao Song (Massachusetts General Hospital); Jeffrey Reimer (UC Berkeley); Ashok Ajoy (UC Berkeley)

An inherent challenge facing the use of nuclear spins in quantum systems is the limited spin coherence arising from rapid relaxation mechanisms. This work demonstrates prolonged rotating frame lifetimes of 13C spins in Nitrogen-Vacancy (NV) diamonds. We have developed a novel NMR instrument capable of accessing a wide range of magnetic fields and temperatures. Using this instrument and a pulsed spinlock control sequence, we measure decay profiles of the nuclear spins with high fidelity. Introducing a transmitter offset tilts the axis of the rotating frame, allowing for access to certain protected spin angles, such as the magic angle and extending lifetimes. A future direction of this work will be controlling spin lifetimes as a function of temperature.

Hyperpolarization Methodologies (Posters 074-115)

POSTER 074

Rational Design of Dinitroxide Polarizing Agents for Dynamic Nuclear Polarization to Enhance Overall NMR Sensitivity

Presenter: Ran Wei (Ecole polytechnique federale de Lausanne (EPFL))
All Authors: Amrit Venkatesh (National High Magnetic Field Laboratory); Gilles Casano (Aix Marseille University); Ran Wei (Ecole polytechnique federale de Lausanne (EPFL)); Yu Rao (EPFL); Hugo Lingua (Aix Marseille University); Hakim Karoui (Aix Marseille University); Maxim Yulikov (ETH Zurich); Olivier Ouari (Aix Marseille University); Lyndon Emsley (EPFL)

We evaluate the overall sensitivity gains provided by eighteen state-of-the-art nitroxide biradicals for magic angle spinning (MAS) Dynamic nuclear polarisation (DNP) solid-state NMR at 9.4 T and 100 K, including eight new biradicals. We identified the best performing biradicals in organic and water-based solutions, respectively. It was found that the factors contributing to the overall sensitivity gains often compete with each other leading to similar overall sensitivity gains. We therefore pinpointed that the current design strategies in play today has appeared to reach a glass ceiling in DNP performance for cross effect dinitroxide biradicals at 9.4 T and 100 K, and new considerations need to be introduced to make further progress in the future.

POSTER 075

Polymer-Assisted Signal Amplification by Reversible Exchange in Shield Enables Alignment Transfer to Heteronuclei (SABRE-SHEATH)

Presenter: SHIRAZ NANTOGMA (Wayne State University)
All Authors: Shiraz Nantogma (Wayne State University); Caitlin Tremucha (Wayne State University); Eduard Chekmenev (Wayne State University)

We show that using polymers as co-ligands, the SABRE-SHEATH hyperpolarization of [15N]pyridine can be substantially improved (by 5-fold) over 15N polarization of [15N]pyridine when using no co-ligand. Three co-ligand polymers were investigated: poly-4-vinyl pyridine (P4VP), poly-N-vinyl imidazole (PVI), and poly-2-vinyl pyridine (P2VP). The results show that co-activation of the [15N]pyridine substrate together with PVI leads to enhancement levels that is 5 times higher than polarization of [15N]pyridine alone. A control experiment using P2VP shows that tuning monomer units of the polymers could lead to better enhancements and better strategies for scavenging the cytotoxic iridium catalyst out of prepared hyperpolarized solutions. Furthermore, the technique could potentially be extended to other substrates such as biologically relevant molecules: 4-amino-[15N]pyridine, [1-13C]pyruvate, [15N3]metronidazole and others.

POSTER 076

Developing 15N-enriched Parahydrogen Hyperpolarized Tracers for in vivo Applications

Presenter: Stefan Glöggler (Max Planck Society)
All Authors: Ruhuai Mei (Max Planck Society); Anil P. Jagtap (Max Planck Society); Philip Saul (Max Planck Society); Stefan Gloeggler (Max Planck Society)

Parahydrogen-based hyperpolarization techniques have celebrated crucial breakthroughs over the past years leading to several demonstrations of the in vivo metabolic imaging capabilities with 13C-hyperpolarized molecules.

In order to develop new tracers to expand the feasibility of disease detection, we have set one focus on developing new molecules based on 15N-enrichment and their hyperpolarization using para-hydrogen. We will give an overview about our developments and investigations with respect to designing molecules with long 15N T1s for >10 minutes traceability and show demonstrations of newly designed molecules that change their chemical structure in the presence of reactive oxygen species. We will also show first in vivo demonstrations and work up

procedures introduced by us to obtain clean contrast agents in injection buffers.

POSTER 077

Multivalent Xenon Host Options for HyperCEST NMR Provided by Various Micro-Environments in Polymer Resin Beads

Presenter: Samuel Lehr (German Cancer Research Center (DKFZ))

All Authors: Samuel Lehr (German Cancer Research Center (DKFZ)); Jabadurai Jayapaul (German Cancer Research Center (DKFZ)); Leif Schroeder (Deutsches Krebsforschungszentrum (DKFZ))

We immobilized Cryptophane-A (CrA) on polymer resin beads, resulting in multivalent host structure assemblies for HyperCEST NMR using hyperpolarized Xenon-129. Three scenarios were investigated, including functionalized beads with CrA, bare beads with uncoupled CrA, and only bare beads. Z-spectra analysis revealed several distinct molecular micro-environments perceived by Xe spins. Our results demonstrate successful immobilization of CrA on polymer beads and provide insights into the Hyper-CEST response from both CrA and the polymer matrix. This approach holds promise for creating effective in vitro phantoms for Hyper-CEST MRI applications and, importantly, also suggests such polymer beads as xenon host with efficient CEST build-up.

POSTER 078

Dynamic Nuclear Polarization of Selectively 29Si-Isotope and Radical-Enriched Onion-Shaped Silica Nanoparticles

Presenter: Youngbok Lee (Hanyang University)

All Authors: Youngbok Lee (Hanyang University)

Silica nanoparticles exhibit promising characteristics conducive to their development as 29Si MRI probes. For effective utilization of silica material in MRI, Dynamic Nuclear Polarization technique can be applied to greatly amplify the NMR signals. Here, onion-shaped silica nanoparticles, which are selectively embedded with uniform global radical concentration and 29Si-isotope ratio in distinct regions: throughout the entire particles, solely in the first 10 nm shell or exclusively in the second 10 nm shell of core@shell@shell structures, are demonstrated. The enrichment in 29Si ratio contributes to a higher hyperpolarization signal, meanwhile, the grafting of free radicals renders the particles readily polarized. This research provides valuable insights into effective strategies for 29Si-isotope and radical enrichment, facilitating the development of potential 29Si MRI probes.

POSTER 079

Next-Generation Radical Designs for Direct Polarization Transfer in High-Field DNP

Presenter: Ribal Jabbour (New York University - Abu Dhabi)

All Authors: Ribal Jabbour (Department of Chemistry, New York University Abu Dhabi); Amaria Javed (Center for Quantum and Topological Systems, New York University Abu Dhabi); Asif Equbal (Department of Chemistry, New York University Abu Dhabi)

Dynamic Nuclear Polarization (DNP) enhances Nuclear Magnetic Resonance (NMR) sensitivity via polarization transfer from electrons to nuclear spins using microwave irradiation. Polarizing agents are pivotal for this transfer's efficiency. Direct transfer occurs without intermediaries, while indirect transfer involves an intermediary proton phase. Our study shows AMUPol's high efficacy in indirect transfer at 14 T in DNP juice but drops drastically in direct transfers without spin diffusion. Quantum calculations pinpoint the importance of dipolar coupling within biradicals for direct transfer. We demonstrate ASYMPol-POK's superior direct transfer capability in deuterated juice. We further investigated the effect of a spectator radical's presence on biradical transfer efficiency, noting that closer distances diminish efficiency compared to isolated biradicals, despite quicker build-up times, as validated experimentally.

POSTER 080

Design of a Switchable MRI Reporter Addressing Alterations in ECM Composition: HyperCEST Signatures of Functionalized Xe Targeting Collagen IV

Presenter: Patrick Werner (German Cancer Research Center (DKFZ))

All Authors: Patrick Werner (German Cancer Research Center (DKFZ)); Jabadurai Jayapaul (German Cancer Research Center); Leif Schröder (German Cancer Research Center (DKFZ) Heidelberg, Molecular Translational Imaging, Germany)

This study is dedicated to develop biosensors for MRI visualization, specifically targeting the ECM of cells. Utilizing the HyperCEST technique with hyperpolarized 129Xe nuclei, the biosensors aim to detect subtle alterations in collagen distribution at nanomolar concentrations. Validation of the CrA molecule-carrying biosensors for non-fibrillar collagen VI free in solution and bound to collagen IV loaded beads provides insights into the signal changes caused by immobilization of the senor. Initial outcomes using a collagen IV-directed sensor suggest potential optimization of MRI protocols for enhanced sensitivity in disease detection. Despite being in its early stages, this research fosters novel applications for disease detection, particularly in tumors or fibrosis.

POSTER 081

Reverse dynamic nuclear polarization for indirect detection of nuclear spins close to unpaired electrons

Presenter: Nino Wili (Aarhus University)

All Authors: Nino Wili (Aarhus University); Gunnar Jeschke (ETH Zurich); Niels Chr. Nielsen (Interdisciplinary Nanoscience Center / Aarhus University)

While NMR spectroscopists are typically interested in the "bulk" nuclei, EPR spectroscopists are often more interested in the nuclei in the vicinity of the electron. They can usually not be observed by direct NMR detection, due to fast relaxation and hyperfine couplings that exceed the detection bandwidth. In this presentation, we will show how with pulsed DNP, it is possible to transfer the polarization from electron to nuclei, and then from nuclei back to electrons. The experiments yield information about nuclear spin diffusion away from the paramagnetic center. With selective rf-irradiation, it is possible to determine the hyperfine couplings of nearby nuclei. We will show similarities and differences to established ENDOR experiments.

POSTER 082

The RASER Approach: Exploiting Stimulated Emission in NMR and MRI

Presenter: Sören Lehmkuhl (KIT)

All Authors: Sören Lehmkuhl (KIT); Jing Yang (Karlsruhe Institute of Technology); Simon Fleischer (Karlsruhe Institute of Technology); Moritz Becker (Karlsruhe Institute of Technology); Eduard Chekmenev (Wayne State University); Thomas Theis (North Carolina State University); Stephan Appelt (Forschungszentrum Jülich); Mazin Jouda (Karlsruhe Institute of Technology); Jan G. Korvink (Karlsruhe Institute of Technology)

Conventional NMR and MRI rely on radio frequency (rf) pulses for excitation. An alternative to external rf-excitation is self-excitation using a RASER (Radiofrequency Amplification by Stimulated Emission of Radiation). I will discuss the potential of the RASER approach in NMR and MRI, looking at the most recent results and highlight its advantages and disadvantages. First, I will focus on the RASER "regime", understanding the different nonlinear effects that can be observed in a RASER. Second, I will focus on the precision in frequency resolution RASER-based sensing can achieve. Finally, I will discuss the potential of RASER MRI. We recorded an EPI image without applying an rf-pulse and trained a convolutional neural network to remove image artifacts.

POSTER 083

Magnetic dipole localization using optically-pumped RF atomic magnetometers

Presenter: Ayşe Maraşlı (George Mason University)

All Authors: Ayşe Maraşlı (George Mason University); Lucia Rathbun (TwinLeaf LLC); Casey Oware (TwinLeaf LLC); Mark Limes (TwinLeaf LLC); Nancy Ford (TwinLeaf LLC); Tom Kornack (Twinleaf LLC); Karen Lee Sauer (George Mason University)

An RF atomic magnetometer, a form of quantum sensor using atoms hyperpolarized by optical pumping, fundamentally makes more sensitive measurements than conventional coil detection for frequencies lower than ~50 MHz. This makes it appealing for low or zero-field NMR studies. Historically, DC magnetometers have been used to localize static dipole magnetic moments for various purposes including magnetic tracking, explosive detection, and ordnance detection. In this study, the focus is in on localizing oscillating dipole moments as from an NMR sample, using RF atomic magnetometers. Initial results, using the first ever integrated atomic magnetometer, show how a single magnetometer can measure a 2D field and give the direction towards a dipole oscillating at 423 kHz and constrained to a 1D line.

POSTER 084

Long-Lived Coherences in the Direct Dimension: Hyperpolarized Persistent Oscillations

Presenter: Aude Sadet (ELI-NP/IFIN-HH)

All Authors: Aude Sadet (ELI-NP/IFIN-HH); Ertan Turhan (Institute of Biological Chemistry, Faculty of Chemistry, University of Vienna); Milan Zachrdla (Institute of Biological Chemistry, Faculty of Chemistry, University of Vienna,); Dorte Brandis (Institute of Biological Chemistry, Faculty of Chemistry, University of Vienna,); Octavian Ianc (Biophysics and Biomedical Applications Lab, ELI-NP, Extreme Light Infrastructure - Nuclear Physics, IFIN-HH); Andrei Ciumeica (University of Bucharest, ISDS Doctoral School,); Dennis Kurzbach (University Vienna); Paul Vasos (ELI-NP/IFIN-HH)

Long-lived coherences (LLC's) in high-resolution magnets require sustaining by a radio-frequency field. For 1D hyperpolarized experiments, this poses the challenge of applying sustaining fields in conjunction with direct-dimension detection. We overcame this difficulty by alternating short continuous-wave sustaining periods with acquisition delays. We thus detected direct-dimension LLC's between the pair of glycine aliphatic protons in peptide AlaGly hyperpolarized via dissolution-DNP with a signal enhancement epsilon=30+/-5. The relaxation time constant of hyperpolarized LLC's was slower by a factor >2 compared to that of standard transverse magnetization, even in the presence of organic free-radicals used for DNP enhancement ([TEMPO]=0.1 mM). Real-time studies of chemical exchange via LLC's are rendered possible by this new development in both hyperpolarized and room-temperature applications.

POSTER 085

A prototype low field MRI device with integrated SEOP HP capabilities for GAMMA-MRI HP mXe imaging

Presenter: Stavroula Pallada (University of Applied sciences and arts of Western Switzerland (HESSO))

All Authors: Stavroula Pallada (University of Applied sciences and arts of Western Switzerland (HESSO)); Dimitrios Sakellariou (KU Leuven); Magdalena Kowalska (CERN/UNIGE); Ashley Cooper (HESSO); Nicola Giandomenico (HESSO); Anastasios Kanellakopoulos (HESSO); Bryan Musy (HESSO); Quentin Mario Rogliardo (HESSO); Victor Sanchez-Tembleque (HESSO); Harley Stoeckli (HESSO); Pauline De Pellegars (RS2D); Luis Mario Fraile (Complutense University of Madrid); Mateusz Jerzy Chojnacki (CERN); Renaud Jolivet (University of Maastricht); Julien Rivoire (RS2D); Rodrigo De Oliveira Silva (KU Leuven)

The GAMMA-MRI is an EU Horizon 2020 funded project that aims to bring innovation in clinical molecular imaging, developing a device and technique based on the physical principle of anisotropic gamma emission from hyperpolarized (HP) isomers of Xenon (mXe). With GAMMA-MRI we promise to overcome the limitations of existing imaging techniques, i.e., the low NMR/MRI sensitivity, while using its spatial resolution. For improving the signal detection sensitivity, radioactive, gamma-emitter isotopes with a gamma energy like the one of 99mTc (SPECT isotope) are used as contrast agents. Our consortium is currently at the stage of the proof-of-principle experiments using the developed GAMMA-MRI prototype.

POSTER 086

Toward biocompatible formulations, cellular and metabolic studies of [1-13C]pyruvate via Re-D SABRE-SHEATH

Presenter: Joseph Gyesi (Wayne State University)

All Authors: Joseph Gyesi (Wayne State University); Isaiah Adelabu (Wayne State University); Faisal Asif (Wayne State University); Shiraz Nantogma (Wayne State University); Hieu Tran (Wayne State University); Eduard Chekmenev (Wayne State University)

We present a progress in developing biocompatible formulation of aqueous hyperpolarized [1-13C]pyruvate contrast agent using Re-Dissolution (Re-D) SABRE-SHEATH hyperpolarization technique. This process was made feasible through fast injection of hot D2O into HP methanol solution, followed by organic solvent evaporation and precipitation of Ir-based SABRE catalyst in the aqueous phase. The separation conditions employed vacuum (100 mbar) for 20 seconds at 95 °C inside permanent magnet, yielding 13C polarization of 4% (greater polarization is feasible in the future through process optimization). SABRE-hyperpolarized aqueous [1-13C]pyruvate contrast agent will be tested in cells and animal models of cancer to ascertain the metabolic status of tumors, paving the way to ultra-fast and cost-effective approach for HP [1-13C]pyruvate production for biomedical use.

POSTER 087

In situ SABRE Hyperpolarization Monitoring with Zero- to Ultra-Low Field NMR

Presenter: Adam Ortmeier (North Carolina State University)

All Authors: Adam Ortmeier (North Carolina State University); Danila Barskiy (Johannes Gutenberg University Mainz); Nicolas Kempf (Max Plank Institute for Cybernetics); Kai Buckenmaier (Max Plank Institute for Cybernetics); Thomas Theis (North Carolina State University)

Hyperpolarized [1-13C]pyruvate is the leading metabolite used in the emerging field of hyperpolarization-enhanced MRI. Signal amplification by reversible exchange (SABRE) is a particularly simple hyperpolarization method that has recently been shown to hyperpolarize [1-13C]pyruvate at low (microtesla and below) magnetic fields. Here we show that commercial Rb-vapor magnetometers can be used to readily monitor build-up and decay of hyperpolarization. In addition, we measure ZULF-NMR spectra in various conditions, ranging from a J-coupling-dominated regime transitioning into a Zeeman-dominated regime when going from a sub-nT field to a ⦁T field. The experimentally acquired spectra are matched well by numerical simulations.

POSTER 088

Background-Free Dual-Detection of Polarization Transfer from Hyperpolarized 129Xe to Thermally Polarized Nuclei at Ultra-Low Magnetic Field

Presenter: Sebastian Atalla (The University of North Carolina at Chapel Hill)

All Authors: Sebastian Atalla (The University of North Carolina at Chapel Hill); Nikolas M. Jauch (The University of North Carolina at Chapel Hill); Rosa T. Branca (The University of North Carolina at Chapel Hill)

This investigation demonstrates direct transfer of polarization between HP 129Xe and 19F and simultaneous imaging of 129Xe and 1H via SPINOE at ultra-low field. Experiments were conducted on a lab-built NMR spectrometer operating at 2.2 mT. HP 129Xe was bubbled into samples of hexafluorobenzene (HFB) and perfluoro(methylcyclohexane) (PFMCH) for spectroscopy, and into pentane for imaging. The SPINOE-enhanced 19F signal in HFB persisted for over 200 times longer than its T1 of 1.1 s at 2.2 mT, and a minimum 19F signal enhancement of ε = 178 was observed. Preliminary images yielded an SNR of 1.7 for 129Xe and 1.3 for 1H. Simultaneous acquisition of polarization transfer enables the study of SPINOE dynamics at ultra-low field strengths.

POSTER 089

Generation of 100 MHz B1e fields with EIK-based 200 GHz/300 MHz EPR/NMR Spectrometer for Room-Temperature DNP

Presenter: Alex Smirnov (North Carolina State University)

All Authors: Antonin Marek (North Carolina State University); Sergey Milikisiyants (North Carolina State University); Alex I. Smirnov (North Carolina State University); Alexander Nevzorov (North Carolina State University)

Photonic Band-Gap resonators (PBGRs) reduce sample heating by separating the electric and magnetic mm-wave components and enhance B1e fields at the sample. The latter are essential for pulse DNP in samples with short relaxation times. Here we demonstrate highly improved 200 GHz PBGRs yielding quality factors of ca. Q=1,500. Together with an Extended-Interaction Klystron (EIK) amplifier, they yield B1e fields of ca. 100 MHz as directly measured in the induction mode. Mm-wave pulse forming and shaping was achieved by mixing the base 94 GHz frequency with a 4 GHz output of an arbitrary wave-form generator followed by the frequency doubling and amplification by EIK. Applications of our setup for pulse DNP of various samples at room temperature will be presented.

POSTER 090

NMR Detection of Picomoles of Pyruvate at Nanomolar Concentrations Using Bullet-DNP

Presenter: Pooja Narwal (Institute of Biological Interfaces-4, Karlsruhe Institute of Technology)

All Authors: Pooja Pooja (Institute of Biological Interfaces-4, Karlsruhe Institute of Technology); Nils Lorz (Department of Biology, ETH Zürich); Masoud Minaei (Institute of Biological Interfaces-4, Karlsruhe Institute of Technology); Sami Jannin (Centre de Résonance Magnétique Nucléaire à Très Hauts Champs – FRE 2034 Université de Lyon / CNRS / Université Claude); Karel Kouřil (Institute of Biological Interfaces 4, Karlsruhe Institute of Technology); Alvar D. Gossert (Department of Biology, ETH Zürich); Benno Meier (Karlsruhe Institute of Technology)

In this study, we show how a combination of bullet-DNP instrumentation, an optimized sample preparation and a further sensitivity increase via a 13C-1H polarization transfer after dissolution enable the observation of pyruvate at a concentration of 100nM immediately after dissolution. The experiment exhibits excellent mass sensitivity employing a total mass of pyruvate of only 8ng or 70pmol.

POSTER 091

Signal amplification by >10^5 times via SABRE enables rapid, simultaneous 13C coil calibration and T1 measurement of hyperpolarized [1-13C]pyruvate at 6.5mT

Presenter: Thomas Boele (University of Sydney)

All Authors: Thomas Boele (University of Sydney); Stephen McBride (North Carolina State University); Megan Pike (North Carolina State University); Erica Curran (North Carolina State University); Patrick TomHon (Vizma Life Sciences Inc.); Sheng Shen (1. A. A. Martinos Center for Biomedical Imaging, Department of Radiology, MGH, 2.Harvard Medical School); Thomas Theis (North Carolina State University); Matt Rosen (MGH/Martinos Center)

We present a convenient method for simultaneous coil calibration and measurement of spin-lattice relaxation time (T1) for hyperpolarized magnetic resonance (MR) experiments at ultra-low (mT) magnetic field. Low field MR offers affordable, portable MRI for clinical cases and treatment environments incompatible with high magnetic fields. However, the inherently low sensitivity of MRI, scaling as B0^3/2, is a challenge. Hyperpolarized MR offers a solution by boosting the MR signal available and offers new contrast methods. We present results using a set of hyperpolarized pyruvate experiments at 6.5mT to simultaneously extract tip angle and T1. With a properly calibrated coil and known T1, NMR and MRI sequences can take maximum advantage of large, non-renewable magnetization delivered by hyperpolarization techniques involving sample shuttling.

POSTER 092

Tailored Binitroxides for DNP at High Field and Fast MAS: from Proton Density to Solvation Effects

Presenter: Lorenzo Niccoli (CERM, University of Florence)

All Authors: Lorenzo Niccoli (CERM, University of Florence); Gilles Casano (Aix Marseille Univ, CNRS, ICR); Georges Menzildjian (CRMN); Maxim Yulikov (Department of Chemistry and Applied Biosciences, ETHZ); Thomas Robinson (CRMN (CNRS/ENS-Lyon/UCBL)); Zhouran Wang (CRMN (CNRS/ENS-Lyon/UCBL)); Christian Reiter (Bruker BioSpin); Didier Siri (Aix Marseille Univ, CNRS, ICR); Amrit Venkatesh (National High Magnetic Field Laboratory, Florida State University); Lyndon Emsley (EPFL); David Gajan (CRMN (CNRS/ENS-Lyon/UCBL)); Olivier Ouari (Aix Marseille Univ, CNRS, ICR); Moreno Lelli (CERM-University of Florence); Anne Lesage (CRMN (CNRS/ENS-Lyon/UCBL))

We have designed a new series of Polarizing Agents (PAs) suitable for high field and fast MAS DNP solid state NMR. These are improved TinyPol structures functionalized with long protonated chains, that outperform previously designed binitroxides in terms of overall sensitivity gain. The best radicals in the series yields an enhancement > 200 at 18.8 T and 65 kHz MAS. Through a combined approach of 2H ESEEM experiments and molecular dynamics in various DNP formulations we investigated the environment around the nitroxides. Our findings indicated that the deuterated glycerol molecules occupy the second solvation sphere, supporting our hypothesis that protons in the chains are key to distribute the polarization across the spin diffusion barrier.

POSTER 093

Variable Resolution Hyperpolarized 13C Imaging with Partial Fourier Enabled by Low-Rank Modeling of Local K-Space Neighborhoods (LORAKS)

Presenter: Daniel Vigneron (UCSF)

All Authors: Tanner Nickles (UCSF); Yaewon Kim (University of California, San Francisco); Jasmine Hu (UCSF); Hsin-yu Chen (UCSF); Peder Larson (UCSF); Daniel Vigneron (UCSF); Jeremy Gordon (UCSF)

Hyperpolarized (HP) dynamic nuclear polarization (DNP) MRI with a variable resolution approach utilizes a metabolite-selective spectral-spatial RF excitation with an EPI readout to produce high-resolution images for [1-13C]pyruvate and coarser resolution images [1-13C]lactate and [13C]bicarbonate with adequate SNR. However, the relatively long TE associated with this imaging approach leads to increased T2* weighting, reducing SNR for 13C metabolites with shorter observed T2s. Here, we retrospectively explored whether Partial Fourier (PF) image reconstruction through low-rank modeling of local k-space neighborhoods (LORAKS) could maintain image fidelity of [1-13C]pyruvate HP MR acquired with a variable resolution imaging approach. PF imaging and reconstruction using LORAKS was shown to maintain signal fidelity and preserve image quality in variable resolution human HP [1-13C]pyruvate brain data.

POSTER 094

Toward Lung Ventilation Imaging Using Hyperpolarized Diethyl Ether Gas and Other Proton-Hyperpolarized Inhalable Contrast Agents

Presenter: Anna Samoilenko (Wayne State University)

All Authors: Nuwandi Ariyasingha (Wayne State University); Anna Samoilenko (Wayne State University); Md Raduanul Chowdhury (Wayne State University); Clementinah Oladun (Wayne State University); Oleg G. Salnikov (International Tomography Center); Nikita V. Chukanov (International Tomography Center); Larisa M. Kovtunova (International Tomography Center); Igor V. Koptyug (International Tomography Center); Boyd Goodson (Southern Illinois University); Eduard Chekmenev (Wayne State University)

Hyperpolarized Xe-129 gas was FDA-approved as an inhalable contrast agent for functional lung imaging of a wide range of pulmonary diseases in December 2022. However, hyperpolarized Xe-129 production is expensive and slow. Here, we report on development of proton-hyperpolarized contrast agents that can be produced on demand using ultra-fast disposable hyperpolarization equipment. Hyperpolarized diethyl ether gas was produced via heterogeneous parahydrogen induced polarization and utilized for ventilation imaging of excised rabbit lungs with 4x4 mm2 resolution. Other proton-hyperpolarized gases (propane and butane) are also amenable to this new technology with production capacity of 0.7 liters in one second. It becomes feasible to perform high-resolution imaging

(1.6x1.6-mm2 pixel size) using conventional clinical MRI scanner without any hardware or pulse sequence modifications.

POSTER 095

RF Surface Coil Design for High-Throughput Metabolic Imaging using Microfluidics

Presenter: Gergo Matajsz (Institute for Bioengineering of Catalonia)

All Authors: Gergo Matajsz (Institute for Bioengineering of Catalonia); David Gomez Cabeza (Institute for Bioengineering of Catalonia); Irene Marco-Rius (Institute for Bioengineering of Catalonia)

Organ-on-a-chip platforms provide low-cost and high-throughput alternatives to current clinical methods in accurately assessing complex diseases. Combining them with Hyperpolarized Magnetic Resonance Spectroscopic Imaging (HP-MRSI), we acquire signals of 8 biological models simultaneously in a physiologically accurate environment. Our challenge is efficiently acquiring the quickly decaying hyperpolarized signal in parallel for all samples.

Most commercially available radiofrequency coils are sized for rodents rather than microfluidic platforms. Our solution is designing a parallel array transmit-receive surface coil with separate array elements covering each biological model. After maximizing our filling factor, we expect to see our most robust signal detection system up to date with the highest signal-to-noise ratio, showcasing the full potential of HP-MRSI to detect 13C-labelled compounds in parallel.

POSTER 096

Development of New Dual-Element 13C/1H Endorectal Coil for Improved Multiparametric MR-TRUS Guided Fusion Prostate Biopsies with Hyperpolarized C-13 Molecular Imaging

Presenter: Daniel Gebrezgiabhier (University of California)

All Authors: Daniel Gebrezgiabhier (University of California); Lucas Carvajal (University of California); Hsin-Yu Chen (University of California); Yaewon Kim (University of California, San Francisco); Robert A. Bok (University of California); Matthew R. Cooperberg (University of California); Hao G. Nguyen (University of California); Katsuto Shinohara (University of California); Kimberly Okamoto (University of California); Mary Frost (University of California); Zhen J. Wang (University of California); Michael A. Ohliger (University of California); Jeremy Gordon (University of California); Peder E.Z Larson (University of California); Rahul Aggarwal (University of California); Daniel Vigneron (University of California)

We designed and fabricated a novel dual-element 13C/1H endorectal coil (ERC) that, compared to prior designs, would provide substantially improved sensitivity, rf isolation, and reliability with improved electronics for hyperpolarized 13C prostate MRI for guiding biopsies. It was validated with bench testing and then evaluated in 3T MRI studies. Phantoms were imaged using the new dual-element 13C/1H ERC and the original ERC. Acquired data was then analyzed and respective SNR compared. The new ERC has demonstrated substantially improved 13C and 1H MRI sensitivity on bench electronic testing. In addition, the new ERC demonstrated substantial improvement in signal-to-noise ratio, and since then coil has successfully been used in hyperpolarized 13C-pyruvate, co-polarized 13C-urea+pyruvate multiparametric MRI patient studies for guiding prostate cancer biopsy.

POSTER 097

Hyperpolarization of 1,5-[13C2-Z-OMPD] via SABRE-SHEATH for MR pH Sensing

Presenter: Mustapha Abdulmojeed (North Carolina State University)

All Authors: Mustapha Abdulmojeed (North Carolina State University); Martin Grashei (Technical University of Munich); Seth Dilday (North Carolina State University); Atli Davidsson (North Carolina State University); Erica Curran (North Carolina State University); Stephen McBride (North Carolina State University); Keilian MacCulloch (North Carolina State University); Austin Browning (North Carolina State University); Patrick TomHon (Vizma Life Sciences); Andreas Schmidt (University of Freiburg); Franz Schilling (Technical University of Munich); Thomas Theis (North Carolina State University)

Magnetic resonance (MR) techniques are attractive non-invasive methods of measuring both intra- and extra-cellular pH. However, the inherent low sensitivity of MR techniques, due to very low thermal nuclear polarization (P13C ≃ 0.0001 % at 1.1 T), have limited the ability to measure precisely both intra- and extra- cellular pH. This limitation can be addressed using novel hyperpolarization protocol to increase the sensitivity of MR contrast agents beyond thermal conditions. [1,5-13C2]-Z-4-methyl-2-oxopen-3-enedioic acid (Z-OMPD) is an MR contrast agent which have been used to measure pH in vivo. In this work, we have used Signal Amplification by Reversible Exchange in shield enabling alignment transfer to heteronuclei (SABRE-SHEATH) to hyperpolarize [1,5-13C2]-Z-OMPD for the first time and showed its pH sensing potential.

POSTER 098

A Newly Designed Hyperpolarized Aminopeptidase N Probe Sensitively Detects Early Therapeutic Responses Heterogeneously on Pancreatic Tumors

Presenter: Kazutoshi Yamamoto (National Institutes of Health / National Cancer Institute)

All Authors: Norikazu Koyasu (National Institutes of Health / National Cancer Institute); Hiroyuki Yatabe (The University of Tokyo); Yoichi Takakusagi (National Institutes for Quantum and Radiological Science and Technology); Yutaro Saito (The University of Tokyo); Shinsuke Sando (The University of Tokyo); Murali C. Krishna (National Institutes of Health / National Cancer Institute); Kazutoshi Yamamoto (National Institutes of Health / National Cancer Institute)

Molecular imaging is a promising methodology for diagnosing cancer and monitoring its treatments by noninvasively visualizing the alternations of cancer metabolisms. Here, dissolution Dynamic Nuclear Polarization (DNP) is an emerging technology to observe therapeutic responses in spatiotemporal enzymatic activities particularly at earlier stages before the volumetric changes can be observed. However, the number of available dissolution DNP probes are still limited for in vivo as well as clinical applications. In this presentation, we will demonstrate a framework for a rationally designed novel dissolution DNP probe, an aminopeptidase-N (CD13) probe, which is targeted to the highly-selective enzymatic activity of aminopeptidase-N and can non-invasively detect heterogenetic treatment responses in tumor xenografts with an anti-angiogenic/antitumor drug, sunitinib, at earlier stages.

POSTER 099

Biocompatible Assessment of N-15 Hyperpolarized Metronidazole for Hypoxia Sensing

Presenter: Faisal Asif (Wayne State University)

All Authors: Faisal Asif (Wayne State University); Mohammad Shah Hafez Kabir (PhD Candidate, Department of Chemistry, Ibio, Wayne State University, Karmanos Cancer Institute (KCI)); Joseph Gyesi (Wayne State University); Isaiah Adelabu (Wayne State University); Shiraz Nantogma (Wayne State University); Eduard Chekmenev (Wayne State University)

Recent advances in the development of hyperpolarization techniques enabled a wide range of in vivo MRI applications. We have recently demonstrated that [15N3]metronidazole, a potential hyperpolarized contrast agent, can be 15N-hyperpolarized to 15% via SABRE technique, and its hyperpolarized state has an ultra-long T1 of 10-minute. Here, we perform SABRE-SHEATH hyperpolarization in CH2Cl2 media, followed by the phase-extraction of hyperpolarized [15N3]metronidazole into aqueous media, whereas the toxic catalyst is retained in the CH2Cl2 layer, allowing us to prepare aqueous solutions free from organic solvent and Ir-based catalyst. Pilot results demonstrated polarization of 4%, with ongoing efforts to surpass 20%. We aim to employ aqueous solutions of HP [15N3]metronidazole for hypoxia sensing applications in cellular and animal models of cancer.

POSTER 100

Role of the Electron Relaxation Times in MAS DNP of Hybrid Biradicals

Presenter: Moreno Lelli (CERM-University of Florence)

All Authors: Lorenzo Niccoli (CERM, University of Florence); Lyndon Emsley (EPFL); Anne Lesage (CRMN-ENS Lyon); Moreno Lelli (CERM-University of Florence)

Electron relaxation times are among the most significant parameters that regulate the efficiency of the DNP process. Increasing the electron relaxation times of the polarizing agent is usually beneficial for MAS DNP efficiency allowing one to extend the operative DNP temperatures above 100 K. We investigated, through simulations, the role of the electron relaxation times in MAS DNP showing that electron T1 and T2 are important not only in permitting electron saturation, but they deeply affect several aspects of the DNP mechanism under MAS. In particular, we will describe the case of hybrid biradicals like HyTEK2 discussing how the experimental decrease of DNP enhancement with temperature could be interpreted.

POSTER 101

Towards the Development of Novel "Dual-Reporter" MRI Contrast Agents Hyperpolarized via SABRE

Presenter: Ishani Senanayake (Southern Illinois University Carbondale)

All Authors: Ishani Senanayake (Southern Illinois University Carbondale); Nadiya Iqbal (Southern Illinois University Carbondale); Zahid Siraj (Southern Illinois University Carbondale); Annie Y. Vargas Lizarazo (Southern Illinois University Carbondale); Sunil Basnet (Southern Illinois University Carbondale); Punit Kohli (Southern Illinois University Carbondale); Sean D. Moran (Southern Illinois University Carbondale); Eduard Chekmenev (Wayne State University); Yuqing Hou (Southern Illinois University Carbondale); Boyd Goodson (Southern Illinois University)

The hyperpolarization technique SABRE (Signal Amplification By Reversible Exchange) has drawn growing interest because it is rapid, inexpensive, and easy to perform, However, increasing the capabilities of agents amenable to SABRE is necessary to realize envisioned applications. We describe ongoing efforts to create and characterize dual-modality agents that can simultaneously report on physiological effects via both magnetic resonance and optical emission. We report on our efforts to synthesize, characterize, and hyperpolarize the fluorescent lysosomal-targeting agent 4-[(1E)-2 [4-(1H-Imidazol-1-yl)phenyl]ethenyl]-N,N-diphenylbenzenamine (here, dubbed structure G). Optical (UV/Vis, fluorescence) and NMR characterization confirm synthesis of G; Successful observation of 1H SABRE of a key precursor bodes well for ongoing attempts to achieve 15N SABRE hyperpolarization of G—a prelude to planned labeling and cellular studies.

POSTER 102

Nitrogen-15 dynamic nuclear polarization of nicotinamide and derivatives in biocompatible solutions

Presenter: Josh Philipp Peters (Section Biomedical Imaging, UKSH, Kiel)

All Authors: Josh Philipp Peters (Section Biomedical Imaging, UKSH, Kiel); Arne Brahms (Kiel University, Otto Diels Institute for Organic Chemistry, Kiel, Germany); Vivien Janicaud (University Medical Center Kiel, Kiel University, Section Biomedical Imaging, Molecular Imaging North Competence Center (MOIN CC), Department of Radiology and Neuroradiology, Kiel, Germany); Maria Anikeeva (University Medical Center Kiel, Kiel University, Section Biomedical Imaging, Molecular Imaging North Competence Center (MOIN CC), Department of Radiology and Neuroradiology, Kiel, Germany); Eva Peschke (University Medical Center Kiel, Kiel University, Section Biomedical Imaging, Molecular Imaging North Competence Center (MOIN CC), Department of Radiology and Neuroradiology, Kiel, Germany); Frowin Ellermann (University Medical Center Kiel, Kiel University, Section Biomedical Imaging, Molecular Imaging North Competence Center (MOIN CC), Department of Radiology and Neuroradiology, Kiel, Germany); Arianna Ferrari (University Medical Center Kiel, Kiel University, Section Biomedical Imaging, Molecular Imaging North Competence Center (MOIN CC), Department of Radiology and Neuroradiology, Kiel, Germany); Janka Held-Feindt (Universität zu Lübeck, Lübeck, Germany); Na-mi Kim (University Medical Center Kiel, Kiel University, Institute of Clinical Molecular Biology, Kiel, Germany); Johannes Meiser (Cancer Metabolism Group, Department of Cancer Research, Luxembourg Institute of Health, Luxembourg.); Konrad Aden (University Medical Center Kiel, Kiel University, Institute of Clinical Molecular Biology, Kiel, Germany); Rainer Herges (Kiel University, Otto Diels Institute for Organic Chemistry, Kiel, Germany); Jan-Bernd Hövener (University Medical Center Kiel, Kiel University, Section Biomedical Imaging, Molecular Imaging North Competence Center (MOIN CC), Department of Radiology and Neuroradiology, Kiel, Germany); Andrey Pravdivtsev (University Medical Center Kiel, Kiel University, Section Biomedical Imaging, Molecular Imaging North Competence Center (MOIN CC), Department of Radiology and Neuroradiology, Kiel, Germany)

Dissolution dynamic nuclear polarization (dDNP) enables real-time in vivo metabolic imaging to be performed non-invasively. Here, we are developing a group of dDNP polarized tracers based on nicotinamide (NAM). We synthesized 1-15N-NAM and 1-15N-nicotinate and hyperpolarized them with dDNP, reaching (13.0 ± 1.9)% 15N-polarization. We found that the lifetime of hyperpolarized 1-15N-NAM is strongly field- and pH-dependent, with T1 being as long as 41 s at a pH of 12 and 1 T while as short as a few seconds at neutral pH and fields below 1 T. This effect was not observed with methylated NAM. Using 15N dDNP and an inexpensive rodent imaging probe designed in-house, we acquired a 15N MRI of hyperpolarized 1-15N-NAM in less than 1 s.

POSTER 103

Nanocarriers with Variable Membrane Fluidity as HyperCEST Agents with Superior Performance of Membrane-Bound Xenon Hosts

Presenter: Leif Schroeder (Deutsches Krebsforschungszentrum (DKFZ))

All Authors: Leif Schroeder (Deutsches Krebsforschungszentrum (DKFZ)); Felix Schnabel (Deutsches Krebsforschungszentrum (DKFZ)); Jabadurai Jayapaul (German Cancer Research Center)

CEST agents for hyperpolarized Xe-129 (so-called HyperCEST reporters) should provide efficient exchange of Xe in/out of tailored host structures. For liposomal designs, this may be influenced by the phospholipid membrane fluidity. Cholesterol, which is often added for liposome stability, may thus impact the HyperCEST performance of such nanocarriers. We compared the changes in saturation transfer buildup from liposomes with variable cholesterol content for either membrane-anchored (i.e., lipopeptide-based) or freely diffusing Xe hosts. The HyperCEST efficiency for membrane-anchored Xe hosts is much less sensitive to membrane stiffening than for unbound hosts. Lipopeptide-based agents are thus a promising, flexible platform for translating the HyperCEST MRI approach towards in vivo applications.

POSTER 104

Isotope Shifts, Geometric Double-Quantum Excitation, and Singlet NMR of Oxygen-18 Enriched Squarate

Presenter: Urvashi Heramun (University of Southampton)

All Authors: Urvashi Heramun (University of Southampton); Christian Bengs (University of Southampton); Mohamed Sabba (University of Southampton); Gamal Moustafa (University of Southampton); Malcolm H. Levitt (University of Southampton)

Nuclear singlet order is protected against common relaxation mechanisms, and decays slower than longitudinal order. The small, symmetrical 1,3-13C2-squarate molecule is a promising candidate, yet unexplored by singlet NMR due to difficulties in synthesis. To generate singlet order, the magnetic equivalence of the system is broken by oxygen-18 enrichment. We observe isotope shifts, "dynamic" isotope shift effects, and implement a novel double-quantum excitation method exploiting the geometric Aharonov-Anandan phase. It demonstrated higher efficiency in exciting double-quantum coherences in systems at near-magnetic equivalence, compared to standard techniques. We apply the procedure to isolate signals arising exclusively from 13C2-isotopologues present within an 18O-enriched 13C1-squarate sample, and measure the singlet lifetime TS of 1,3-13C2-squarate for the first time.

POSTER 105

Yeast as a convenient platform studying metabolism with hyperpolarized contrast agents

Presenter: Jan-Bernd Hövener (Section Biomedical Imaging, UKSH, Kiel)
All Authors: Josh Philipp Peters (Section Biomedical Imaging, UKSH, Kiel); Charbel Assaf (Section Biomedical Imaging, UKSH, Kiel); Farhad Haj Mohamad (Section Biomedical Imaging, UKSH, Kiel); Sanjay Tiwari (Section Biomedical Imaging, UKSH, Kiel); Konrad Aden (University Medical Center Kiel, Kiel University, Institute of Clinical Molecular Biology, Kiel, Germany); Jan Hoevener (UKSH, Kiel University); Andrey Pravdivtsev (Section Biomedical Imaging, UKSH, Kiel)

Metabolism, an immediate indicator of organismal state, demands non-invasive monitoring for understanding biochemistry in vivo. Hyperpolarized NMR enables µmol/L resolution within seconds. Employing dDNP, we studied Saccharomyces cerevisiae's metabolism of pyruvate and fumarate. Yeast was found to be very active, metabolizing up to 10% of the initial substrate within seconds. Fitting first-order-kinetic functions to the data yielded robust metabolic metrics, which were further improved by normalization (python-toolbox). Environmental stress did not significantly alter the metrics, underscoring yeast's resilience.

This highlights yeast's potential as a benchmark for hyperpolarization studies, also offering insights into metabolism and responses to stimuli like drugs and treatments. Yeast's abundance and human-like metabolism position it as a standard for calibration of hyperpolarization equipment and in vitro biochemistry.

POSTER 106

Fully-Automated Batch-Mode Clinical-Scale 4th-Generation SEOP Xenon-129 Hyperpolarizer

Presenter: Michael Barlow (University of Nottingham)
All Authors: Michael Barlow (University of Nottingham); Clementinah Oladun (Wayne State University); Firoz Ahmed (Wayne State University); Md Raduanul Chowdhury (Wayne State University); Abdulbasit Tobi Gafar (Southern Illinois University Carbondale); Anton Shcherbakov (Xeus Technologies LTD); Boyd Goodson (Southern Illinois University); Eduard Chekmenev (Wayne State University); Panayiotis Nikolaou (Xeus Technologies LTD)

Hyperpolarized 129 Xe gas is a revolutionary MRI contrast agent that has been recently FDA approved for clinical use. Hyperpolarized 129 Xe is typically produced via spin-exchange optical pumping. Our clinical-scale batch-mode generation-4 SEOP hyperpolarizer employs batch-mode production, as well as in-situ polarimetry of Rb electron and 129 Xe nuclear spin polarization. Here, we demonstrate next-generation advanced automation and systems integration embodied by our 129 Xe hyperpolarizer. For example, NMR and NIR polarimetry are performed in real time using an ARM-based SMT32 microcontroller and guide the production process of SEOP hyperpolarization in real time. We anticipate this next-generation clinical-scale automation technology will improve access to HP Xe contrast agent for scientific and bio-medical communities.

POSTER 107

A Low-Cost ODNP System for Single-Sided NMR

Presenter: Olivia Cassara (William & Mary)
All Authors: Olivia Cassara (William & Mary); Daphna Shimon (The Hebrew University of Jerusalem); Tyler Meldrum (William & Mary)

NMR is limited by low sensitivity and expensive, bulky hardware. We aim to circumvent these limitations by applying DNP techniques to single sided NMR in situ. Single sided NMR provides a non-temperature controlled, open geometry system that can be used for bulk characterization. Increased insensitivity due to a larger field gradient makes polarization techniques very attractive in optimizing measurements. Construction of a DNP system on single sided NMR includes using a microwave horn, instead of a resonator cavity. Positioning the horn directly above a thin sample maximizes power at the region of interest. Our low-cost experimental setup has been shown to be a functional DNP technique providing a preliminary negative enhancement factor of at least 1.2 without a resonator.

POSTER 108

SABRE Hyperpolarization Ultralow-field MRI Platform

Presenter: Nicolas Kempf (Max Planck Institute for Biological Cybernetics)
All Authors: Nicolas Kempf (Max Planck Institute for Biological Cybernetics); Andrey N. Pravdivtsev (Section Biomedical Imaging, Molecular Imaging North Competence Center (MOIN CC), Department of Radiology and Neuroradiology, University Medical Center, Kiel University); Markus Plaumann (Institute for Molecular Biology and Medical Chemistry, Medical Faculty, Otto-von-Guericke University); Rainer Körber (Division Medical Physics and Metrological Information Technology, Physikalisch-Technische Bundesanstalt); Jörn Engelmann (High-Field Magnetic Resonance Center, Max Planck Institute for Biological Cybernetics); Richard Neumann (High-Field Magnetic Resonance Center, Max Planck Institute for Biological Cybernetics); Friedemann Bullinger (High-Field Magnetic Resonance Center, Max Planck Institute for Biological Cybernetics); Klaus Scheffler (High-Field Magnetic Resonance Center, Max Planck Institute for Biological Cybernetics); Thomas Theis (North Carolina State University); Kai Buckenmaier (High-Field Magnetic Resonance Center, Max Planck Institute for Biological Cybernetics)

The zero and ultra-low field regime opens up new opportunities for NMR by parahydrogen-based hyperpolarization. Our in situ combination of SABRE with a SQUID between 10 fT and 30 mT embodies a highly versatile magnetic resonance system. Recent efforts have improved 15N polarization by 30 % with alt-SABRE-SHEATH and polarized [1-13C]pyruvate in situ with LIGHT-SABRE continuously without magnetic field cycling. We obtained 3-dimensional 13C images of [1-13C]pyruvate in a phantom with sub-millimeter resolution at 120 µT. Using a SABRE-SHEATH ZULF sequence we measured the hyperpolarized signals of [1-13C]pyruvate between 1 nT and 100 µT, showing the transition between the Zeeman dominated and the J-coupling dominated regime. The results pave the way for in vivo ULF imaging with SABRE.

POSTER 109

Triplet Dynamic Nuclear Polarization of Pentacene-Doped Crystals for Quantum Sensing

Presenter: Noella D'Souza (University of California, Berkeley)
All Authors: Noella D'Souza (University of California, Berkeley); Harpreet Singh (UC Berkeley); Joseph Garrett (University of California, Berkeley); Keyuan Zhong (Tsinghua University); Julianne Oshiro (University of California, Los Angeles); Angel Zhang (University of California, Berkeley); Ushoshi Basumallick (University of California, Berkeley); Jonathan Breeze (University College London); Riccardo Montis (University of Urbino Carlo Bo); Jeffrey Reimer (University of California, Berkeley); Ashok Ajoy (UC Berkeley)

Record-long T1 coherences ranging from 37 min. at room temperature to 800 hr. at 6 K have been reported for proton spins in pentacene-doped crystals (PDC), ideal for quantum sensing. PDCs can be grown cost-effectively and at scale, relative to other quantum sensors, offering more nuclear spin sensors in the sample volume and increased SNR.

PDCs can be optically initialized and readout either inductively via 1H NMR or optically via ODMR. Peak shifts in the ODMR spectrum are proportional to the local magnetic field strength. Phase perturbations of the nuclear spin ensemble under pulsed-spin locking indicate a local magnetic field. The instrumentation built here enables both readout methods, variable temperature (77K-RT), and variable magnetic field (0.02-7.04T) experiments.

POSTER 110

P1 diamonds as an efficient source of nuclear hyperpolarization under magic angle spinning at 14.1 T from 30 K to room temperature

Presenter: Celeste Tobar (UCSB)
All Authors: Celeste Tobar (UCSB); Raj Chaklashiya (UCSB); Martyna Judd (The Australian National University); Quentin Stern

(Northwestern Univeristy); Jinlei Cui (Northwestern); Songi Han (Northwestern University)

P1 center diamond is recognized for its robust polarization capabilities in room temperature DNP studies, attributed to its clustered and isolated forms, prolonged spin quantum states, and enhanced coherence and relaxation times, facilitated by the nitrogen-vacancy (NV) center. Our investigation aims to unveil the pivotal role of the P1 center diamond in solid-state DNP across varying experimental conditions. Using an advanced gyrotron design enabling frequency control through temperature stepping of the gyrotron cavity, we perform MAS-DNP experiments at 14.1 T across temperatures from 298K to 30K. Quantum simulations complement our experimental findings, revealing the efficacy of diamonds as polarizing agents for MAS-DNP and shedding light on overlooked features like a hidden population at ultra-low temperatures.

POSTER 111

A 65 K Liquid Nitrogen-Cooled Parahydrogen Generator using Injected Hydrogen Gas Bubbles

Presenter: Nicholas Whiting (Rowan University)
All Authors: Vincent Ferraro (Rowan University); Nicholas Whiting (Rowan University)

Generation of parahydrogen gas can be a relatively straightforward and affordable approach to hyperpolarizing nuclear spins for enhanced NMR and MRI applications. Liquid nitrogen-cooled generators typically produce parahydrogen enrichments of ~51%; while impactful and widespread, there are further benefits to having access to even higher parahydrogen fractions for MR applications without the cost and complexity of employing a helium cold head. Here, we introduce a technique to lower the boiling temperature of liquid nitrogen via internal evaporation into injected hydrogen gas bubbles. This simplistic modification cools the liquid nitrogen from 77 K to 65 K and should allow for the generation of ~61% parahydrogen, which can be further used to hyperpolarize the nuclei of interest in substrate molecules.

POSTER 112

Quantifying Sensitivity and Performance of SABRE- and PHIP-Enhanced Spectroscopy and Imaging on a Clinical "Point-of-Care" Low-Field MRI Scanner.

Presenter: Nadiya Iqbal (Southern Illinois University Carbondale)
All Authors: Nadiya Iqbal (Southern Illinois University Carbondale); Brockton Tonazzi (Southern Illinois University Carbondale); Ishani Senanayake (Southern Illinois University Carbondale); Praveen J. Daluwathumullagamage (Southern Illinois University Carbondale); Drew O. Brittin (Southern Illinois University Carbondale); Anthony Petrilla (Southern Illinois University Carbondale); Margaret Pugh (Southern Illinois University Carbondale); Lillian Sordello (Southern Illinois University Carbondale); Sudarshan Ragunathan (Hyperfine Inc.); Megan Poorman (Hyperfine Inc.); Laura Sacolick (Hyperfine Inc.); Thomas Theis (North Carolina State University); Matt Rosen (MGH/Martinos Center); Eduard Chekmenev (Wayne State University); Boyd Goodson (Southern Illinois University)

We describe our ongoing efforts to integrate rapid and inexpensive SABRE- and PHIP-based hyperpolarization approaches with portable "point-of-care" low-field (0.064 T) clinical MRI scanners (Hyperfine). Efforts include fast scanning with a large-volume (~30 mL) continuously-recirculating SABRE setup, determining limits of detection and quantification in the imaging as functions of substrate concentration (scanning substrates down to a few mM) and polarization, and imaging PHIP-hyperpolarized gases. We are also exploring the new fast (~1 s) spectroscopy capability on the scanner, both as a platform for developing rudimentary SLIC-based sequences and for mapping the homogeneity of the scanner across the detection volume.

POSTER 113

Probing the Efficiency of Adiabatic Pulses in SLIC-SABRE Experiments

Presenter: Stephen McBride (North Carolina State University)
All Authors: Stephen McBride (North Carolina State University); Patrick TomHon (Vizma Life Sciences); Eduard Chekmenev (Wayne State University); Thomas Theis (North Carolina State University)

Spin-Lock Induced Coupling Signal Amplification By Reversible Exchange (SLIC-SABRE) employs a static magnetic field and transverse continuous wave (CW) pulse to drive spin order transfer to target heteronuclei. SLIC-SABRE can be performed at any static magnetic field, enabling the use of fields with favorable relaxation dynamics for deuterated pyruvate. SLIC-SABRE outside of a spectrometer requires the use of an adiabatic pulse that translates transverse magnetization into axial magnetization, preventing dephasing during transfer to a detection field. In this present work, we probe the adiabatic pulse efficiency by observing the relative impact on protonated pyruvate z-polarization through parameters such as pulse length, frequency modulation, amplitude modulation, and maximum amplitude for the simplest linearly ramped adiabatic half passage pulse.

POSTER 114

SABRE Dynamics of 15N-Nitriles as Potential Hyperpolarized Zinc (Zn2+) Sensors

Presenter: Brojo Kishor Shachib Dhali (North Carolina State University)
All Authors: Brojo Kishor Shachib Dhali (North Carolina State University); Megan Pike (North Carolina State University); Keilian MacCulloch (North Carolina State University); Austin Browning (North Carolina State University); Adam Ortmeier (North Carolina State University); Xuedan Wu (UNC School of Medicine); Zibo Li (UNC School of Medicine); Thomas Theis (North Carolina State University)

Zn2+ is a crucial trace element involved in numerous biological activities. A Zn2+ sensor can detect and monitor Zn2+ - ion and play a significant role in biomedical research. Since Zn2+ is susceptible to forming coordination bond with the nitrogen nuclei of nitriles, we are aiming to monitor this interaction in real time in NMR / MRI. The limiting factor to monitor this interaction in low concentrations is the notorious insensitive nature of traditional NMR. In this work we use SABRE hyperpolarization to overcome this limitation by ~11200 - fold signal enhancement. We hyperpolarized 2,6-dimethoxybenzonitrile and obtained a remarkably prolonged T1 relaxation time of ~396 seconds for the nitrogen nuclei that enables its application as a hyperpolarized zinc (Zn2+) sensor.

POSTER 115

1H/15N NMR and Low-Field 1H MRI Studies of SABRE-Hyperpolarized Pyrazinamide: An FDA-Approved Antibiotic and Potential MRI Contrast Agent

Presenter: Zahid Siraj (southern illinois university carbondale)
All Authors: zahid siraj (southern illinois university carbondale); Ishani M. Senanayake (southern illinois university carbondale); Fathima iqbal (southern illinois university carbondale); Anthony Petrilla, (SIUC); Margaret Pugh (siuc); Sudarshan Ragunathan (hyperfine); Roman V. Shchepin (South Dakota School of Mines & Technology (SDSM&T),); Eduard Y. Chekmenev (Wayne state); Boyd M. Goodson (SIUC)

Pyrazinamide (PZA), an FDA-approved antibiotic, is of interest as a potential MRI contrast agent. Following the work of others, PZA was readily hyperpolarized via SABRE at ~5.5 mT. SABRE-SHEATH achieved direct 15N hyperpolarization of naturally abundant 15N spins in PZA, yielding >4100-fold enhancement at 0.2 uT. The resonance (332.7 ppm) is tentatively assigned to the N meta to the amide group based on the 1H enhancements. Increasing the hyperpolarization through optimization should allow characterization of the 15N relaxation properties in this system. We are currently working to develop synthetic strategies for achieving 15N labeling of the N sites in PZA's heterocycle. Finally, we are studying low-field 1H MRI of hyperpolarized PZA using a 64 mT portable point-of-care clinical scanner.

Instrumentation (Posters 116-144)

POSTER 116

CNC Laser Etching vs. CNC Micro-milling: A Comparison of Methods for 3D Volume Microcoil Production

Presenter: Vincent Moxley-Paquette (University of Toronto)
All Authors: Vincent Moxley-Paquette (University of Toronto); Jacob Pellizzari (University of Toronto); Daniel Lane (University of Toronto); Ronald Soong (University of Toronto); Peter M. Costa (University of Toronto); William W. Wolff (University of Toronto); Daniel H. Lysak (University of Toronto); Rajshree Ghosh Biswas (University of Toronto); Dmitri Zverev (NSCNC Manufacturing LTD); Peter De Castro (Bruker BioSpin AG); Thomas Frei (Bruker BioSpin AG); Danijela Al Adwan-Stojilkovic (Bruker BioSpin AG); Stephan Graf (Bruker BioSpin AG); Simon Gloor (Bruker BioSpin AG); Daniel Schmidig (Bruker BioSpin AG); Rainer Kuemmerle (Bruker BioSpin AG); Till Kuehn (Bruker BioSpin AG); Falko Busse (Bruker Biospin GmbH); Andressa Lacerda (Synex Medical); Ben Nashman (Synex Medical); André J. Simpson (University of Toronto)

5-axis CNC micromilling was demonstrated to be an excellent tool for prototyping custom microcoil technology that can be tailored to various mass-limited samples. However, this technique lacks the precision required for machining complex 3D volume microcoils (i.e. solenoids and saddle coils). To counter this, a high-precision ELARA 4-axis CNC milling machine and a MiRA7L 5-axis CNC machine outfitted with a Pssat UV picosecond Laser were developed. Solenoid and saddle coils were machined onto a Cu-coated Acrylic rod using the ELARA and a Solenoid was Laser-etched into a Zero-Susceptibility Alloy-coated capillary. Here, the ability of a high-precision 4-axis CNC milling machine to develop complex 3D volume microcoils will be compared to a 5-axis Laser CNC machine (ideal for fragile substrates).

POSTER 117

Towards Microcoil Arrays for High Throughput Environmental Nuclear Magnetic Resonance

Presenter: Daniel Lysak (University of Toronto)
All Authors: Daniel Lysak (University of Toronto); Marco Grisi (Annaida Technologies SA); Kathryn Marable (Annaida Technologies); Carl Michal (UBC); Gaurasundar Conley (Annaida Technologies); Ronald Soong (University of Toronto Scarborough); 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); Andre Simpson (University of Toronto)

The superior mass sensitivity offered by micro-coils can be an excellent choice for tiny, mass limited, but environmentally important, samples such as eggs (<400 µm) and small organisms. However, despite the advantages offered by micro-coils, throughput can still be low for complex biological samples. Here, recent work addressing the challenges of microcoil arrays is discussed, laying the foundation for high throughput multiple coil arrays. Namely, complementary metal oxide semiconductor based micro-coils, low-cost adapted receiver boards and specialized pulse sequences were successfully applied to create "receive-only" planar micro-coil arrays used to study D. magna eggs, and current work involves expanding array sizes and applying them to metabolomics studies. Overall, high-throughput microcoil arrays have great potential in the field of environmental NMR.

POSTER 118

Advancements in microchip-based: bridging sensitivity and sample-handling for nanoliter samples

Presenter: Gaurasundar Conley (Annaida Technologies SA)
All Authors: Marco Grisi (Annaida Technologies SA); Giulia Sivelli (Annaida Technologies SA); Kathryn M. Marable (Annaida Technologies SA); Guillaume M. Gruet (Annaida Technologies SA); Gaurasundar M. Conley (Annaida Technologies SA)

Recent advancements in microscale NMR, particularly through microchip-based hardware, have sparked interest due to their state-of-the-art sensitivity and robust, versatile design. Here we discuss how microchips and advanced micro-engineering can create a new Magnetic Resonance platform, facilitating previously unreachable applications. Our approach integrates a flexible, low-cost microchip-based NMR sensor, enhancing sensitivity and reducing sample volumes. These systems, featuring CMOS-based designs, 3D micro-printing, and compatibility with standard NMR spectrometers, are ideal for high-precision monitoring of nanoliter cell cultures and early mammalian embryos. Their potential applications in classification and characterization studies of cell cultures are significant. We here highlight the system's architecture, scalability, and show its use on early mammalian embryos and oocytes, discussing future prospects in the field.

POSTER 119

Transforming NMR Probe Heads into Sensors for Multi-Diagnostic Dielectric+NMR Spectroscopy

Presenter: Alysson Morais (Katholieke Universiteit Leuven)
All Authors: Alysson Morais (Katholieke Universiteit Leuven); Sambhu Radhakrishnan (NMRCoRe, KU Leuven); Gavriel Arbiv (NMRCoRe, KU Leuven); Dirk Dom (NMRCoRe, KU Leuven); Karel Duerinckx (NMRCoRe, KU Leuven); Vinodchandran Chandrasekharan (Chandrasekharan); Johan Martens (NMRCoRe, KU Leuven); Eric Breynaert (NMRCoRe, KU Leuven)

Introduction of a sample in an NMR coil modifies the frequency response of the probe circuit, a phenomenon revealed by the detuning of the probe seen from the wobble curve. This contribution will demonstrate that this detuning - 'the dielectric shift' – can be used to calibrate an NMR probe head for dielectric permittivity measurements, transforming NMR probe heads into multi-diagnostic sensors able to simultaneously perform NMR and dielectric spectroscopy. As a proof of concept, this method was evaluated on a series of aliphatic alcohols using commercial CPMAS probe heads. Subsequently, the method was applied to investigate the solvent-surface interactions of water confined in the pores of a series of microporous and mesoporous silicate materials with tuned surface chemistry.

POSTER 120

Integrated Low-Field Polarimetry Device for Hyperpolarized Contrast Media Applications

Presenter: Clementinah Oladun (Wayne State University)
All Authors: Clementinah Oladun (Wayne State University); M Firoz Ahmed (Shahjalal University of Science and Technology); Md Raduanul Chowdhury (Wayne State University); Isaiah Adelabu (Wayne State University); Abubakar Abdurraheem (Wayne State University); Panayiotis Nikolaou (Xeus Technologies LTD); Anton Shcherbakov (Customs Medical Systems Ltd); Boyd Goodson (Southern Illinois University); Eduard Chekmenev (Wayne State University)

The use of hyperpolarized contrast agents has helped to overcome the drawbacks of low sensitivity associated with low-field (LF) NMR. We report on a portable, integrated, low-field polarimetry device for spectroscopic characterization of hyperpolarized contrast agents. This polarimetry device has been successfully utilized for the polarimetry studies and relaxation dynamics of HP Xenon-129 as well as HP [1-13C]pyruvate at 41 kHz. Moreover, this device allows for fully automated and integrated polarimetry studies including self-calibration, polarization quantification and relaxation dynamics. This device is compatible with diverse hyperpolarization techniques and contrast agents for various applications of polarization quantification. This device can also be useful for process optimization and quality assurance which is crucial for HP contrast agent production and administration.

POSTER 121

Compact cryogen-free 400 MHz (9.4 T) Solid state MAS NMR system for multi-field research

Presenter: Jeremy Good (Cryogenic Ltd)
All Authors: Eugeny Kryukov (Cryogenic Ltd); Denis Langlais (Cryogenic Ltd); Alexander Karabanov (Cryogenic Ltd); Paul Jonsen (Talaverascience Ltd); Jeremy Good (Cryogenic Ltd)

We present an NMR system for the use at different magnetic fields up to 9.4 T. The field stability and homogeneity meet the requirements for high-resolution solid state NMR. The system comprises a cryogen-free magnet, a Phoenix HX NMR 4 mm MAS probe, main and shim coils power supplies and a Tecmag Redstone NMR console. The compact

system design is convenient for laboratories with limited space. In the event of a magnet quench, the cryocooler recovers the superconducting state. The absence of cryogenic liquids prevents the risk of suffocation. We also developed a method for the fast post-ramp field stabilization that enables the field to be changed every day without compromising the data resolution.

POSTER 122

Autonomous reaction self-optimization using in-line high-field NMR spectroscopy

Presenter: Nour El Sabbagh (Nantes Universite, CNRS, CEISAM UMR 6230, F-44000 Nantes, France)

All Authors: Nour El Sabbagh (Nantes Universite, CNRS, CEISAM UMR 6230, F-44000 Nantes, France); Margherita Bazzoni (University of Nantes); Yuliia Horbenko (Nantes Universite, CNRS, CEISAM UMR 6230, F-44000 Nantes, France); Aurélie Bernard (Nantes Universite, CNRS, CEISAM UMR 6230, F-44000 Nantes, France); Daniel Cortés-Borda (Nantes Universite, CNRS, CEISAM UMR 6230, F-44000 Nantes, France); Patrick Giraudeau (Nantes Universite, CNRS, CEISAM UMR 6230, F-44000 Nantes, France); François-Xavier Felpin (Nantes Universite, CNRS, CEISAM UMR 6230, F-44000 Nantes, France); Jean-Nicolas Dumez (Nantes Universite, CNRS, CEISAM UMR 6230, F-44000 Nantes, France)

The integration of high-field NMR with autonomous self-optimization in flow chemistry has the potential to transform reaction optimization. Our approach, using high-field NMR, offers higher structural and quantitative insights compared to conventional methods like HPLC or benchtop NMR. By connecting a custom flow reactor to a 500 MHz spectrometer through a commercial flow tube, we established a setup that analyzes reaction mixtures in real-time. To autonomously control the system, we developed MATLAB-based programs and associated graphical user interfaces that handle flow synthesis, NMR experiments and optimization. Using 1D 1H NMR spectroscopy, we optimized the yield of a formal [3+3] cycloaddition within a single day. Our methodology promises enhanced precision, and efficiency in optimizing chemical synthesis, particularly for complex reaction mixtures.

POSTER 123

Large bore, closed-cycle MRI system for greenhouse-based plant root imaging

Presenter: Hilary Fabich (ABQMR, Inc.)

All Authors: Hilary Fabich (ABQMR, Inc.); Stephen A. Altobelli (ABQMR, Inc.); Mark Conradi (Abqmr, Inc.); Peng Lei (ABQMR Inc.); Matt Rosen (MGH/Martinos Center)

It is well-known that the plant root structure plays an important role in supporting plants, especially in hotter, drier soils. However, studying root architecture has been a challenge as the architecture changes in different soil types and soils are opaque. Typically, root systems must be washed before analysis which eliminates important information about root branching angle. This MRI system is a greenhouse deployable device that has been built to image plant roots in natural soils. The magnet is a closed-cycle, superconducting system with printed circuit board gradient coils and a saddle RF coil. This is a vertical bore magnet with a 20 cm diameter imaging region. The acquired images will be valuable in plant breeding efforts.

POSTER 124

Integrating NMR to the Nano and Microtomography Beamline at Sirius, the Brazilian Synchrotron Light Source

Presenter: Tito Jose Bonagamba (Sao Carlos Institute of Physics - University of Sao Paulo)

All Authors: Everton Lucas-Oliveira (Brazilian Synchrotron Light Laboratory (LNLS), Brazilian Center for Research in Energy and Materials (CNPEM)); Nathaly Lopes Archilha (Brazilian Synchrotron Light Laboratory (LNLS), Brazilian Center for Research in Energy and Materials (CNPEM)); Vitor Soares (Brazilian Center for Research in Energy and Materials (CNPEM)); João Henrique Ramos Silva (Brazilian Center for Research in Energy and Materials (CNPEM)); Edson Luiz Gea Vidoto (Sao Carlos Institute of Physics - University of Sao Paulo); Aparecido Donizeti Fernandes de Amorim (Sao Carlos Institute of Physics - University of Sao Paulo); Arthur Gustavo de Araujo-Ferreira (Sao Carlos Institute of Physics - University of Sao Paulo); Tito Jose Bonagamba (Sao Carlos Institute of Physics - University of Sao Paulo)

The Brazilian Synchrotron Light Laboratory (LNLS) is part of the Brazilian Center for Research in Energy and Materials, an organization under the supervision of the Brazilian Ministry of Science, Technology, and Innovations. LNLS is responsible for operating Sirius, one of the world's most advanced fourth-generation synchrotron light sources.

One of the LNLS beamlines is Mogno, dedicated to obtaining 3D images of materials at multiscale, quickly and non-invasively. In order to complement the measurements, a low-field NMR system is being coupled to the Mogno beamline to carry out simultaneous measurements. Among the first materials to be studied, porous media saturated with static fluids or in flow conditions that impose physicochemical changes on these materials stand out.

POSTER 125

Parallel Tube Recupertors (PTRs) to Enable Routine, Affordable Ultra-low-temperature (ULT) MAS-DNP

Presenter: F. David Doty (Doty Scientific, Inc.)

All Authors: F.David Doty (Doty Scientific, Inc.); Scott Deese (Doty Scientific, Inc); JB Spitzmesser (Doty Scientific, Inc.); Glenn N. Doty (Doty Scientific, Inc.); John Staab (Doty Scientific, Inc.); Judy M. Doty (Doty Scientific, Inc.); Daniel Arcos (Doty Scientific, Inc.); Laura Holte (Doty Scientific, Inc.); Paul D. Ellis (Doty Scientific, Inc.)

We report progress on the development of a novel approach toward ULT NMR probe (with or without DNP, and with or without MAS) that is compatible with the standard helium recycling systems. The probe is expected to permit routine operation down to 8 K and 20K for static NMR and MAS-DNP, respectively. The combination of ULT capability and a novel high-mode THz cavity promises to make high-field DNP possible using solid-state sources instead of gyrotrons. The over-arching objective of this development effort is to dramatically reduce entry level cost into ULT NMR and NMR-DNP, both static and MAS, while reducing certain operational challenges. S/N gains from the combination of ULT and DNP could exceed a factor of 10,000.

POSTER 127

Tools for Optimizing MAS-DNP NMR through Hardware: Probe, Cooling Cabinet, and Microwave Customizations

Presenter: Faith Scott (National High Magnetic Field Laboratory)

All Authors: Faith Scott (National High Magnetic Field Laboratory); Jason Kitchen (National High Magnetic Field Laboratory); Steven Ranner (National High Magnetic Field Laboratory); Wenping Mao (National High Magnetic Field Laboratory); Peter Gor'kov (National High Magnetic Field Laboratory); Robert Schurko (FSU and NHMFL); Thierry Dubroca (National High Magnetic Field Laboratory); Joanna Long (University of Florida); Frederic Mentink-Vigier (National High Magnetic Field Laboratory, Florida State University)

Hardware is often overlooked as an easy means of increasing Magic Angle Spinning-Dynamic Nuclear Polarization (MAS-DNP) performance. I will present the design and performance of a new probe custom-built at NHMFL along with other improvements to the DNP spectrometer. Our probe features "tuning card" traps for better RF (Radio Frequency) efficiency. Microwave performance has been achieved through a custom-built corrugated waveguide and tailored Teflon microwave lens. Two new 1.3 mm and 1.9 mm probes built at NHMFL feature a new vacuum-jacketed internal transfer line set built in-house to improve the cryogenic and spinning capabilities. Finally, we will show methods for increasing spinning speed at 100 K on our 3.2 mm stator probe.

POSTER 128

OPTO: Software for Objective Optimization of Solid-state NMR Parameters

Presenter: Collin Borcik (Department of Biochemistry, University of Wisconsin-Madison)

All Authors: Collin Borcik (Department of Biochemistry, University of Wisconsin-Madison); Barry DeZonia (National Magnetic Resonance

Facility at Madison, University of Wisconsin-Madison); Benjamin D. Harding (Department of Biochemistry, University of Wisconsin-Madison); Rajat Garg (Department of Biochemistry, University of Wisconsin-Madison); Chad M. Rienstra (Department of Biochemistry, University of Wisconsin-Madison)

Solid state NMR experiment parameter setup is vital to achieving high quality spectra with a high signal to noise ratio, but parameter setting is time intensive to the operator and does not necessarily identify an objective optimal condition. We introduce OPTO, a program that performs objective optimization to robustly improve parameters for shimming and cross polarization (CP) for single and multidimensional NMR experiments. OPTO drives spectrometer controlling software to organize, track, and update parameters during operation, freeing operator time and attention. We demonstrate OPTO's efficiency at automated shimming and optimization of CP parameters on micro-crystalline GB1 and Alpha-synuclein fibril samples.

POSTER 129

Automation in solid state NMR with half integer quadrupole nuclei

Presenter: Sebastian Wegner (Bruker BioSpin)
All Authors: Christof Oliver Johann (Bruker BioSpin GMBH); Jochem Struppe (Bruker BioSpin)

Automation in solid state NMR is meanwhile mostly technically mature. We focus herein on automating experiment setup. We discussed recently how using radio frequency (rf) fields, which are key parameters in solid state NMR experiments can be used for easy and fully automated setup for spin ½ nuclear systems. Here we want to propose this method as tool for rapid setup of experimental conditions for half integer quadrupole nuclei. An important step forward in this general approach is the use of internal rf-field references. The figure shows the optimization of 31P{27Al} CPMAS conditions. The method permits easy and quick setup and optimization of more complicated heteronuclear correlation NMR experiments like the PT-D/J-HMQC HX or HXY experiments.

POSTER 130

Detailed Characterization, Optimization and Quantification of Signal and Noise for the Integration of Instrumentation

Presenter: Alexandria Guinness (Syracuse University)
All Authors: Alexandria Guinness (Syracuse University); Alec Beaton (Syracuse University); John Franck (Syracuse University)

A robust and versatile methodology to quantify, isolate and identify the various sources of noise, and quantify signal is presented in an insightful protocol. The quantification, isolation and identification of noise sources is performed using open-source programming, common off-the-shelf components and/or the NMR console. Signal averaging and post-processing methods yield unique and valuable plots of the noise PSD that have not previously been seen in literature. Such plots yield detailed information which the spectroscopist can act on to improve subsequent measures. Quantification of the absolute signal is derived by reorganizing the standard theory to focus on the (power-to-field) conversion factor as the fundamental measure of sensitivity, and further disentangling the influences of the field distribution factor vs. the Q-factor.

POSTER 131

Monitoring Wood Drying by NMR

Presenter: Yi-qiao Song (Harvard University)
All Authors: Guang Yang (Harvard University); Chandra Shekar (Harvard University); Yi-qiao Song (Harvard University); Donhee Harvard (Harvard University)

Wood products, crucial in construction, undergo kiln drying to eliminate free water, but prolonged exposure can alter structures, causing shrinkage and warping. Quantitative water content monitoring during drying is vital to prevent overexposure and reduce energy consumption, which significantly contributes to CO2 emissions. Utilizing a miniature NMR system with a semiconductor RF transceiver, 1H T2 relaxation times were measured during the drying process for different wood species. The T2 distributions revealed trends: a decrease in overall signal as the sample dried and a faster loss of free water compared to bound water. The NMR system proves effective in monitoring wood moisture, distinguishing free and bound water, offering real-time feedback for optimal drying.

POSTER 132

A tachometer for fast magic angle spinning

Presenter: Wenping Mao (National High Magnetic Field Laboratory)
All Authors: Wenping Mao (National High Magnetic Field Laboratory); Peter Gor'kov (National High Magnetic Field Laboratory)

Fast sample spinning has enabled direct and easy observation of high-resolution 1H NMR in solids. The way to sustainably increase spinning rate is to reduce rotor diameter. As rotor diameter reduces to 1.3 mm, 0.7 mm and below, accurate spinning detection could become an issue due to weaker light contrast. We present a circuit with a bandwidth of 1 MHz for the detection of fast MAS beyond 60 kHz. The circuit outputs spin signal in either TTL or pulse with specific low and/or high voltages as required by some MAS controllers.

POSTER 133

Impact of Center Frequency and Pulse Duration on 13C NMR Quantitation

Presenter: Joseph Vasquez (Dow)
All Authors: Joseph Vasquez (Dow); Nathan J. Rau (Dow); Daisy Kimenai (Dow); Caden Bosma (Dow); XiaoHua (Sam) Qiu (Dow); Xiaohong (Susan) Zong (Dow); Benjamin R. Reiner (Dow)

Cryoprobes that display a biased response, where peaks of nuclei that resonate upfield from the center frequency are observed with more or less signal than those that resonate downfield from the center frequency, had the center frequency and the pulse duration optimized to provide a solution for obtaining quantitative data with as wide a bandwidth and as much signal as possible.

POSTER 134

Zero-field NMR in natural isotopic abundance with multichannel modality

Presenter: Blake Andrews (University of California, Berkeley)
All Authors: Blake Andrews (University of California, Berkeley); Matthew Lai (University of California, Berkeley); Norihisa Kato (Hamamatsu Corporation); Zhen Wang (University of California, Berkeley); Emanuel Druga (University of California, Berkeley); Ashok Ajoy (UC Berkeley)

Zero-field (ZF) NMR is an emerging technique that offers structural information similar to conventional NMR by detecting analytes at zero magnetic field following preparation at high magnetic field. We report the first ZF NMR apparatus with a 9.4T inhomogeneous superconducting magnet. Having made significant progress towards solving sensitivity issues, we report organic compounds measured at zero-field in natural isotopic abundance and without hyperpolarization. We aim to extend our current system with multichannel capabilities, having successfully measured 3 distinct enriched chemical samples simultaneously as proof of concept as well as characterizing the crosstalk (the extent to which one sensor's spectrum "bleeds" into the other) as a function of the sensors' spatial separation.

POSTER 135

National Resource for Advanced NMR Technology: Increasing NMR sensitivity to enable characterization of biomolecular systems and advanced materials

Presenter: Ilya Litvak (National High Magnetic Laboratory, Florida State University)
All Authors: Ilya Litvak (National High Magnetic Laboratory, Florida State University); Frederic Mentink-Vigier (National High Magnetic Field Laboratory, Florida State University); Zhehong Gan (National High Magnetic Field Laboratory, Florida State University); Faith Scott (National High Magnetic Field Laboratory); Amrit Venkatesh (National High Magnetic Field Laboratory, Florida State University); Wenping Mao (National High Magnetic Field Laboratory); Matthew Merritt (University of Florida); William Brey (Florida State University); Joanna Long (University of Florida); Robert Schurko (FSU and NHMFL)

The National Resource for Advanced NMR Technology (NIH RM1 GM148766) is developing new NMR technologies for research on biomolecules and advanced materials, including: (i) solution NMR probes with high temperature superconductor (HTS) resonators that provide unmatched sensitivity for metabolomics studies; (ii) novel and optimized MAS-DNP ssNMR approaches and sample protocols that maximize DNP sensitivity; and (iii) molecular-level characterization via ultra-high field ssNMR (i.e., the 36 T/1.5 GHz series-connected hybrid (SCH) magnet) and world-unique probes, with applications to quadrupolar nuclei in materials that have never been probed with NMR. All of these technologies can be accessed through the NHMFL NMR/MRI User Program. Information on collaborations, workshops, ssNMR protocols, NMR probe development, and online tutorials can be found at our website: NMRprobe.org.

POSTER 136

The Network for Advanced NMR at UW-Madison: 1.1 GHz Solid-State NMR and Knowledgebases

Presenter: Alexander Paterson (University of Wisconsin-Madison)

All Authors: Alexander Paterson (University of Wisconsin-Madison); Songlin Wang (University of Wisconsin-Madison); Arthur Edison (University of Georgia); Jeffrey Hoch (UConn Health); Katherine Henzler-Wildman (UW-Madison); Chad Rienstra (University of Wisconsin-Madison)

The mission of NAN, the Network for Advanced NMR, is to provide state-of-the-art NMR instrumentation to investigators. One 1.1 GHz NMR spectrometer has recently been installed at UW-Madison for SSNMR and is already generating data.

NMRFAM is developing knowledgebases focused on biological and materials SSNMR. These knowledgebases demonstrate best practices, provide SOPs for reproducible data acquisition, and facilitate implementation of new experiments. Expert content includes pulse sequences, example data, and instrument files, along with processing protocols for validation. Non-expert content demystifies NMR for scientists in other fields, demonstrates research questions that NMR can address, and explores practical considerations for addressing if NMR is appropriate for their research. NAN is seeking feedback on existing protocols and community demand for new knowledgebases.

POSTER 137

High-Speed, High-Memory NMR Spectrometer and Hyperpolarizer

Presenter: William Beatrez (University of California, Berkeley)

All Authors: William Beatrez (University of California, Berkeley); Jason Ball (Tabor Electronics); Emmanuel Druga (University of California, Berkeley); Joan Mercade (Tabor Electronics); Mark Elo (Tabor Electronics); Ashok Ajoy (University of California, Berkeley)

We report on the development of a novel nuclear magnetic resonance (NMR) spectrometer, incorporating a high-speed, commercially available arbitrary waveform transceiver (AWT). The spectrometer is optimized for integrated electron-nuclear spin control and dynamic nuclear polarization. High sampling rates permit NMR pulse synthesis and signal reception directly at the Larmor frequency up to 3GHz. Additionally, the spectrometer features on-board, phase-sensitive detection, enabled by numerically controlled oscillators (NCO); and windowed acquisition can be carried out over extended periods and across millions of pulses. The device is compact and rack-mountable, highly cost-effective with respect to contemporaneous spectrometers. It opens up new avenues for NMR pulse control and DNP, including closed-loop feedback control, electron decoupling, 3D spin tracking, and potential applications in quantum sensing.

POSTER 138

A Low-cost Method to Optimize Spatial Resolution on Single-sided Magnets

Presenter: Tyler Meldrum (William & Mary)

All Authors: Tyler Meldrum (William & Mary); Samuel Rubin (William & Mary, Department of Chemistry); Shinjer Li (College of William and Mary Department of Chemistry)

Single-sided NMR instruments facilitate non-destructive analyses, both relaxometry and diffusometry, of samples with very few restrictions on sample geometry. In addition, their strong field gradient makes these instruments suitable for a range of 1D imaging or "profilometry" experiments. To optimize the spatial resolution of profilometry experiments, the sample and sensitive volume of the magnet need to be co-planar to high precision. We describe a low-cost method to optimize co-planarity of the sample and the sensitive volume using gradient coils.

POSTER 139

NMRFAM User Program

Presenter: Paulo Falco Cobra (NMRFAM)

All Authors: Paulo Falco Cobra (NMRFAM); Marco Tonelli (NMRFAM); Songlin Wang (University of Wisconsin-Madison); Alexander Paterson (University of Wisconsin-Madison); Thirupathi Ravula (Univeristy of Wisconsin-Madison); Chad Rienstra (University of Wisconsin-Madison); Katherine Henzler-Wildman (UW-Madison)

The NMRFAM user program provides access to 12 NMR spectrometers equipped for a variety of solution and solid-state NMR experiments. Our scientists provide advice and assistance in experimental design, data acquisition and data 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. All our solution spectrometers are equipped with cryoprobes, including a HFCN and a HPCN QCI cryoprobes, and a 1.7 mm HCN cryoprobe. Our 500 MHz and one 600 MHz are equipped with samplejet for automated data collection. We have a range of MAS probes from 0.7 mm to 5 mm optimized for both biological and materials samples.

POSTER 140

Benchtop LC Circuit Apparatus for Probing RF Coil Homogeneity

Presenter: Jose Uribe (UC Irvine)

All Authors: Jose Uribe (UC Irvine); Matthew Jimenez (UC Irvine); Rachel W. Martin (UC Irvine)

Achieving homogenous radiofrequency (rf) magnetic fields in solid-state NMR transceiver coils is essential for maximizing sensitivity during experimentation. An automated method that uses inexpensive and open-source equipment to create a modular, yet specialized tool, is presented, the Auto-Ball Shift (ABS). In combination with our "probe in a box", the sample coil can be tested outside of the NMR probe while simulating the frequency dependent B1 fields. This apparatus was built with a dual resonance balanced LC circuit. With this hands-free method we can obtain the rf homogeneity map before soldering the transceiver coil onto the NMR probe circuit.

POSTER 141

Implementation of Machine Learning in Assisting NMR Microcoil Design Optimization

Presenter: Bing Wu (Radboud University Nijmegen)

All Authors: Bing Wu (Radboud University Nijmegen)

Machine-learning-assisted optimization (MLAO) has recently been widely introduced to accelerate the design process of telecommunication antennas. Machine learning (ML) methods, including Gaussian process regression, support vector machine (SVM), and artificial neural networks (ANNs), have been applied to build surrogate models of antennas to achieve fast response prediction. With the help of these ML methods, various MLAO algorithms have been proposed for different applications.

In this study, we first introduce this concept into designing an RF micro coil for NMR application. Using MLAO, several RF coil designs were theoretically optimized for samples with different geometries. Some simple RF micro-coils are also manufactured to validate the simulation.

POSTER 142
Diamond Rotors
Presenter: Robert Griffin (Massachusetts Institute of Technology)
All Authors: Lauren Schaffer (Massachusetts Institute of Technology), Robert Griffin (Massachusetts Institute of Technology)
Single crystal diamond rotors can enable unprecedented advances in both the

sensitivity and resolution of magic angle spinning (NMR under ambient and

DNP conditions) Diamond has extremely high tensile and elastic moduli, is nearly transparent at THz frequencies, and excellent thermal conductivity While diamond is an optimal material for DNP MAS rotors, significant fabrication challenges have prevented the realization of diamond rotors. We have refined our previous laser machining process fabrication 0 7 mm diamond rotors with improved stability. We demonstrate MAS results using the MAS3 0.7 mm automatic spinning profile with linear correlation between drive gas and spinning frequency as well as stability of 6 separate rotors at 111 kHz with a standard deviation 4 Hz.

POSTER 143
A Ground-Up and Integrated Approach to Overhauser-Dynamic Nuclear Polarization
Presenter: John Franck (Syracuse University)
All Authors: John Franck (Syracuse University); Alec Beaton (Syracuse University); Alexandria Guinness (Syracuse University); Warren Kincaid (Syracuse University)
In this contribution, we focus particularly on two aspects of ODNP development. First, because reverse micelles (RMs) can confine water to nanometer lengthscales, and because the translational dynamics of water responds dramatically to confinement, changing the size of the RMs alter the ODNP response by two orders of magnitude. Both PRE-ROSY and ²H ROSY complement the ODNP measurements of the confined water inside the RMs to provide an excellent benchmark measurement. Second, a focus on improving the sensitivity of the NMR experiment in the instrumentally adverse environment imposed by ODNP has led, and will continue to lead, to improved sensitivity of the ODNP measurement that will enable full dynamics studies at very low concentrations and/or low levels of water dynamics.

POSTER 144
Home-Built 1HXY MAS DNP Probes for 600 MHz DNP Spectrometer
Presenter: Peter Gorkov (National High Magnetic Field Laboratory)
All Authors: Peter Gorkov (National High Magnetic Field Laboratory)
We report construction of home-built MAS DNP 3-resonance 1HXY probes for our 600 MHz external user DNP facility. These probes are utilizing MAS spinners and several components purchased from Bruker to ensure compatibility with their LTMAS cabinet. The internal parts of the probe frame, however, were redesigned to house more versatile and sensitive radio circuit, different microwave waveguide and lens design, and to cool probe more efficiently with reduced internal cold volume. Our improved design of internal vacuum-jacketed transfer line will be presented. The 1HXY RF circuit utilizes swappable tune cards, with flexibility to run 3-resonance experiments in multiple XY isotope combinations. Its twice higher 1H channel sensitivity, in comparison to similar Bruker probe, fares favorably for 1H detection experiments.

Metabolomics (Posters 145-158)

POSTER 145
Advancing operando NMR techniques to study degradation processes in commercial Li metal batteries
Presenter: Asya Svirinovsky Arbeli (Columbia University)
All Authors: Asya Svirinovsky Arbeli (Columbia University); Yongbeom Kwon (Columbia University); Piotr Lepucki (ePROBE GmbH); Oliver Pecher (ePROBE GMbH); Lauren Marbella (Columbia University)
Despite extensive Li metal anode research, achieving high Coulombic efficiency remains challenging. Understanding the correlation between surface composition, internal cell pressure, electrolyte, and electrochemical treatment in practical batteries is elusive. Nuclear magnetic resonance (NMR) spectroscopy offers a non-invasive unique approach to explore these factors, providing quantitative structural, chemical, and dynamical insights. Performing operando NMR on practical batteries faces challenges due to metal casing interactions, including bulk magnetic susceptibility, reduced sensitivity, and skin depth. Here I show methods to mitigate signal attenuation and manage magnetic interactions in commercial cells using novel hardware. These operando NMR measurements capture plating and stripping instabilities, monitor 'dead' Li formation, and investigate pre-cycling electrolyte evolution in multilayer pouch cells and coin cells.

POSTER 146
Application of 15N-Edited 1H-13C Correlation NMR Spectroscopy - Toward Fragment-Based Metabolite Identification and Screening via HCN Constructs
Presenter: William Wolff (University of Toronto)
All Authors: Daniel Lysak (University of Toronto); William Wolff (University of Toronto); Ronald Soong (University of Toronto); Wolfgang Bermel (Bruker Biospin); Eriks Kupce (Bruker UK Ltd); Amy Jenne (University of Toronto); Rajshree Ghosh Biswas (University of Toronto); Daniel Lane (University of Toronto); Genevieve Seabrook (University Health Network-Princess Margaret Hospital); Andre Simpson (University of Toronto)
Nitrogen containing functional groups are common in many important metabolites, yet despite their prevalence and the importance of nitrogen-containing compounds, 15N NMR experiments have been uncommon for metabolomics research. This has limited the impact of existing pulse sequences in metabolomics that directly correlate 1H-15N over their respective JNH coupling constants, as extensive metabolomics databases use 1H-13C correlations, and often lack 15N shifts completely. Here, a 15N-edited HSQC is presented that allows HCN moieties to be indirectly accessed by selecting 1H-13C correlations bound only to adjacent 15N groups. This greatly improves the number of assignable peaks in [15N, 13C]-labelled samples, and permits a facile route to extend 1H-13C databases to 15N chemical shifts in combination with the 15N-HMBC.

POSTER 147
Heteronuclear NMR and 13C Statistical TOCSY to Unravel In Vivo Processes Without Enrichment
Presenter: Peter Costa (University of Toronto)
All Authors: Peter Costa (University of Toronto); Daniel Lysak (University of Toronto); Ronald Soong (University of Toronto); Kiera Ronda (University of Toronto Scarborough); Katelyn Downey (University of Toronto); William Wolff (University of Toronto); Katrina Steiner (University of Toronto); Vincent Moxley-Paquette (University of Toronto); Jacob Pellizzari (University of Toronto); Clemens Anklin (Bruker BioSpin); Gary Sharman (Mestrelab Research); Carlos Cobas (Mestrelab Research); Santi Domínguez (Mestrelab Research); Andre Simpson (University of Toronto)
In vivo NMR is an effective, non-invasive analytical technique to study organisms in their native and unaltered state. Typical experiments utilize 13C enriched organisms to enhance sensitivity and resolution. However, with rising costs and difficulty in upkeep, the long-term study of 13C enriched organisms is economically unviable. This study demonstrates the application of heteronuclear NMR techniques in combination with 13C Statistical Total Correlation Spectroscopy as effective methods to assess and visualize changes in organisms without enrichment. A 10 mm probe is utilized to increase the amount of biomass available, compared to a 5 mm probe. When applied to in vivo NMR, these techniques provide excellent resolution to identify temporal changes in key metabolites, providing greater insight into complex biological processes.

POSTER 148
Tracing 12C/13C in biological samples using a 2D IPAP-TOSCY isotope discrimination experiment

Presenter: Katrina Steiner (University of Toronto)
All Authors: Katrina Steiner (University of Toronto); Wolfgang Bermel (Bruker Biospin GmbH); Ronald Soong (University of Toronto Scarborough); Daniel Lysak (University of Toronto); William Wolff (University of Toronto); Katelyn Downey (University of Toronto); Peter Costa (University of Toronto); Jacob Pellizzari (University of Toronto); Kiera Ronda (University of Toronto Scarborough); Venita Busse (Bruker Switzerland AG); Benjamin Goerling (Bruker Biospin GmbH); Falko Busse (Bruker Switzerland AG); Colin Elliott (Bruker Biospin GmbH); Agnes Haber (Bruker Biospin GmbH); Andre Simpson (University of Toronto)

This presentation introduces a 2D isotope filtered in-phase anti-phase (IPAP) total correlation spectroscopy (TOCSY) experiment. The IPAP TOCSY works by producing four sub-spectra: (1) 13C-13C, (2) 13C-12C, (3) 12C-13C, and (4) 12C-12C which can be used to identify site specific information within intact biological samples. The presentation demonstrates the applications of the 2D IPAP TOCSY on standard mixtures, and its ability to monitor biological processes at 500 MHz and 80 MHz. These processes include yeast fermentation using 1-13C glucose which incorporates the anomeric 13C into ethanol. 13C enrichment inside Daphnia magna (freshwater fleas) fed a 13C enriched diet will also be demonstrated. The IPAP TOCSY has significant isotopic tracing applications across various fields including environmental, toxicological, and medical.

POSTER 149
Unstable Coenzymes and their Anomalous Dynamics in Cold Blood: New Insights from 1H NMR Spectroscopy
Presenter: G. A. NAGANA GOWDA (University of Washington)
All Authors: G. A. NAGANA GOWDA (University of Washington); Vadim Pascua (University of Washington); Dan Raftery (Seattle)

Efforts in our laboratory have enabled analysis of >100 metabolites using simple 1D NMR in human blood, which includes the unstable energy coenzymes, redox coenzymes and antioxidants that are fundamental to cellular functions. Further investigations using 1H NMR showed surprisingly anomalous dynamics of these coenzymes in blood stored on ice, which is contrary to the current knowledge about metabolite stability. Such dynamics potentially arises from the active cellular metabolism. From the metabolomics perspective, the massive variation of the labile metabolite levels in cold blood is alarming and stresses the critical need to quench the cellular metabolism for reliable analyses. Overall, the results provide compelling evidence that warrants a paradigm shift in sample collection protocol for blood metabolomics involving labile metabolites.

POSTER 150
A-SIMA/A-MAP: A Comprehensive Methodology for NMR-based Metabolomics Analysis
Presenter: Abigail Chiu (University of Colorado Denver)
All Authors: Abigail Chiu (University of Colorado Denver); Mehdi Rahimi (University of Colorado Denver); Woonghee Lee (University of Colorado Denver)

As the prominence of metabolomics grows, advancements and versatility in analytical functionalities remain critical to keep up with the research demand. Current tools not only lack in developmental strides, but also user-friendly features. Thus, we proudly introduce two cutting-edge tools, A-SIMA: Advanced-Software for Interactive Metabolite Analysis and A-MAP: A Multivariate Analysis Program to improve the overall methodology of NMR-based metabolomics. With an easy-to-use graphical user interface, A-SIMA allows for users to perform NMR metabolite identification on 1D and 2D NMR data via two modes, "Computer Assisted" and "User Based". A-MAP performs multivariate analysis on user's NMR data via Principal Component Analysis and Orthogonal Partial Least Squares-Discriminant Analysis. Both programs are pre-built in the POKY suite.

POSTER 151
13C Steady-State Free Precession for Sensitivity Enhancement of Environmental Samples Using High- and Low-Field NMR Spectroscopy
Presenter: Katelyn Downey (University of Toronto)
All Authors: Katelyn Downey (University of Toronto); Peter Costa (University of Toronto); Kiera Ronda (University of Toronto Scarborough); Ronald Soong (University of Toronto Scarborough); Flavio Kock (University of Toronto Scarborough); Daniel Lysak (University of Toronto); William Wolff (University of Toronto); Katrina Steiner (University of Toronto); Jacob Pellizzari (University of Toronto); Agnes Haber (Bruker Biospin GmbH); Venita Busse (Bruker Switzerland AG); Falko Busse (Bruker Switzerland AG); Benjamin Goerling (Bruker Biospin GmbH); Tiago B. Moraes (Universidade de São Paulo); Luiz A. Colnago (Embrapa Instrumentação); Andre Simpson (University of Toronto)

NMR is a valuable complementary tool in environmental research, but the costs and esoteric operation of high-field NMR preclude widespread use. Low-field NMR is more accessible, but its lower sensitivity and increased spectral overlap limit effective environmental analysis, especially with fast-relaxing samples that produce broad signals. Here, 13C Steady-State Free Precession (SSFP) is used to enhance SNR of complex environmental samples (egg white, dissolved organic matter, crude oil) at high- and low-field, and in vivo 31P-SSFP is used to monitor earthworm death. SSFP increased 13C sensitivity by over 1500% and enabled detection of signals that were undetectable by standard 13C NMR. Ultimately, SSFP holds great promise for improving low-field NMR analysis of environmental samples with broad, low intensity spectra.

POSTER 152
13C-Depleted Algae as Food: Permitting Background Free In-vivo Nuclear Magnetic Resonance of Daphnia magna at Natural Abundance
Presenter: Jacob Pellizzari (University of Toronto Scarborough)
All Authors: Jacob Pellizzari (University of Toronto Scarborough); William Wolff (University of Toronto); Ronald Soong (University of Toronto Scarborough); Daniel Lysak (University of Toronto); Katrina Steiner (University of Toronto); Kiera Ronda (University of Toronto Scarborough); Peter Costa (University of Toronto); Katelyn Downey (University of Toronto); Vincent Moxley-Paquette (University of Toronto); Chris Suszczynski (ISOTEC Stable Isotope Division, Millipore Sigma); Steven Boehmer (ISOTEC Stable Isotope Division, Millipore Sigma); Jacob R. Prat (ISOTEC Stable Isotope Division, Millipore Sigma); Andre Simpson (University of Toronto)

In-vivo NMR is important for environmental applications, including the toxicity assessment of environmental pollutants. Researchers can identify the biochemical pathways impacted by stressors through 2D-NMR experiments (e.g., 1H-13C HSQC) of 13C-enriched organisms. 13C-enriched organisms are typically sustained in-vivo with algae at natural abundance 13C, which avoids spectral interference. Sensitivity improvements associated with novel NMR technologies increase the feasibility of in-vivo experiments on natural abundance organisms, however, generating a need for background-free food sources. To address this, we use algae grown using 99.99% 13C-depleted CO2. Algae grown under 13C-depleted conditions show a 99.87% reduction in signal after 21 hours on a TCI cryoprobe, compared to natural abundance algae. The presented 13C-depletion scheme broadens the scope of possible biological NMR experiments.

POSTER 153
Longitudinal HRMAS NMR Study of Human Prostate Cancer Metabolomics with Biopsies
Presenter: Leo Cheng (MGH Harvard Medical School)
All Authors: Ella J. Zhang (Massachusetts General Hospital); Jiaqi Lu (NYU); Chin-Lee Wu (Massachusetts General Hospital); Adam S. Feldman (Massachusetts General Hospital); Leo Cheng (MGH Harvard Medical School)

Standard prostate cancer (PCa) screening and diagnostic tools, including prostate specific antigen (PSA) testing and 12-core transrectal ultrasound (TRUS) biopsies, suffer from a lack of PCa-specificity and sensitivity, resulting in overtreatment of indolent PCa. Metabolomics, as an emerging field for cancer biomarker discovery, can provide new perspectives for evaluating PCa prognosis, aggressiveness, and clinical significance. Using high-resolution magic angle spinning (HRMAS) NMR, we analyzed 426 biopsy cores from

patients suspicious for PCa, with >15 years follow-up. Our metabolomic analyses (based on principal component and canonical discriminant analyses) were not only capable of distinguishing between PCa and benign tissues, but could also predict positive PCa diagnosis within 5 years of initial PCa-negative biopsy. Currently, evaluations of major contributing metabolites are underway.

POSTER 154

Python Software Tool for Automated Analysis of Blood Metabolites

Presenter: Vadim Pascua (University of Washington)

All Authors: Vadim Pascua (University of Washington); G. A. NAGANA GOWDA (University of Washington); Dan Raftery (Seattle)

We describe the development of automation software for analysis of metabolites using 1D NMR spectra. Written in Python, it integrates powerful libraries such as nmrglue, numpy, pandas, and matplotlib, harnessing their robust data processing and visualization capabilities. Using predefined peak regions and the concentration of internal standard reference compound, the software integrates peaks and determines absolute concentrations of metabolites in large data sets on the fly. Importantly, it enable analysis of spectra obtained at any magnetic fields. The software also incorporates a binning function for applications involving untargeted, global analysis using multivariate statistical methods. It is simple, easy to use, and requires minimal hardware, making it accessible to wide array users with standard computing resources.

POSTER 155

Modeling Blood Metabolite Homeostasis Reduces Unexplained Variance and Reveals Basal Metabolism Levels and Network Relationships

Presenter: Daniel Raftery (Seattle)

All Authors: Dan Raftery (Seattle); Danni Liu (University of California Irvine); G. A. NAGANA GOWDA (University of Washington); Zhongli Jiang (Purdue University); Kangni Alemdjrodo (Purdue University); Min Zhang (University of California Irvine); Dabao Zhang (University of California Irvine)

Blood metabolite levels are affected by numerous factors, including demographic, clinical, genetic, and pre-analytical factors such as collection methods and geographical sites, resulting in deleterious effects on biomarker studies. Here, we developed quantitative models of blood metabolite levels in healthy adults based on multisite sample cohorts to address this challenge. By modeling each of the 50 metabolites quantified using 1H NMR, we achieved a dramatic reduction in the site-to-site and sample to sample variation of metabolite levels (>95%, PC1 and PC2) using demographic and clinical factors and especially other metabolites. The study demonstrates an intriguing, cross-pathway network effect of metabolites that can be utilized to better define basal homeostatic metabolite levels, which may have implications for improved health monitoring.

POSTER 156

Investigation of cancer-stromal cells metabolic crosstalk with NMR metabolomics

Presenter: Teklab Gebregiworgis (University of Western)

All Authors: Fatema Bhinderwala (Univeristy of Pittsburgh); Pankaj K. Singh (University of Oklahoma Health Sciences Center); Robert Powers (University of Nebraska-Lincoln)

Pancreatic ductal adenocarcinoma (PDAC) is an aggressive and deadly disease characterized by a poor prognosis and limited treatment options. The PDAC tumor microenvironment is mainly composed of cancer-associated fibroblasts (CAFs), with CAFs accounting for 90% of the total tumor mass. There are conflicting reports on the role of CAFs in cancer progression and therapeutic response, necessitating an in-depth investigation to elucidate the crosstalk between CAFs and cancer cells. To address this, we developed a method that employs stable isotope-resolved NMR metabolomics to monitor the metabolite-based communication between cancer cells and stromal cells. The presentation will illustrate the use of NMR selective isotopes, cell culture arrangements, and the application of 2D 1H-13C NMR experiments to investigate cancer-stromal metabolite crosstalk.

POSTER 157

Simultaneous 1H NMR quantification of stable isotope enriched metabolite in multiplexed labeling Stable Isotope Resolved Metabolomics

Presenter: Penghui Lin (University of Kentucky)

All Authors: Penghui Lin (University of Kentucky); Ricard M. Higashi (University of Kentucky); Teresa W-M. Fan (University of Kentucky); Andrew N. Lane (University of Kentucky)

Stable isotope-enriched precursors are frequently used to trace the metabolic fate of different atoms, including 13C, 15N and 2H. Due to their low gyromagnetic ratios, these isotopes are relatively insensitive by direct NMR detection. Here we report a means to determine isotope fractional enrichment in multiplex labeling experiments by only analyzing the proton spectrum, utilizing the isotope shift peak patterns due to J-coupling to neighboring nuclei. By quantifying the split peaks, detailed isotopomer distributions of metabolites could be accurately determined. We showcase the applications of determining the fractional enrichments of 2H-Lac, 2H-Ace, 15N-ATP as well as 13C-enriched uracil in UTP, which are metabolites frequently quantified in metabolomic studies.

POSTER 158

1H-NMR metabolomics with chemometrics for detection of Mexican Apis Mellifera honey adulterations

Presenter: Nuria Esturau Escofet (Instituto de Qui-mica, UNAM)

All Authors: Andrea Montserrat Mier y Teran Lugo (Instituto de Quimica, UNAM); NURIA Esturau-Escofet (Instituto de Qui-mica, UNAM)

Mexico is the ninth greatest producer of honey in the world; unfortunately, the production is not enough to satisfy the high demand for this product leading to a growing occurrence of adulterated honey. To increase the production of honey, adulterations can be directly or indirectly with cheap sweeteners such as corn syrup, high fructose corn syrup, cane sugar syrup and inverted sugar syrup. In certain cases, honey is combined with water or apple cider vinegar to mimic honey produced by stringless bees.

The aim of this project is to develop a methodology using 1H-NMR with chemometrics to establish the metabolic profile differences of directly and indirectly adulterated Apis Mellifera honey with the most common adulterants.

MRI MRS (Posters 159-179)

POSTER 159

Metabolomic Profile Analysis of the Stability of Wine samples through 1H-NMR and Chemometrics Methods

Presenter: Martha Elena García Aguilera (Instituto de Química Universidad Nacional Autónoma de México)

All Authors: Martha Elena García Aguilera (Instituto de Química Universidad Nacional Autónoma de México); Eduardo Rodríguez de San Miguel Guerrero (UNAM); Francisco Ruiz Terán (UNAM); Ronna Delgado Altamirano (ANY); NURIA Esturau-Escofet (Instituto de Qui-mica, UNAM)

The physicochemical characteristics of wine can be modified by variations in temperature, time, and exposure to light and oxygen. These changes can alter its metabolomic fingerprint and affect quality/quantitative analyses. The aim of this work is to compare the information obtained through different chemometric methods used in a complementary way to study the variations of the 1H-NMR spectrum of four red wine samples preserved at different temperatures and time periods. Chemometric analysis reveal variations related to the wine sample, temperature, and time; as well as the interactions among these factors. The magnitude and statistical significance of the effects is accounted by ASCA; while PARAFAC modeling finds variations in time-related effects. The metabolites that present the most important variations were identified.

POSTER 160

Pyruvate vs glucose as metabolic imaging agents: Is one better than the other?

Presenter: Elton Montrazi (Weizmann)

All Authors: Elton Tadeu Montrazi (Weizmann Institute); Keren Sasson (Weizmann Institute); Lilach Agemy (Weizmann Institute); Avigdor Scherz (Weizmann Institute); Lucio Frydman (Weizmann Institute)

Metabolic MRI can highlight cancer via the glycolytic transformation of precursors like pyruvate and glucose into lactate. This study compares the MRI pictures emerging upon using deuterated pyruvate and deuterated glucose, as reporters of a pancreatic cancer murine model. Pyruvate injections produced a short-lived lactate signal throughout the body, fading rapidly and showing only a slight concentration bias at the tumors' edges. By contrast, glucose generated a strong, localized, long-lasting lactate signal within the tumors. Deuterated/non-deuterated glucose chasing experiments indicate a correlation between lactate generation and glucose availability at the tumor, evidencing a continuous and avid glucose consumption reflective of the Warburg effect. By contrast, the fate of pyruvate appears dominated by transport considerations. Implications of these findings are addressed.

POSTER 161

Pi-Network Diamagnetism Provides High Chemical-Shift, Endogenous CEST Contrast for Reporter Proteins

Presenter: David Korenchan (Athinoula A. Martinos Center for Biomedical Imaging)

All Authors: David Korenchan (Athinoula A. Martinos Center for Biomedical Imaging); Nicolas Scalzitti (Department of Chemical Engineering and Materials Science, Michigan State University); Michael T. McMahon (F.M. Kirby Research Center for Functional Brain Imaging, Kennedy Krieger Institute); Assaf A. Gilad (Department of Chemical Engineering and Materials Science, Michigan State University); Christian T. Farrar (Athinoula A. Martinos Center for Biomedical Imaging)

Reporter gene protein products containing exchangeable protons with large chemical shifts distinct from endogenous proton pools would provide high selectivity and specificity for treatment monitoring with CEST MRI. We explored several short peptides and proteins and discovered new CEST resonances downfield of 3.5 ppm amide signals, up to 9.8 ppm from water. The protons producing the discovered resonances all appear to experience electron deshielding caused by cation-pi interactions between aromatic amino acid sidechains. The new CEST peaks can be engineered into proteins using endogenous protein synthesis for use with monitoring viral gene therapy or oncolytic virotherapy in vivo. Future work will focus on designing a stable reporter gene product with high CEST contrast for MRI therapeutic monitoring.

POSTER 162

Evaluating the Immunotherapy Efficacy of Lung Cancer by 129Xe MRI

Presenter: Xin Zhou (Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences)

All Authors: Maosong Qiu (Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Shizhen Chen (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)

Cancer immunotherapy has great potential in the therapy of lung cancer but suffers from immunosuppressive tumor microenvironment (TME). Using efficient imaging protocols to monitor the immunotherapy effect of lung cancer for optimizing immunotherapy schedules in real time represents a promising avenue. With high sensitivity and without ionizing radiation, hyperpolarized 129Xe MRI is suitable for multiple detection of lung diseases such as COVID-19. Herein, we developed a reactive oxygen species-responsive Fe3O4 nanoprobe with the capability to induce ferroptosis for revising TME, enhancing the immunotherapy efficiency of lung metastases. Significantly, the 129Xe MRI displayed the complete shape of the lung, whereas the control group exhibited serious ventilation defects. Those results confirm that 129Xe MRI is a robust technology for monitoring immunotherapy effects.

POSTER 163

A Reversible 19F MRI Probe for Real-Time Monitoring the Glutathione Level in Vivo

Presenter: Xin Zhou (Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences)

All Authors: Yunhui Xiang (Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Lei Zhang (Innovation Academy for Precision Measurement Science and Technology, Chinese Academy of Sciences); Shizhen Chen (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)

It is vital to employ real-time in vivo imaging and quantify GSH dynamics to gain a deeper understanding of the pathophysiological processes. However, existing few probes are unsuitable for these purposes due to their irreversible signal-generating property or limited optical penetration depth. In this study, we've effectively addressed the issues by introducing a reversible 19F MRI probe. This innovation capitalizes on the distinctive, tunable reactivity of α,β-unsaturated organic molecules. The reactivity can transition from irreversible to reversible, depending on the electron-withdrawing capacity of the substituent at the α-position within the α,β-unsaturated region. The synthesized probe exhibits concentration-dependent, reversible and rapid 19F NMR/MRI changes, as well as appropriate Kd value.

POSTER 164

Parallel Metabolic Imaging Using MRI and Microfluidics for Personalised Medicine

Presenter: David Gomez-Cabeza (IBEC)

All Authors: David Gomez-Cabeza (IBEC); Marc Azagra (IBEC); Alba Herrero-Gomez (IBEC); Lluis Mangas-Florencio (IBEC); James Eills (IBEC); Alejandro Portela (Vitala); Gergo Matajsz (IBEC); Irene Marco-Rius (IBEC)

The high complexity of metabolic diseases drives researchers to seek holistic, yet personalised, strategies to treat them. Organs-on-a-chip (OoC) are promising candidates to develop complex patient-derived biological models to study such diseases and potential treatments in a parallel manner. Yet, to truthfully evaluate metabolism to infer cellular or organ state, we need fast, non-invasive and non-destructive methodologies. In this work, we combine microfluidics and hyperpolarised magnetic resonance spectroscopic imaging (HP-MRSI) to non-destructively study cell metabolism in real-time and establish a platform for parallel studies of disease treatments. Our results are the first to show such level of parallelisation with high spatial resolution for cellular studies using microfluidics and HP-MRSI, contributing to incorporating hyperpolarised NMR into the field of precision medicine.

POSTER 165

Systematic Insights into RF Heating from Pulsed Saturation in 129Xe HyperCEST Experiments: Consequences for Pulse Optimization and NMR Thermometry

Presenter: David Hernandez (Charite)

All Authors: David Hernandez Solarte (Charite); Ingolf Sack (Charite); Leif Schroeder (Deutsches Krebsforschungszentrum (DKFZ))

Hyperpolarized 129Xe receives increasing attention as an MR contrast agent. In the context of CEST applications, RF heating, and NMR thermometry aspects become relevant. Examining the effects of temperature changes using different pulse shapes and power levels, we studied two Xe hosts, cryptophane-A (CrA) and cucurbit-[6]-uril (CB6). Findings reveal that the magnet drift has an impact on temperature measurements, and pulsed saturation demonstrates

equivalent heating to cw saturation. Additionally, the solvent impacts the observed heating (saline solutions exhibiting higher temperatures than water). The study provides valuable insights into pulse shape comparisons and reveals differences in the chemical shift sensitivity of 129Xe hosts to temperature changes, thereby advancing the understanding of their applications.

POSTER 166

Real-time Adaptive MRI using Neural Networks

Presenter: Dan Xiao (University of Windsor)

All Authors: Tristhal Parasram (University of Windsor); Dan Xiao (University of Windsor)

MRI experiments are traditionally conducted using predefined pulse sequences, including k-space sampling/undersampling trajectories, which cannot be optimal for all scans. There is potential to adaptively schedule the next segment of a pulse sequence based on the partially acquired data to obtain the most informative data points throughout the experiment. In this proof-of-concept work, we propose using neural networks to predict adaptively which k-space points contain the most important information based on previously acquired partial k-space data. We demonstrated the feasibility of real-time, sample-specific adaptive MRI schemes optimized based on the signal. It has the potential to offer the most effective and efficient MRI scans. This technique can also be applied in NMR spectroscopy experiments.

POSTER 167

Bridging Health Disparities: Accessible MRI in Underserved African Regions

Presenter: Jingting Yao (National Institute of Environmental Health Sciences)

All Authors: Israa Hissein (National Institute of Environmental Health Sciences); Jingting Yao (Massachusetts General Hospital/Harvard Medical School); Foksouna Sakadi (National Reference Teaching Hospital); Ming Zhao (Massachusetts General Hospital and F.X. Massé Associates, Inc.); André J.W. van der Kouwe (Massachusetts General Hospital/Harvard Medical School); Jerome Ackerman (Massachusetts General Hospital/Havard Medical School)

Neurological disorders, including cerebral malaria and HIV/AIDS-associated complications, are leading causes of death in underserved African nations, hindered by a lack of medical equipment, particularly MRI facilities. We seek to address two key questions: the need for MRI facilities in underserved African regions and the potential of "accessible MRI" in reducing healthcare disparities related to medical imaging. We employed a multifaceted approach, involving literature review, interviews, and evaluations of benefits offered by accessible MRI and necessary enhancements for contemporary scanners. The marked disparities in MRI capabilities in Africa underscore the pressing need for investment in enhanced MRI infrastructure and customized imaging technologies tailored to resource-limited settings, all of which align with the vision set forth by the accessible MRI concept.

POSTER 168

Using the RASER Phenomenon to Enable 2D-MRI Without Radiofrequency Pulse Excitation

Presenter: Simon Fleischer (IMT (Karlsruhe Institute of Technology))

All Authors: Simon Fleischer (IMT (Karlsruhe Institute of Technology)); Jing Yang (IMT (Karlsruhe Institute of Technology)); Mazin Jouda (IMT (Karlsruhe Institute of Technology)); Stephan Appelt (Forschungszentrum Juelich GmbH); Jan G. Korvink (IMT (Karlsruhe Institute of Technology)); Sören Lehmkuhl (KIT)

RASER (Radiofrequency Amplification by Stimulated Emission of Radiation) can be used to generate magnetic resonance images without the need for radiofrequency excitation, using a comparably simple setup, as previously demonstrated. The resulting 2D images were plagued by artifacts stemming from nonlinear interactions between the modes of a freely evolving RASER.

Here, we present an approach combining RASER excitation with spatial encoding based on fast gradient echos. The interaction between individual RASER modes is much less pronounced, due to the strong magnetic field gradients involved, reducing the intensity of nonlinear artifacts. This opens up RASER-based imaging for new venues of application without having to accomodate for these nonlinear artifacts in processing.

POSTER 169

Functional neuroimaging of the tree shrew (Tupaia belangeri) brain

Presenter: Sonjong Hwang (Caltech)

All Authors: Sonjong Hwang (Caltech); Joseph Wekselblatt (UCLA); Kevin T. Hitchens (Univ. of Pittsburgh); Michael Arcaro (Univ. of Pennsylvania)

Neuroimaging bridges cross-species comparative studies from rodents to primates. However, its application to organisms closer to primates remains limited. Here, we present functional neuroimaging of the northern tree shrew (Tupaia belangeri), a close relative of the primate. We performed 7T (Bruker) MR imaging on six anesthetized adult tree shrews. We evaluated fluctuations of the BOLD fMRI signal. Our analyses identified several networks exhibiting functional coupling locally and across distant cortical regions. This includes mapping the within-network topographic organization of the somato-motor cortex. These findings are being integrated with an MRI anatomical atlas of the tree shrew brain that we have been developing. Our aim is to offer the scientific community with a comprehensive resource for functional and anatomical investigations.

POSTER 170

Variable Field Multinuclear MRI: From Optimization to In-Vivo Experiments at 0.3 - 3.0 T on 1H, 13C, and 15N

Presenter: Megan Pike (North Carolina State University)

All Authors: Megan Pike (North Carolina State University); Austin Browning (North Carolina State University); Patrick TomHon (Vizma Life Sciences); Keilian MacCulloch (North Carolina State University); Stephen McBride (North Carolina State University); Erica Curran (North Carolina State University); Adam Ortmeier (North Carolina State University); Mustapha Abdulmojeed (North Carolina State University); Thomas Theis (North Carolina State University)

Magnetic resonance imaging (MRI) is a ubiquitous tool for medical imaging. High field MRIs have high signal-to-noise ratio (SNR) which helps to produce high resolution images; however, they present numerous technical and financial limitations. Low field MRI can be used to image samples such as plant roots that are encapsulated by soil, that would cause significant susceptibility distortions at higher fields, but suffers from lower sensitivity. However low-field MRI in tandem with a hyperpolarization method can be used to mitigate the low sensitivity of low-field MRI by maintaining high levels of polarization. In our lab we assembled a multifield MRI, which utilizes 5 distinct fields, to unlock and explore the benefits of both low and high field MRI.

POSTER 171

1 mT MRI detected with a volume low-Tc SQUID gradiometer

Presenter: Dimitri Labat (Chipiron)

All Authors: Zineb Belkacemi (Chipiron); Yacine Belkhodja (Chipiron); Romain Couvreur (Chipiron); Bastien Dassonneville (Chipiron); Guillaume Daval-Frerot (Chipiron); Ricardo Ferreira (Chipiron); Marco Fiorito (Chipiron); Eva Grimaldi (Chipiron); Alexandre Jaoui (Chipiron); Ijee Mohanty (Chipiron); Isabelle Saniour (Chipiron); Dimitri Labat (Chipiron)

Chipiron develops an ultra-low field medical MRI device working at 1 mT. Reasons to go this low in field include the sharp increase in T1 contrast below 10 mT, and the versatility in use cases permitted by the light design of the copper electromagnet producing the B0 field.

To navigate the very low SNR at such low fields, we designed a novel volume gradiometric coil amplified by a low-Tc SQUID current sensor. We recently got the first phantom 3D GRE images on our first lab prototype. Mid-term goal is to get images in clinically-compatible acquisition times by the end of 2024, by improving the hardware of the detection system and leveraging acceleration strategies and EDITER-based active noise cancellation.

POSTER 172
Parallel Ultra-low Field Magnetic Resonance Imaging with a Multichannel Atomic Magnetometer
Presenter: Young Jin Kim (Los Alamos National Laboratory)
All Authors: Young Jin Kim (Los Alamos National Laboratory)
Magnetic resonance imaging (MRI) at ultra-low field (ULF) (<<1 T) is a promising non-invasive anatomical imaging tool. Previously, multichannel ULF MRI was realized only with multiple sensitive superconducting quantum interference device (SQUID) sensors. A serious drawback of SQUID sensors is the need for cryogenic infrastructure. We address this limitation by designing an ambient-temperature magnetic sensor composed of a 4-channel atomic magnetometer (AM) coupled to four flux transformers (FTs). In this talk, we will describe the approach of our ULF MRI device and present our progress on the development of the 4-channel RF AM-FT sensor.

POSTER 173
PETALUTE: Preclinical Applications of the Novel Rosette for FAST-Flexible Contrast MR(S)I
Presenter: Uzay Emir (Purdue University)
All Authors: uzay emir (Purdue University); Stephen Sawiak (University of Cambridge); Matthew Scarpelli (Purdue University); Xin Shen (UCSF); Alex Lipka (Purdue University); Joseph Rispoli (Purdue University); Gregory Tamer (Purdue University); Thejas Vishnu Ramesh (Purdue University); Jessica Leigh Veenstra (Purdue University); Deng-Yuan Chang (Purdue University)
Shen et al. 2021, 2022, and 2023 demonstrated the addition of novel rosette trajectory (PETALUTE) measurements in MRI and MRSI at ultra-high and high-field scanners (1-3). The dual-echo 3D rosette trajectories offer greater efficiency, allowing a center-out (1st echo) and center-in (2nd echo) sampling pattern that provides more outer and center k-space per petal samples than radial spokes. In addition, the rosette k-space trajectory samples center k-space in a more incoherent pattern. There, it offers the potential for further acceleration using higher under-sampling factors and the compressed sensing technique for reconstruction. This study demonstrated PETALUTE MR(S)I sequences for preclinical ultra-high field (7T, 9.4T) scanners.

POSTER 175
MRS in Neurosciences – Recent advances in In-vivo spectroscopy methods and applications at the Stanford Center for Cognitive and Neurobiological Imaging (CNI)
Presenter: Laima Baltusis (Stanford University)
All Authors: Laima Baltusis (Stanford University); Donna Murray (Stanford University); Ralph Hurd (Stanford); Meng Gu (Stanford University); Hua Wu (Stanford University); Sache Coury (University of California, Los Angeles); Troy Murray (Loyola University); Daniel Spielman (Stanford University)
The spectroscopy group at CNI develops and supports new MRS data acquisition and analysis capabilities. We have been optimizing and evaluating focal 2D MRSI in both cortical and subcortical regions of the brain as an alternative to short TE single voxel data acquisition. For optimal quantification of metabolites in both focal 2D MRSI and single voxel data analyses have included: evaluation of improved coil combination methods; improvements in data analysis using LCModel by (1) mitigation of baseline and macromolecular contributions and (2) improvement of the accuracy of the LCModel basis set using a largely experimental 23 metabolite basis set.

POSTER 176
Developing the SI-traceable NMR measurement of T1rho on musculoskeletal MRI phantom material
Presenter: Cassandra Stoffer (National Institute of Standards and Technology)
All Authors: Cassandra Stoffer (National Institute of Standards and Technology); Jeehun Kim (Program of Advanced Musculoskeletal Imaging (PAMI), Cleveland Clinic); Karl Stupic (National Institute of Standards and Technology); Stephen E. Russek (National Institute of Standards and Technology); Kathryn E. Keenan (National Institute of Standards and Technology); Jeff Kammerman (Calimetrix); David Rutkowski (Calimetrix); Jean H. Brittain (Calimetrix); Xiaojuan Li (Program of Advanced Musculoskeletal Imaging (PAMI), Cleveland Clinic)
Recent development of a musculoskeletal (MSK) MRI phantom for T1, T2, and T1rho measurements has sparked particular interest in SI-traceable relaxometry measurements. NIST has a well-established protocol using NMR techniques to provide traceable T1 and T2 measurements, and now NIST aims to expand these capabilities to T1rho, which is a particular parameter of interest for MSK studies. In this work, we develop a T1rho measurement protocol for MSK phantom materials and observe T1rho dispersion. Significant proton resonance frequency shifts were observed between measurements at different temperatures, and this led to measurement inaccuracies. We will revise the automated data acquisition software to adjust center frequency, which should minimize measurement error and further the development of traceable T1rho measurements.

POSTER 177
Implementation of 3D Dual Echo Rosette (PETALUTE) K-space Trajectory for Preclinical Bone MRI: Comparison to 3D Radial Ultrashort- and Zero Echo Time Approaches
Presenter: Farhan Sadik (Weldon School of Biomedical Engineering, Purdue University)
All Authors: Farhan Sadik (Weldon School of Biomedical Engineering, Purdue University); Mohseu Subah (Weldon School of Biomedical Engineering, Purdue University); Ali Ozen (Department of Radiology, University of Freiburg); Stephen Sawiak (University of Cambridge); Mark Chiew (University of Oxford); uzay emir (Purdue University); Rachel Surowiec (Weldon School of Biomedical Engineering, Purdue University)
The inherently ultrashort T2* component of cortical bone poses challenges for conventional MRI techniques, necessitating the development of ultrashort echo time MRI (UTE). Common UTE techniques utilize center-out sampling patterns like radial, spiral, and cones, enabling near-immediate gradient readout following the RF pulse. However, these trajectories fail to cover the inner and outer k-space efficiently, which may lead to poor quantification of biomarkers. In this study, we implemented a 3D center out dual echo rosette (PETALUTE) trajectory for preclinical bone imaging. PETALUTE more densely samples outer k-space while ensuring a nominal TE to capture the ultrafast-decaying T2* signal, making it an ideal sequence for bone MRI. Further, PETALUTE retains incoherence, making it suitable for accelerated methods such as compressed sensing.

POSTER 178
Spherical Field Mapping Method for Unshielded, Single Sided, Low-Field MRI system
Presenter: Dnyanada Mahendra Kadiyal (Promaxo Inc)
All Authors: Dnyanada Mahendra Kadiyal (Promaxo Inc); Vikram Venkidu (Promaxo Inc); Scott B. King (Promaxo Inc); Ram Narayanan (Promaxo Inc)
In conventional MRI, image reconstruction relies on linear encoding gradients, facilitating fast Fourier transforms. However, for single-sided MRI system such as Promaxo's incorporates a static gradient in its main magnetic field, requiring B0 field distribution knowledge for reconstruction. To address this, we propose a methodology that employs field measurement on a surface of a sphere. With desired accuracy achieved, this could provide as a fast, portable, optimal method of field measurement for any MRI systems with similar challenges.

POSTER 179
MR-based body composition analysis, resting state fMRI and autonomic function evaluation for phenotyping
Presenter: Chetna Banga (All India Institute of Medical Sciences)
All Authors: Chetna Banga (All India Institute of Medical Sciences)
Growing interest towards precision medicine, phenotyping has become as important as genotyping. This study has evaluated and correlated the MR-based resting–state functional connectivity and body composition with autonomic functions-based physiological marker. Resting state fMRI and mDIXON-Quant sequence for fat distribution in abdomen and thigh at 3T MR scanner and in autonomic

functions sympathetic (head up tilt, cold pressor and hand grip) and parasympathetic (deep breathing and head up tilt) studies were performed in healthy volunteers (n=40). Volunteers were categorized into two on the basis of their BMI values - < 25 kg/m2 (Group I) and > 25 kg/m2 (Group II). This study on phenotyping markers assumes to be beneficial to identify risk groups for specific diseases, customizing diet and lifestyle.

Organic Inorganic and Hybrid Materials (Posters 180-200)

POSTER 180

Breakdown of Spin Inversion in Force-Gradient Detection of Magnetic Resonance

Presenter: Michael Boucher (US Naval Research Laboratory)

All Authors: Michael Boucher (US Naval Research Laboratory)

A primary goal of Magnetic Resonance Force Microscopy (MRFM) research is to enable the imaging of spin ensembles as small as a few hundred nuclei or a single electron. Figure 1 depicts an experimental setup for MRFM. The sample spins, either nuclei or electron radicals, are deposited on a waveguide radiofrequency source. A cantilever, tipped with a micron- or nanoscale magnet is brought normal to the sample surface. The detection scheme, Cantilever-Enabled Readout of Magnetization Inversion Transients (CERMIT), allows for the detection of the z-magnetization of both fast- and slow-relaxing spins and with a reduced irradiation duty cycle when compared to other methods

POSTER 181

Phase Transitions in Hydrated Biominerals: Variable Temperature ssNMR and Combined Experimental-Computational Approaches

Presenter: Danielle Laurencin (CNRS)

All Authors: Ieva Goldberga (Sorbonne Université); Adam Nelson (Sorbonne Université); Christel Gervais (Sorbonne Université); Vincent Sarou Kanian (CNRS Orléans); Ivan Hung (NHMFL - MagLab); Zhehong Gan (NHMFL - MagLab); Nicolas Birlirakis (ENS-PSL); David Gajan (ENS-Lyon); Thomas-Xavier Métro (CNRS); César Leroy (CNRS); Dorothée Berthomieu (CNRS); Christian Bonhomme (Sorbonne Université); Danielle Laurencin (CNRS)

Calcified tissues like bone and kidney stones contain complex mineral phases. Notably, the structure of hydrated biominerals has been found to be challenging to investigate.

Here, we will illustrate our recent studies on two of these biominerals :

- Calcium oxalate monohydrate (CaC2O4.H2O), the main mineral found in kidney stones;

- Octacalcium phosphate (Ca8(HPO4)2(PO4)4.5H2O), a phase considered as one of the precursors of bone mineral.

In both cases, it will be shown that the combination of multinuclear ssNMR analyses at different temperatures (including temperatures as low as 100 K, and on challenging nuclei like 17O and 43Ca), and of computational modeling (molecular dynamics simulations and GIPAW-DFT calculations of NMR parameters) is key to elucidate the structure of the materials.

POSTER 182

Investigating Wastewater Treatment Plant Processes: NMR Spectroscopy as a Complementary Technique for Characterizing Complex Environmental Samples

Presenter: Kiera Ronda (University of Toronto Scarborough)

All Authors: Kiera Ronda (University of Toronto Scarborough); Katelyn Downey (University of Toronto); Daniel Lysak (University of Toronto); Peter Costa (University of Toronto); William Wolff (University of Toronto); Katrina Steiner (University of Toronto); Jacob Pellizzari (University of Toronto); Myrna J. Simpson (University of Toronto); Karl J. Jobst (Memorial University of Newfoundland); Sonya Kleywegt (Ministry of the Environment, Conservation and Parks); Andre Simpson (University of Toronto)

Water scarcity is becoming an increasingly serious issue, making the recycling of water resources through wastewater treatment plants (WWTPs) imperative. However, current WWTPs are not capable of removing many contaminants of concern. Understanding the limitations of current WWTPs is an important step toward improving their efficiency, but this requires an in-depth understanding of the compositions of both raw influents and treated effluents. To address this, and investigate the changes that occur with treatment, NMR spectroscopy and gas chromatography-mass spectrometry are used as complementary techniques. Preliminary results suggest that long-chain acids decrease in concentration, whereas urea and fluorinated substances increase following treatment. This work highlights the complementary nature of NMR spectroscopy and GC-MS for understanding the limitations of current WWTPs.

POSTER 183

Chemical Upcycling of Polymers: Insights from NMR Methodology

Presenter: Shira Haber (Lawrence Berkeley National Laboratory)

All Authors: Shira Haber (Lawrence Berkeley National Laboratory); Julia Im (UC Berkeley); Nicodemo R. Ciccia (University of California, Berkeley); Mutian Hua (Lawrence Berkeley National Laboratory); Sophia Fricke (University of California, Berkeley); John F. Hartwig (University of California, Berkeley); Brett A. Helms (Lawrence Berkeley National Laboratory); Jeffrey A. Reimer (University of California, Berkeley)

One novel solution to the world's growing plastic waste problem is to create a circular polymer-waste system, by chemically recycling plastics to reusable monomers, thus forming an efficient closed loop. Another direction, is to functionalize polyethylenes, modifying the polymer's bulk and surface properties. An understanding of mechanistic underpinnings is critical to guide rational improvements in reaction efficiency and future materials design.

Here, NMR spectroscopy, relaxometry, and diffusometry, are applied to provide molecular insight into bond activation, reaction selectivity and reactivity. In addition, real-time transformations of polymer decomposition can be monitored, shedding light on the heterogeneity of polymer degradation processes. Quantified phase compositions of functionalized polymers, coupled with molecular chain mobility, enable correlation of new surface properties with polymer bulk morphology.

POSTER 184

Progressive Saturation of the Proton Reservoir Under Spinning (PROSPRUS)

Presenter: Lucio Frydman (Weizmann Institute)

All Authors: Tamar Wolf (Weizmann Institute of Science); Yuval Goobes (Weizmann Institute of Science); Lucio Frydman (Weizmann Institute)

We have recently introduced PROSPR, an indirect-detection scheme for the sensitivity enhancement of dilute, unreceptive species in static solids, that operates by depolarizing spin order from an abundant 1H reservoir. While dipolar order is averaged away by MAS, this study describes an efficient MAS experiment that now relies on residual dipolar order. The resulting PROgressive Saturation of the Proton Reservoir Under Spinning (PROSPRUS) experiment shows significant sensitivity enhancement in ultrawideline spin-½ and spin-1 nuclei, as demonstrated by cisplatin's 195Pt and 14N. Using low RF amplitudes of 1-2 kHz, good lineshapes can be achieved with high SNR per unit time compared to other broadband methods under MAS. PROSPRUS experiments on 14N in trigonelline.HCl and 119Sn in tin diacetate, illustrate remaining challenges.

POSTER 185

Experimental and Theoretical Solid-State NMR Investigations of the Platinum Group Elements

Presenter: Robert Schurko (FSU and NHMFL)

All Authors: Sean Holmes (Florida State University); Adam B. Philips (University at Buffalo); James Kimball (Florida State University); Sara Termos (Florida State University); Jasmin Schoenzart (Colorado School of Mines); Adam Altenhof (Florida State University); Yijue Xu

(Florida State University); Christopher A. O'Keefe (University of Windsor); Jochen Autschbach (University at Buffalo); Robert Schurko (FSU and NHMFL)

The platinum group elements (PGEs), which include Ru, Rh, Pd, Os, Ir, and Pt, face significant challenges in their usage, including issues with scarcity, high costs, recycling, and environmental concerns. Replacement metals for PGEs must be found - an understanding of structure and bonding, through a combination of NMR and first principles calculations, are key for these efforts. NMR explorations of three platinum group elements (PGEs) will be described, including novel 99Ru, 103Rh, and 195Pt solid-state NMR experiments, relativistic quantum chemical computations of chemical shift and electric field gradient tensors, new NBO/NLMO analyses of MO contributions to these tensors, and applications to different classes of inorganic and organometallic compounds and materials.

POSTER 186

Unravelling the 1H NMR relaxation mechanism in polymeric viscosity-standards and bitumen using measurements, MD simulations, and models

Presenter: Yunke Liu (Rice University)
All Authors: Yunke Liu (Rice University); Arjun Valiya Parambathu (Department of Chemical and Biomolecular Engineering, University of Delaware); Thiago J. Pinheiro dos Santos (Department of Chemical and Biomolecular Engineering, Rice University); George J. Hirasaki (Department of Chemical and Biomolecular Engineering, Rice University); Dilip Asthagiri (Oak Ridge National Laboratory); Walter G. Chapman (Department of Chemical and Biomolecular Engineering, Rice University); Philip Singer (Department of Chemical and Biomolecular Engineering, Rice University)

We investigate 1H NMR relaxation measurements of a polymeric viscosity-standard over an extended range of Larmor frequencies (f0 = 2 − 400 MHz) and viscosities (η = 10,650 – 333,400 cP) using T1 and T2 in static fields. We account for the anomalous behavior of the measured (log-mean) relaxation times T1LM∝f0 and T2LM∝(η/T)^(-1/2) with a phenomenological model of 1H−1H dipole−dipole relaxation. We also find that molecular dynamics simulations predicting the T1∝f0 dispersion at high viscosity in agreement with measurements. We investigate the effect of 1H NMR cross relaxation σ1 (a.k.a., spin diffusion), which manifests itself as a narrowing in the T1 distribution, using a proposed metric |σ1 |/R1 for the relative strength in cross relaxation.

POSTER 187

The Calcium Sites of Hydroxyapatite with Different Morphologies and Crystal Phases Investigated by 43Ca MAS Solid-State NMR

Presenter: Yuan Li (San Diego State University)
All Authors: Yuan Li (San Diego State University); Gregory Holland (San Diego State University)

Hydroxyapatite (HAP) nanoparticles with different morphologies and crystallographic orientations including nanowires (aHAP) with the (100) lattice plane preferentially developed, nanosheets (cHAP) with the (001) facet highly expressed and nanorods (rHAP), a polycrystalline HAP that present hybrid lattices were synthesized with 43Ca-enriched material. The calcium sites of these HAP are investigated with 1D 43Ca MAS, 1H {43Ca} TRAPDOR and 2D 3QMAS. Two calcium sites from the bulk crystal and resonances potentially associated to the surface groups are observed. TRAPDOR dephasing observed for various proton species that are spatially adjacent to the calcium sites.

POSTER 188

Towards Efficient Direct Air Capture: NMR Spectroscopy Analysis of CO2 and H2O Adsorption on amine-functionalized sorbents

Presenter: Ah-Young Song (University of California, Berkeley/Lawrence Berkeley National Laboratory)
All Authors: Ah-Young Song (University of California, Berkeley/Lawrence Berkeley National Laboratory); John Young (Heriot-Watt University); Jieyu Wang (University of California, Berkeley); Katia Piscina (Heriot-Watt University); Raynald Giovine (University of California, Berkeley); Sophia Fricke (University of California, Berkeley); Susana Garcia (Heriot-Watt University); Mijndert van der Spek (Heriot-Watt University); Jeffrey A. Reimer (University of California, Berkeley/Lawrence Berkeley National Laboratory)

The sorbents for direct air capture (DAC) have been extensively studied to achieve net-zero carbon dioxide emissions. A key factor to address the challenges of the sorbent is to elucidate CO2 adsorption behavior in CO2-dilute gas with the presence of water. This study demonstrates the adsorption characteristics via solid-state NMR spectroscopy when the amine-functionalized sorbent undergoes co-adsorption of CO2 and H2O. The co-adsorption apparatus provides a dilute CO2 concentration in the gas mixture to simulate the ambient air. The 13C and 1H NMR study illustrates the interaction with CO2 and the influence of H2O on CO2 adsorption in CO2-dilute gas environment. Hence, the findings contribute to its adsorption performance such as capacity and kinetic.

POSTER 189

Observing aqueous ion behaviour within microporous carbon electrode materials using NMR spectroscopy

Presenter: Ryan Bragg (Lancaster University)
All Authors: Ryan Bragg (Lancaster University); Kieran Griffiths (Lancaster University); John Griffin (Lancaster University)

The distribution of ions and the mechanisms controlling their exchange directly impact the performance of adsorption-based applications, such as water desalination and electrical double-layer capacitors. Currently, a large degree of ambiguity surrounds ionic behaviour within microporous environments (≤ 2 nm). Here, a quantitative NMR approach to investigate aqueous electrolytes within microporous carbon materials is detailed. Data on the effects of carbon structure and ion-specific properties on the ion distribution within uncharged microporous carbon networks is presented for aqueous electrolytes. Quantitative NMR measurements reveal complex relationships between the solvation properties of the ionic species, the measured changes in the electrolyte pH due and the observed preference towards charge-balancing mechanisms measured using in situ NMR.

POSTER 190

Room-temperature quantum sensing with photoexcited triplet electrons in organic crystals

Presenter: Harpreet Singh (UC Berkeley)
All Authors: Harpreet Singh (UC Berkeley); Noella D'Souza (UC Berkeley); Keyuan Zhong (UC Berkeley); Emanuel Druga (UC Berkeley); Julianne Oshiro (UC Berkeley); Brian Blankenship (UC Berkeley); Jeffrey A. Reimer (UC Berkeley); Jonathan D. Breeze (Department of Physics & Astronomy, University College London, Gower Street, London, WC1E 6BT, UK); Ashok Ajoy (UC Berkeley)

Quantum sensors have made significant strides in high-sensitivity magnetic field detection. Our study introduces quantum sensors utilizing polarized spin-triplet electrons within photoexcited organic chromophores, notably pentacene-doped para-terphenyl (=0.1%). At room temperature, we unveil crucial quantum sensing features: electronic optical polarization and state-dependent fluorescence contrast. Through leveraging differential pumping and relaxation rates between triplet and ground states, we achieve a high optically detected magnetic resonance (ODMR) contrast of the triplet states. Additionally, long coherence times are measured under the CPMG sequence, with T2DD =18.4us, limited solely by triplet lifetimes. Our material presents several advantages for quantum sensing, including low-cost growth of large (cm-scale) crystals, absence of paramagnetic impurities, and diamagnetism of electronic states when not optically illuminated.

POSTER 191

Spatially Resolved NMR Methods for Determination of Solute Partitioning

Presenter: Leo Gordon (University of California - Santa Barbara)
All Authors: Leo Gordon (University of California - Santa Barbara); Rahul Sujanani (University of California - Santa Barbara); Rachel A.

Segalman (University of California - Santa Barbara); Raphaële J. Clément (University of California - Santa Barbara)

Octanol-water partitioning behavior is a cornerstone metric for understanding the relative hydrophobicity of solutes in solvent mixtures. The octanol-water partition coefficient (Kow) is important in a multitude of areas, including pharmaceuticals and the field of separation science. Herein, we describe a simple NMR approach to determine Kow of solutes in biphasic octanol-water mixtures that can be performed rapidly (minutes): we apply small-bandwidth, spatially-selective pulses to excite slices either side of the octanol-water phase boundary to determine the solute ratio in each phase. With this method we were able to faithfully reproduce KOW values for a range of solutes, which can be measured quickly and easily. Our findings have wider implications for materials chemistry and chemical engineering.

POSTER 192

High-Resolution Heteronuclear Correlations Between Spin-1/2 and Quadrupolar Nuclei Using T-HMQC and cos-lp MQMAS

Presenter: Yusuke Nishiyama (JEOL)
All Authors: Manoj Kumar Pandey (Indian Institute of Technology (IIT) Ropar); Yusuke Nishiyama (JEOL)

Indirect observation of an X nucleus via 1H detection yields X/1H heteronuclear correlation spectra and is widely used to enhance sensitivity under fast MAS conditions. While CP based experiments are widely used in correlation between spin-1/2 nuclei, HMQC based experiments are generally more efficient for correlations between spin-1/2 and half-integer quadrupolar nuclei. However, the X dimension suffers from the second-order quadrupolar broadening resulting in broad resonances. In this study, we demonstrate the combination of cos-lp pulse-based MQMAS and HMQC experiments to achieve high resolution X/1H correlation spectra for half-integer quadrupolar nuclei.

POSTER 193

NMR as a Tool for Optimizing Composite Electrolytes

Presenter: Sergey Krachkovskiy (Hydro Quebec)
All Authors: Sergey Krachkovskiy (Hydro Quebec); Martin Dontigny (Hydro Quebec); Abdelbast Guerfi (Hydro Quebec)

Composite electrolytes, which are mixtures of organic and inorganic compounds, exhibit improved properties with respect to traditional systems. Since NMR is very sensitive to multiple parameters such as chemical structure, local dynamics, translational motion, it can be used as a versatile tool to provide critical information for the development and optimization of these materials. Herein, we demonstrate the application of NMR to the investigation of a family of highly conductive composite electrolytes based on an amorphous polymer matrix and micrometer-sized Li(1+x)AlxTi(2–x)(PO4)3 (LATP) particles.

POSTER 194

2D exchange 15N NMR analyses of adsorbed reaction intermediates in zeolite catalysts

Presenter: Alyssa McNarney (University of California, Santa Barbara)
All Authors: Michael Schmithorst (University of California, Santa Barbara); Alyssa McNarney (University of California, Santa Barbara); Subramanian Prasad (BASF Corporation); Vivek Vattipalli (BASF Environmental Catalyst and Metal Solutions); Ahmad Moini (BASF Environmental Catalyst and Metal Solutions); Bradley Chmelka (University of California, Santa Barbara)

Zeolites are crystalline nanoporous aluminosilicates used as catalysts in a variety of applications, including copper-exchanged zeolites for automotive emissions abatement. Solid-state NMR techniques are well-suited to determine the dynamics of adsorbed or intermediate species, which are crucial to understand reaction properties of the catalyst. However, paramagnetic Cu(II) species presents significant experimental challenges. We demonstrate that 2D exchange spectroscopy (EXSY) 15N NMR can be used to quantify the reversible site-exchange of adsorbed 15NH3 molecules between proximate Brønsted (NH4+) and [Cu(NH3)2]+ adsorption sites, enabling the extraction of exchange rate coefficients. These analyses are combined with complementary continuous-wave EPR measurements of the paramagnetic Cu2+ species to establish the compositions and distributions of Cu2+ to establish atomic-scale origins of reaction kinetics of zeolite catalysts.

POSTER 195

Multi-modality magnetic resonance spectroscopic study of fentanyl salts

Presenter: Harris Mason (Los Alamos National Laboratory)
All Authors: Harris Mason (Los Alamos National Laboratory); Michelle A. Espy (Los Alamos National Laboratory); Adam Altenhof (Los Alamos National Laboratory); Ruilian Wu (Los Alamos National Laboratory); Marc A. Alvarez (Los Alamos National Laboratory); Robert F. Williams (Los Alamos National Laboratory); Michael Malone (Los Alamos National Laboratory)

Fentanyl is a powerful and addictive synthetic opioid which has driven a public health crisis in the US for over a decade. Current methods of detection such as vibrational spectroscopy and wet chemical analysis require direct access to the sample. Our task is to investigate nuclear quadrupole resonance as a method for stand-off detection of synthetic opioids. To this end, we have utilized both solid-state NMR spectroscopy and fast field cycling (FFC) relaxometry to determine relevant search parameters for NQR detection.

POSTER 196

Ion Transport and Solvation Structure in Multivalent Electrolytes with Electrophoretic NMR

Presenter: Karim Karouta (UC Berkeley / LBNL)
All Authors: Karim Karouta (UC Berkeley / LBNL); David Halat (UC Berkeley & LBNL); Helen Bergstrom (UC Berkeley / LBNL); Jeffrey Reimer (UC Berkeley / LBNL)

The primary limitation for battery charging rates is the concentration gradient in the electrolyte. Poor cation transport slows technological progress in lithium-ion batteries as well as in sustainable alternatives such as sodium-, zinc-, and aluminum-ion batteries. While others have employed electrophoretic NMR in lithium-ion electrolytes, yielding electrophoretic mobility values though coherent phase-shifts in the presence of a PFG, to determine 7Li, 19F, and 1H velocities, we access velocities for PFG-inaccessible nuclei such as zinc and aluminum. Combining eNMR-determined solvent and anion velocities with conductivity measurements, we calculate cation mobility and transference across a range of concentrations of zinc triflate in methanol. We observe exceptionally high zinc mobility, showing promise for safe and sustainable zinc batteries.

POSTER 197

Elucidating the Mechanism of SEI Formation in High Concentrated Hybrid Aqueous Zinc Metal Batteries

Presenter: Alexis Scida (Oregon State University)
All Authors: Alexis Scida (Oregon State University); David Ji (Oregon State University); Tom Osborn Popp (Rutgers University)

Aqueous zinc metal batteries offer an affordable, energy dense solution for large-scale grid storage, but to date, have suffered from parasitic side reactions, namely hydrogen evolution reaction (HER). Recently, a unique electrolyte composition was found to form a dual inorganic/organic solid-electrolyte interphase (SEI), resulting in unprecedented Coulombic efficiency. To understand the formation of this SEI, we used NMR, which revealed an acid-catalyzed decomposition of dimethyl carbonate to methanol and carbon dioxide in high concentrated zinc water-in-salt electrolyte. The reaction products were then proven to be key participants in the formation of the SEI. Furthermore, NMR was used to track the temperature dependence of the reaction, which combined with electrochemical techniques, uncovered the optimal amount of decomposition products for improved efficiency.

POSTER 198

Revealing CO2 speciation at electrode-electrolyte interfaces with NMR spectroscopy

Presenter: Zeke Coady (University of Cambridge)
All Authors: Zeke Coady (University of Cambridge); Zhen Xu (University of Cambridge); Benjamin Rhodes (University of

Cambridge); Grace Mapstone (University of Cambridge); Alexander Forse (University of Cambridge)

Interactions of CO2 at aqueous solution-surface interfaces in carbon materials are relevant to electrochemical carbon capture and utilisation processes. In this work we use solid-state NMR spectroscopy to study CO2 speciation and distribution and unpick the complex equilibria involved. CO2 solubility in 1 M Na2SO4(aq) increases 50-fold in the presence of activated carbon. In-pore and ex-pore environments and CO2, bicarbonate and carbonate can be differentiated in our spectra and populations measured quantitatively. The effects of pore structure on carbon capture processes can be rationalised. Overall we demonstrate NMR spectroscopy's unique strengths for understanding CO2-water systems in porous activated carbons.

POSTER 199

Molecular Dynamics assisted NMR Study of dendritic Core size induced chemical and morphological heterogeneity in self-assembled star-shaped polypeptides hydrogel

Presenter: Bing Wu (Radboud University Nijmegen)
All Authors: Bing Wu (Radboud University Nijmegen)

Polypeptide, the building blocks of living organisms, is an important class of biopolymers. Like natural proteins, these polymers possess the ability to self-assemble into complex yet highly ordered structures as a result of the precisely controlled sequences and compositions of their constituent amino acid monomers. Hydrogels derived from synthetic polypeptides have attracted huge attention from the scientific community due to their nontoxicity, biodegradability, and low immunogenicity.

Here we report the first systematic study about the impact of the dendrimer core size on the physico-chemical properties of star-like hydrogels. Systematic NMR relaxometry and diffusometry analyses were used to determine the polymer chain dynamics in these hydrogels. Finally, a series of MD simulations were also performed to rationalize the observed change.

POSTER 200

Magnetic Resonance Imaging on Compression of Elastomers In Situ

Presenter: Maxwell Marple (Lawrence Livermore National Laboratory)
All Authors: Maxwell Marple (Lawrence Livermore National Laboratory); Derrick Kaseman (Lawrence Livermore National Laboratory)

Silicone elastomers are soft composite polymers with highly tunable mechanical properties based on changing their composition and filler materials. Additive manufacturing (AM) of silicone elastomers can further enhance the mechanical properties by printing the elastomer in a complex 3D architecture that can have specific properties under loading. To better understand the connection between the polymer network dynamics and bulk mechanical properties, we use magnetic resonance imaging to bridge the gap between microscopic NMR observables, like proton density and T2, and macroscopic features from AM structures. Collecting T2 maps of AM silicone elastomers under compression enables simultaneous mapping of the strain behavior and effects on the polymer mobility, revealing an influence of thixotropic properties.

Small Molecules Natural Products (Posters 201-219)

POSTER 201

Development of Novel Polymer Electrolytes for Macroscale 3D all Solid-state Batteries: Transport Studies

Presenter: Nishani Jayakody (Division of Chemistry, U.S. Naval Research Laboratory)
All Authors: Nishani Jayakody (Division of Chemistry, U.S. Naval Research Laboratory); Hunter O. Ford (Division of Chemistry, U.S. Naval Research Laboratory); Brian Chaloux (Division of Chemistry, U.S. Naval Research Laboratory); Youngchan Kim (Material Science and Technology Division, U.S. Naval Research Laboratory); Debra R. Rolison (Division of Chemistry, U.S. Naval Research Laboratory); Jeffrey W. Long (Division of Chemistry, U.S. Naval Research Laboratory); Megan B. Sassin (Division of Chemistry, U.S. Naval Research Laboratory); Joel Milller (Division of Chemistry, U.S. Naval Research Laboratory); Christopher Klug (US Naval Research Laboratory)

In order to combine the opposing metrics of high-power density and energy density, battery cell design must transition away from the conventional two-dimensional (2D) layer design towards three-dimensional (3D) all solid-state configuration. One of the challenges is the engineering of a thin solid-state electrolyte that can conform to the 3D electrode architecture. Initiated chemical vapor deposition (iCVD) was use to generate a copolymer system, comprising (dimethylaminomethylstyrene) DMAMS and divinylbenzene (DVB), which are converted to single anion-conducting SSEs post polymerization . Dynamic properties were determined by measuring the 1H self-diffusion coefficient of these hydroxide conducting solid state electrolytes (OH-SSEs) using the fringe field diffusion NMR technique. Observed diffusion results showed the presence of two distinct molecular diffusion mechanism for H2O, OH-.

POSTER 202

Effects of freezing/thawing on T2 relaxation measurements in chicken breast meat

Presenter: Hong Zhuang (US National Poultry Research Center)
All Authors: Hong Zhuang (US National Poultry Research Center); Janghan Choi (US National Poultry Research Center); Brian Bowker (US National Poultry Research Center); Byungwhi Kong (US National Poultry Research Center); Woo Kyun Kim (University of Georgia)

Effects of freezing/thawing on T2 relaxation were investigated in chicken fillets (Pectoralis major). Fillets were frozen at -20, -40, or -200°C and thawed at either 4 or 20°C. T2 relaxation was measured using Bruker LF-NMR 90II pre-freezing and post-thawing. Three water components, referred as T2b, T21, and T22, were identified with average relaxation times of 4.5, 43.9 and 125.2 ms pre-freezing and 4.7, 43.1, and 122.8 ms post-thawing. Freezing/thawing significantly affected T2 component parameters T (peak time), P (relative peak area), and A (normalized peak area). Freezing/thawing tended to increase T2b, P22, and A22 regardless of treatment temperatures. Thawing at 4°C reduced T2b, T21, T22, P21, and A21 but increased P22 and A22 compared with 20°C regardless of freezing temperature.

POSTER 203

Designed Sequences for Higher Sensitivity, Higher Spectral Dispersion and Precise Determination of Homo- and Hetero-nuclear Interaction strengths, and eradication of unwanted evolution

Presenter: Suryaprakash Nagarajarao (Indian Institute of Science)
All Authors: Suryaprakash Nagarajarao (Indian Institute of Science)

Novel techniques have been designed for the simplification of NMR spectral complexity, enhancing the sensitivity of detection, unravelling of the spectral overlap and the accurate determination of homo- and hetero- nuclear interaction strengths. The clean sequences also result in the prevention of the evolution of unwanted couplings, eradication of axial peaks, in addition to enhancing the spectral resolution.

POSTER 204

Structure elucidation of novel cyanobactins isolated from a cultured cyanobacterial strain of Microcystis aeruginosa

Presenter: Caitlyn Agee (University of North Carolina Wilmington)
All Authors: Caitlyn Agee (University of North Carolina Wilmington); Michael Recchia (Simon Fraser University); Robert (Thomas) Williamson (UNC Wilmington); Wendy Strangman (UNC Wilmington)

Cyanobacteria are an auspicious source of new natural products with potent and therapeutically useful biological activities. Among the bioactive compounds produced, cyanobactins have attracted a considerable amount of interest due to their potential as candidates in drug discovery. Cyanobactins are among the growing class of known ribosomally derived and post-translationally modified peptides (RiPPs). Many members of this class, including the cyanobactins, exhibit elements of mixed biosynthetic origin such as incorporation of terpenoid pathways derived isoprene units. Through our investigation

of Microcystis aeruginosa, two unreported prenylated cyanobactin structures have been discovered along with their des-prenylated analogs. In this work, complete structure elucidation of these new cyclic 11-membered peptides using 1D and 2D Nuclear Magnetic Resonance (NMR) experiments will be described.

POSTER 205

Solvent suppression in pure shift NMR

Presenter: Emma Gates (University of Manchester)
All Authors: Emma Gates (University of Manchester); Jonathan P. Bradley (Johnson Matthey); Daniel B. G. Berry (Johnson Matthey); Mathias Nilsson (University of Manchester); Gareth A. Morris (University of Manchester); Ralph W. Adams (University of Manchester); Laura Castañar (Complutense University of Madrid)

Solvent signal suppression is routinely used in many solution-state NMR experiments when protiated solvents are used. Presaturation methods, while simple and efficient, require lengthy continuous-wave irradiation to saturate the solvent signal which causes saturation of signals of protons in exchange with the solvent. Alternatively, WATERGATE solvent suppression limits the attenuation of exchangeable signals. Here, we show the benefits of integrating WATERGATE into pure shift NMR experiments, providing ultrahigh resolution spectra (by collapsing signal multiplicity) with negligible residual solvent signal. The new method is demonstrated in the analysis of cyanocobalamin and an eye-drop formulation of atropine, where the conventional 1H spectra are complicated by signal overlap, high dynamic range, and signal multiplicity.

POSTER 206

The Natural Products Magnetic Resonance Database (NP-MRD.org)

Presenter: Bharat Goel (Division of Biochemistry, Bond Life Sciences Center, MU Metabolomics Center, Interdisciplinary Plant Group, University of Missouri-Columbia)
All Authors: Bharat Goel (Division of Biochemistry, Bond Life Sciences Center, MU Metabolomics Center, Interdisciplinary Plant Group, University of Missouri-Columbia); Zachary M. Tretter (Division of Biochemistry, Bond Life Sciences Center, MU Metabolomics Center, Interdisciplinary Plant Group, University of Missouri-Columbia); James T. Koller (Division of Biochemistry, Bond Life Sciences Center, MU Metabolomics Center, Interdisciplinary Plant Group, University of Missouri-Columbia); John R. Cort (Biological Sciences Division, Pacific Northwest National Laboratory, Richland); Roger G. Linington (Department of Chemistry, Simon Fraser University, Burnaby); David S. Wishart (Department of Biological Sciences, University of Alberta, Edmonton); Lloyd W. Sumner (Division of Biochemistry, Bond Life Sciences Center, MU Metabolomics Center, Interdisciplinary Plant Group, University of Missouri-Columbia)

The NP-MRD.org is an open-access, user-friendly, FAIR complaint database consisting of the NMR data of the majority of known natural products, and available at https://np-mrd.org. NP-MRD.org is populated with NMR data for primary and secondary metabolites derived from plants, fungi, bacteria, marine organisms, and animals. This web-based tool also contains predicted and simulated 1H, 13C and J-coupling data generated via machine learning and density function theory (DFT) methods. NP-MRD.org is equipped with multiple search tools using text, molecular weight, chemical structures, and/or 1H/13C NMR chemical shifts. NP-MRD.org is the world's largest natural products NMR database that will help natural products chemists and metabolomics communities to elucidate and characterize the known as well as new compounds in the real time.

POSTER 207

Modern NMR methods provide insights into a Pd-catalyzed Allylic Alkylation with a unique mechanism

Presenter: Christina Thiele (Technische Universität Darmstadt)
All Authors: Johann J. Primozic (Technische Universität Darmstadt); Julian Ilgen (present adress: Universität Regensburg); Patrick Maibach (Technische Universität Darmstadt); Matthias Brauser (Technische Universität Darmstadt); Jonas Kind (Technische Universität Darmstadt); Christina Thiele (Technische Universität Darmstadt)

To shed light on the unprecedented stereoinduction in a Pd-catalysed allylic substitution reaction we performed mechanistic studies. Herein the details on the preparation and characterization of the Pd–allyl intermediates will be described. Special emphasis will be laid on the determination of the relative configuration of the up to four different diastereomeric eta1-Pd-allyl species observed simultaneously.

Our investigation implies a unique mechanism for allylic substitutions: It splits into a stereoconvergent process during oxidative addition and a diastereodivergent one for the attack of the nucleophile. Key to these studies was the use of modern NMR techniques such as EASY-ROESY, PSYCHEDELIC and fast methods of reaction monitoring. Their use on this challenging reaction will be described.

POSTER 208

Saturation-Transfer Difference NMR Reveals the Influence of Salt on Amino Acids Binding to Nanoplastics

Presenter: Rajan Rai (Clemson University)
All Authors: Rajan Rai (Clemson University); Anup Adhikari (Clemson University); Leah Casabianca (Clemson University)

Saturation-Transfer Difference NMR (STD NMR) spectroscopy is a versatile analytical technique employed for investigating the interplay between a receptor and a small molecule ligand. This approach yields important insights into both the binding epitope and the affinity of the ligand toward the receptor. We used STD NMR experiments to study the ability of several surface-functionalized plastic nanoparticles to bind different amino acids and to understand how the presence of salt influences their binding. The impacts of salts were found to be more pronounced in some nanoplastic-amino acid bindings in comparison to others, which leads to discussions on what factors are responsible for binding.

POSTER 209

Investigating Galactoside-Human Galectin-3 Interactions with Advanced NMR and Computational Methods

Presenter: István Timári (University of Debrecen)
All Authors: László Bence Farkas (Department of Organic Chemistry, University of Debrecen); Álex Kálmán Balogh (HUN-REN-UD Molecular Recognition and Interaction Research Group, University of Debrecen); Fanni Hőgye (Department of Organic Chemistry, University of Debrecen); László Szilágyi (Department of Organic Chemistry, University of Debrecen); Tünde Zita Illyés (Department of Organic Chemistry, University of Debrecen); Krisztina Fehér (HUN-REN-UD Molecular Recognition and Interaction Research Group, University of Debrecen); István Timári (University of Debrecen)

Human Galectin-3 (hGal-3) is a protein that selectively binds to β-galactosides and holds diverse roles in both normal and pathological circumstances. We have recently monitored the binding of some selenoglycosides to hGal-3 by NMR spectroscopy including improved 1H-77Se CPMG-HSQMBC method, X-ray crystallography and molecular dynamics (MD) simulations. As a step forward, we have synthesized and investigated derivatives of thiodigalactoside (TDG) modified with different aromatic substituents. Competition STD NMR method was applied to determine the dissociation constant (Kd) of three TDG derivatives produced. Based on the Kd values determined, the (naphthalen-2-yl)methyl, (quinolin-2-yl)methyl and benzyl derivatives bind to hGal-3 94-, 30- and 24-times more strongly than TDG. Then, we studied the binding modes of the derivatives in silico by molecular docking simulations.

POSTER 210

Comparison of T2 Increase Due to Cell Swelling and Membrane Potential Changes Using the Magnetization Transfer Two-pool Model.

Presenter: Seong-Min Kim (Sungkyunkwan University)
All Authors: Seong-Min Kim (Sungkyunkwan University); Kyeongseon Min (Seoul National University); Jang-Yeon Park (Sungkyunkwan University)

It was recently reported that changes in membrane potential can induce T2 changes that can be measured by MRI. Here, we investigated whether this T2 change due to membrane potential changes can be attributed to cell swelling and changes in macromolecular water pool that accompany membrane potential changes, using a magnetization transfer two-pool model. We demonstrated that T2 changes are influenced by both cell swelling and changes in the macromolecular water pool, with a greater influence of membrane potential changes. In particular, a decrease in the macromolecular water pool with short T2 can lead to an increase in T2. This finding identifies the role of macromolecular water pool in T2 increase during membrane potential changes.

POSTER 211

NMR Methods to Study the Effect of Screening Hits on Serine Racemase Enzyme Kinetics

Presenter: Charles Babu (Novartis Biomedical Research)

All Authors: Charles Babu (Novartis Biomedical Research); Bing Wang (Novartis Biomedical Research)

Functional deficiency in N-methyl-D-aspartate (NMDA) receptor results in schizophrenia-like symptoms in healthy humans. The NMDA receptor requires glutamate and one of the two co-agonists, D-serine, or glycine, to function. Serine racemase is an enzyme that converts L-serine to D-serine and produces pyruvate. Activating serine racemase will increase D-serine levels, thus improving NMDA receptor function. This poster will present several NMR methods that were developed to validate drug discovery screening hits. 2H-NMR was used to monitor conversion of L-Serine (2,3,3-D3) to D-Serine. Using two reagents, a generally applicable NMR method for measuring L- and D-amino acid ratios was developed and used. Finally, a new L-serine-3d substrate was used to measure L- to D-Serine conversion and pyruvate production.

POSTER 212

Enhancing Efficiency of Structure Revision by Combination of CASE and DFT Methods

Presenter: Alexei Buevich (Merck)

All Authors: Alexei Buevich (Merck); Mikhail Elyashberg (ACD/Labs); Sriram Tyagarajan (Merck); Mihirbaran Mandal (Merck)

Structure revisions can be costly, necessitating extensive use of spectroscopic data, computational chemistry, and total synthesis. This becomes especially true when researchers must resynthesize a biologically active compound, only to find it inactive due to an incorrect and unknown structure. To mitigate these costs, we propose utilizing Computer-Assisted Structure Elucidation (CASE) with Density Functional Theory (DFT) methods for structure revisions. Our research demonstrates that with the CASE-DFT approach, incorrect structures can be identified within seconds, while correct structures can be determined within minutes to hours, using the originally published NMR and MS data. This approach remains effective even in cases where the spectral data are incomplete or contain typographical errors.

POSTER 213

Case Studies of Amide Cis-Trans Isomerization in NMR Structural Elucidation

Presenter: Shengtian Yang (Novartis)

All Authors: Shengtian Yang (Novartis); Kevin Hung (Novartis); Adam Lewis (Novartis); Sarah Kochanek (Novartis); Vanessa Marx (Novartis); Joe Young (Novartis); Patrick J. Rudewicz (Novartis)

Many pharmaceutical drug candidates have an amide bond in their structures. Amide formation was a top recurring reaction found in the medchem literature and as many as 25% of all synthetic pharmaceutical drugs contained an amide group. Correctly identifying compound structure and conformation is critical to understanding structure-activity relationships (SAR) in pharmaceutical research. The presence of an amide bond can complicate this process when restricted rotation along the C-N bond at room temperature results in a population of cis and trans rotamers. Activation energies of amide cis-trans isomerization (CTI) is estimated to be 17-21 kcal/mol. In this poster, we present 3 case studies where CTI resulted in a mixture of rotamers and NMR and computational approaches elucidated these structures.

POSTER 214

Investigation of the Effects on Proton Relaxation Times upon Encapsulation in a Water-Soluble Synthetic Receptor

Presenter: Quinlin Dixon-Lim (Scripps College)

All Authors: Quinlin Dixon-Lim (Scripps College); Krishna N. Chaudhary (Pitzer College); Kyra I. Brosnahan (Scripps College); Bethany G. Caulkins (Scripps College)

The effects of the sequestration of small molecule guests in water-soluble deep cavitand hosts on the longitudinal (T1) and transverse (T2) relaxation times of protons in variably sized guests are analyzed here, using inversion recovery and spin-echo experiments. Cavitand-bound neutral organic species experience reduced proton relaxations overall, but the magnitude of this effect varies between protons within the same molecule, induced by a variety of factors including the motion of the guest when bound, the position of the protons in the cavity, and the magnetic anisotropy induced by the aromatic walls of the host. These subtle effects can have large consequences on the environment experienced by the bound guest, shedding light on the nature of small molecules in confined environments.

POSTER 215

MISSTEC-S: A Fast Pulse Calibration from Spectra Simultaneously produced by a Spin Echo and a Stimulated Echo

Presenter: Serge Akoka (Nantes University)

All Authors: Sanchez Margot (CEISAM UMR CNRS 6230); Julien Pontabry (RS2D); Gaetan Assemat (RS2D); Anthony Martinez (RS2D); Serge Akoka (Nantes University)

A new sequence for RF calibration is presented (MISSTEC-S). It is derived from the previously proposed MISSTEC sequence, but observation of echoes in presence of magnetic field gradient is replaced by observation of FIDs.

This modification allows both obtained spectra to be phased, but induces a strong constraint on TM. But the relationship used to calculate the flip angle is only correct when TM is small enough to neglect longitudinal relaxation during this delay. In order to reduce TM the first FID is truncated during acquisition and subsequently lengthened using points from the second FID.

Results obtained with MISSTEC-S were compared to those obtained from a complete nutation curve and an excellent correlation was observed (R2=0.9994).

POSTER 216

Comparisons of Purity Determination Experiments for Organophorphorus and Organochlorine Compounds at High and Low Fields Using Multiple Nuclei

Presenter: David McGarvey (U.S. Army Chemical Biological Center)

All Authors: David McGarvey (U.S. Army Chemical Biological Center); Rachel R. Knoebel (U.S. Army Chemical Biological Center); William R. Creasy (Leidos Corp.)

Absolute NIST-traceable purity determination is an important aspect of many chemical research programs and quality control systems. In recent years, benchtop NMRs have become widely available. While using much lower field strength than NMRs with superconducting magnets, they offer much lower initial cost, and much lower maintenance costs over time. Helium shortages have made the maintenance of superconducting magnets even more difficult.

This research looks at a comparison in the precision and accuracy of higher and lower-field instruments, and offers data on the acceptability of the use of lower-field instruments for purity determinations.

POSTER 217

Solvent Dependence of s-cis/s-trans Conformational Preference in α-cyano 4'-dimethylaminochalcone

Presenter: Matt Wedzina (Grinnell College)

All Authors: Matt Wedzina (Grinnell College); T. Andrew Mobley (Grinnell College); Stephen R. Sieck (Grinnell College); Mitchell JR

McCloskey (Grinnell College); Ruoxu Xia (Grinnell College); Clare M. Favela (Grinnell College)

α-Cyano chalcone solution-state conformational behavior was studied in a variety of polar and nonpolar solvents. Dimethylamino-α-cyano chalcone was synthesized in a 96.9% yield by microwave irradiation of 4-dimethylaminobenzaldehyde and 13C-cyano-labeled-benzoylacetonitrile. The final product was 13C-labeled on the cyano-group carbon. Carbon-carbon coupling constants to this group were experimentally obtained using high resolution 13C NMR spectroscopy to observe s-cis and s-trans isomerism. By analyzing the changes in the coupling constants across different solvents, a trend was established between the conformation exhibited by the molecule in relation to the polarity of the solvent. Finally, DFT calculations on these chalcones were utilized to estimate conformational ratios.

POSTER 218

NMR Spectroscopy of Novichok Agents: From High Field to Zero Field

Presenter: Derrick Kaseman (Lawrence Livermore National Laboratory)

All Authors: Derrick Kaseman (Lawrence Livermore National Laboratory); Saphon Hok (Lawrence Livermore National Laboratory); Carlos Valdez (Lawrence Livermore National Laboratory)

Novichok Agents are small organophosphorus molecules that are lethal in small doses. Identification of these molecules using high field NMR is relatively straightforward using a combination of 1H, 19F, 31P, and 13C NMR, but becomes increasingly more challenging at lower magnetic fields as the chemical shift dispersion reduces and heteronuclear J-couplings dominate. Lower magnetic fields are desirable for portable NMR spectroscopy, which would enable Novichok Agents detection outside of a laboratory setting. To understand the evolution of the NMR spectra as a function of the magnetic field, the Agents are first characterized at 14.1 T to obtain chemical shifts, homonuclear, and heteronuclear J-couplings and subsequently extrapolated to Earth's field and finally in the Zero to Ultra Low Field regime.

POSTER 219

19F Solid-State NMR Analysis of Polymers Used in Food Packaging

Presenter: Jennifer Janovick (US FDA)

All Authors: Jennifer Janovick (US FDA); Clark Ridge (US FDA); Fu Chen (University of Maryland, Department of Chemistry and Biochemistry); Peter Scholl (US FDA); Katherine S. Carlos (US FDA)

Polymers containing fluorine have been applied to paper-based food packaging to impart grease and water resistance. Assessing the identity and structure of additives and coatings on papers and packaging can be challenging. We have applied solid-state, magic-angle-spinning, fluorine-19 NMR (19F-MAS-SSNMR) to several polymers and packaging materials to assess for fluorine content and the structure of the fluorine therein. Analytically determining the total fluorine content and total organic fluorine content in samples will be discussed. Sample handling and spectroscopic techniques were explored to increase sensitivity. Also, we will present steps and recommendations for quantification and the limitations of NMR with current preparation methods for the types of materials we analyzed.

Theory Computation and Data Processing in NMR (Posters 220-241)

POSTER 220

Enhancing Analytical Workflows: A Digital Twin for Automated Structure Verification and Quantification

Presenter: Lauren Lytwak (ACD/Labs)

All Authors: Lauren Lytwak (MilliporeSigma); Sarah Srokosz (ACD/Labs); Albert Farré Pérez (MilliporeSigma); Dimitris Argyropoulos (ACD/Labs); Shahriar Jahanbakht (ACD/Labs); Hans De Bie (ACD/Labs); Markus Obkircher (MilliporeSigma); Coralie Leonard (MilliporeSigma)

Highly pure physical reference materials have traditionally been integral to many structure verification and compound quantification workflows. However, acquiring, handling, and disposing of these materials is resource-intensive. ChemisTwin is an online analytical solution that serves as a digital twin of MilliporeSigma's library of physical reference materials. Using a database of digital reference materials (dRMs) that pair verified datasets with ACD/Lab's NMR prediction algorithms, it makes suitable reference data available to scientists on-demand. The platform automatically analyzes users' raw sample data and compiles the result, providing an efficient and sustainable alternative to conventional analytical testing methods. Illustrated through a case study, the platform proves its efficacy in verifying the identity of a target compound compared to closely related structures.

POSTER 221

POKY is becoming the ultimate AI solution for NMR studies

Presenter: Woonghee Lee (University of Colorado Denver)

All Authors: Woonghee Lee (University of Colorado Denver)

POKY is becoming the ultimate AI solution for NMR studies. It succeeds and surpasses Sparky with unmatched automation and ease. POKY covers commonly conducted tasks including processing (POKY Phaser), peak picking (APES, iPick) and decomposition of overlaps (REDEN), resonance assignments (I-PINE, ssPINE), 3D structure calculations (AUDANA, AUDASA), dynamics and binding studies (POKY Notepad, Perturbation/Intensity Analysis), NMR-based metabolomics (A-SIMA/A-MAP), high quality publishable figure creations, etc. POKY handles all data types (1D-to-nD) for both solution and solid-state NMR types with just a few mouse clicks. Powerful computing cluster servers are freely offered. Thousands of NMR spectra with step-by-step video tutorials are also provided for self-teaching through the SIM-PRAC module. The POKY team answers user questions within minutes from the user group.

POSTER 222

Range and Sensitivity of 17O Nuclear Spin-lattice Relaxation as a Probe for Aqueous Electrolyte Dynamics

Presenter: Chengtong Zhang (New York University)

All Authors: Chengtong Zhang (New York University); Alexej Jerschow (New York University)

The dependence of solution dynamics on the nature of electrolytes and their concentrations has been the subject of many experimental and computational studies, yet it remains challenging to obtain a full understanding of the factors that govern solution behavior. We provide additional insights into the behavior of aqueous solutions of alkali chlorides by combining 17O relaxation data with diffusion and viscosity and contrasting their behavior with 1H NMR relaxation. The main findings are that 17O relaxation correlates well with viscosity data, but not with diffusion data, while 1H relaxation correlates with neither. Notably, we also examine the ranges of the interactions and conclude that the majority of the effects are communicated via local water reorientation dynamics.

POSTER 223

Theory of Zero-Field Nuclear Magnetic Resonance for Fe-based Superconductors

Presenter: Jaafar Ansari (George Mason University)

All Authors: Jaafar Ansari (George Mason University); Karen L. Sauer (George Mason University)

Fe-based superconductors (FeSC) are high-temperature superconductors which can be studied using zero-field NMR (ZNMR) due to the presence of a strong internally-produced magnetic field and an electric field gradient (EFG) at the location of a quadrupolar nucleus. These magnetic and electric properties are intrinsic to the material, and thus can be studied without an external field using a single crystal or powder sample. In this talk, we present a straightforward, exact, closed-form solution to the ZNMR spectrum for FeSCs, including the eigenfrequencies, eigenstates, observed signal, Rabi frequencies, and the detectability of such signals. From these results, we analyze 75As ZNMR data in the literature, comparing to high-field results, and propose ZNMR experiments which can be performed to enhance these measurements.

POSTER 224
Breaking Boundaries: TINTO in POKY for Computer Vision-Based NMR Walking Strategies
Presenter: Zowie Werner (University of Colorado Denver)
All Authors: Andrea Estefania Lopez Giraldo (University of Colorado Denver); Zowie Werner (University of Colorado Denver); Mehdi Rahimi (University of Colorado Denver); Woonghee Lee (University of Colorado Denver)
Peak overlap and variable data quality often hamper traditional assignment methods. Addressing these challenges, we present TINTO (Two and three-dimensional Imaging for NMR sTrip Operation via CV/ML), a novel toolset by computer vision-based strip matching that does not rely on cross-peaks for sequential walking on the spectrum. TINTO utilizes Structural Similarity Index and Principal Component Analysis to perform visual similarity searches of resonances and quickly locate similar strips. We have developed two versions of TINTO within POKY: the stand-alone version of TINTO for 2D, and the integrated strip plot version of TINTO for 3D. Our benchmark results on Nsp7, Cl13 and Ubiquitin demonstrate that TINTO is a promising tool for accurate NMR assignment without the need for peak picking.

POSTER 225
Exploring Alternative Approaches for Meaningful Results after Automatic NMR Data Analysis
Presenter: Dimitris Argyropoulos (ACD/Labs)
All Authors: Dimitris Argyropoulos (Advanced Chemistry Development (ACD/Labs) Inc.); Sergey Golotvin (Advanced Chemistry Development (ACD/Labs) Inc.,); Rostislav Pol (Advanced Chemistry Development (ACD/Labs) Inc.,)
The advancement of NMR spectrometers and methodologies enables rapid acquisition of 1D and 2D spectra, but processing and interpreting the vast data pose challenges in structure elucidation workflows. This bottleneck intensifies with increasing regulatory demands. Automated Structure Verification (ASV) systems gain popularity as a solution. Their primary outcome, the Match Factor (MF), is a numeric value from 0 to 1 which gauges agreement between proposed structures and spectra. Herein, we thoroughly examine the MF's criteria, assessing reliability, real-life applicability, and limitations. We also explore the impact of multiple spectra and propose supplementary metrics for more meaningful ASV outcomes, supported by illustrative examples.

POSTER 226
Analyzing AlphaFold Structures with NMR Data
Presenter: Jake Williams (University of Chicago)
All Authors: Jake Williams (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 of amino acids. The AlphaFold Protein Structure Database currently contains over 200 million predicted structures. As AI protein prediction continues to advance, we examine the potential of hybrid techniques that combine experiment and computation that may yield more accurate structures than AI alone with significantly reduced experimental burden. We present a heuristic comparing N-edited NOESY spectra and AlphaFold predicted structures that seeks to determine whether the predicted structure reasonably describes the true structure of the protein.

POSTER 227
2D Peak Detection by Machine Learning Analysis of Contour Graphics
Presenter: Frank Delaglio (NIST IBBR)
All Authors: Rahul Torlapati (University of Maryland); Colin W. Wilburn (UCONN Health); Yulia Pustovalova (UCONN Health); Mark W. Maciejewski (UCONN Health); Joe Chalfoun (National Institute for Standards and Technology); Jeffrey C. Hoch (UCONN Health); John P. Marino (NIST IBBR); Frank Delaglio (NIST IBBR)
Identifying peaks is a required step in most biomolecular NMR workflows. We propose to perform peak identification using machine learning analysis of contour graphics, noting that identifying a feature as one or more peaks depends on the feature's shape, rather than on its absolute intensity. In our approach, realistic spectral regions are simulated by placing individual simulated or measured peaks so that the true peak positions are known. The simulations are used to generate contour images and corresponding segmentation mask images that show the known peak positions. Then, a semantic segmentation network is trained to reproduce the segmentation maps. We report our results in using this approach to identify unresolved peaks in 2D HSQC spectra of proteins.

POSTER 228
Building The Network for Advanced NMR Community
Presenter: Katherine Henzler-Wildman (UW-Madison)
All Authors: Songlin Wang (University of Wisconsin - Madison); Alexander Paterson (University of Wisconsin-Madison); Paulo Falco Cobra (University of Wisconsin - Madison); Paulo Pinheiro (University of Wisconsin - Madison); Boden Vanderloop (University of Wisconsin - Madison); Alex Eletsky (University of Georgia); Mario Uchimiya (University of Georgia); Abigail Moore (University of Georgia); John Glushka (University of Georgia); Laura Morris (University of Georgia); Jonathan Wedell (University of Connecticut Health Center); Chris Bontempi (University of Connecticut Health Center); Gerard Weatherby (UConn Health); Harrison Burr (University of Connecticut Health Center); Seenat Thongdee (University of Connecticut Health Center); Yulia Pustovalova (UConn Health); Michael Gryk (University of Connecticut Health Center); Qi Cheng (University of Connecticut Health Center); Mark Maciejewski (University of Connecticut Health Center); Arthur Edison (University of Georgia); Chad Rienstra (University of Wisconsin-Madison); Jeff Hoch (University of Connecticut Health Center); Katherine Henzler-Wildman (UW-Madison)
The goal of NAN, the Network for Advanced NMR, is to build an expandable resource to support collaboration and best practices within the NMR community and broaden the use of NMR by researchers across the scientific community. Within the NAN portal, the facility dashboard provides information on the instruments and expertise available across the network, helping users more easily find and access NMR resources. Data acquired on NAN spectrometers is automatically archived upon acquisition and can be accessed through the NAN data browser. The NAN portal also provides access to knowledgebases developed to promote best practices, facilitate sharing of pulse sequences and protocols between institutions, support reproducible acquisition of optimal NMR data, and reduce barriers to access for non-NMR experts.

POSTER 229
NMR Lineshape and Memory Effect Modeling with MD Simulations
Presenter: Louis Bouchard (UCLA)
All Authors: Mohamad Niknam (UCLA); Louis S. Bouchard (UCLA)
The dynamics of viscoelastic fluids are governed by a memory function, essential yet challenging to compute, especially when diffusion faces boundary restrictions. We propose a computational method that captures memory effects by analyzing the time-correlation function of the pressure tensor, a viscosity indicator, through the Stokes-Einstein equation's analytic continuation into the Laplace domain. We integrate this equation with molecular dynamics (MD) simulations to derive necessary parameters. Our approach computes NMR lineshapes using a generalized diffusion coefficient, accounting for temperature and confinement geometry. This method directly links the memory function with thermal transport parameters, facilitating accurate NMR signal computation for non-Markovian fluids in confined geometries.

POSTER 230
Optimal control flow encoding for time-efficient magnetic resonance velocimetry
Presenter: Mehrdad Alinaghian Jouzdani (PhD Student)
All Authors: Mehrdad Alinaghian Jouzdani (PhD Student); Mazin Jouda (KIT); Jan Gerrit Korvink (KIT)

Flow encoding MRI is traditionally achieved by applying bipolar gradients, which are applied after the excitation and cause a delay between the excitation and acquisition. In this study, we show that the velocity can be encoded into phase during the excitation time. It makes this method much more time-efficient. To achieve this goal, the excitation pulse needs to be redesigned. The optimal control theory is used to fulfill this purpose. The final results show a faster flow encoding and a much shorter echo time.

POSTER 231

Quantification of NMR Relaxometry Data with Machine Learning

Presenter: Shinjer Li (College of William and Mary Department of Chemistry)

All Authors: Shinjer Li (College of William and Mary Department of Chemistry); Daniel Vasiliu (College of William and Mary Department of Data Science); Tyler Meldrum (College of William and Mary Department of Chemistry)

The determination of T2s from NMR relaxometry data is difficult. Existing methods, such as the Inverse Laplace Transform and Matrix Pencil, offer some insight. However, both methods require subjective smoothing implementations, which may impact accuracy and reproducibility. We demonstrate a method to quantify T2s from NMR relaxometry data through the use of machine learning. We demonstrate this with two methods. The first is a bottom-up approach that relies on the Markov Chain Monte Carlo algorithm and a Cuckoo grid search algorithm based on Levy flights. The second is a top-down approach that utilizes a sparsity matrix. We show that these two approaches converge and show better T2-resolution than existing methods.

POSTER 232

Expanding the Scope of Neural Network-Based Recognition in NMR Spectra: Unlocking the Potential for Practical Metabolomics Analysis without Metabolite Assignments

Presenter: Kazuo Yamauchi (Okinawa Institute of Science and Technology)

All Authors: Kazuo YAMAUCHI (Okinawa Institute of Science and Technology); Zihao Song (Hokkaido University); Li Gan (Hokkaido University); Jiaxi Jiang (Hokkaido University); Zhiyan Hu (Hokkaido University); Yuki Ohnishi (Hokkaido University); Yasuhiro Kumaki (Hokkaido University); Tomoyasu Aizawa (Hokkaido University)

Our study addresses the manual expertise bottleneck in Nuclear Magnetic Resonance (NMR) spectroscopy, introducing a machine learning neural network system for spectra recognition and classification. Unlike other analytical methods, NMR data analysis lacks automation, requiring specialized skills for spectral assignment. Validated with standard samples, our neural network achieved high recognition accuracy, showcasing its robustness in diverse conditions. Extending this proof of concept to practical metabolomics, we focused on secretor/non-secretor classification of 2'-Fucosyllactose in human breast milk. Using a benchtop 60MHz NMR, we exclusively targeted classification, creating a real-world hospital-friendly protocol with a 96% accuracy rate. This underscores the transformative potential of machine learning neural networks in simplifying NMR analysis, enabling accessibility for non-experts.

POSTER 233

Expanding the Scope of High-Throughput NMR: Handling Peculiar 2D Peaks in Automated Structure Verification

Presenter: Karl Demmans (ACD/Labs)

All Authors: Karl Demmans (ACD/Labs); Michael McCarthy (ACD/Labs); Sergey Golotvin (ACD/Labs); Dimitris Argyropoulos (ACD/Labs); Uliana Bortnik (ACD/Labs)

Automated Structure Verification (ASV) systems can be very valuable in a high throughput environment, reducing the chemists' workload and increasing the confidence in the results. These systems are set up with standard acquisition parameters that work for most samples. However, there are cases where the HSQC-DEPT spectra recorded show either aliased or wrongly phased peaks for particular chemical groups, which can lead to serious problems in the automatic interpretation. In this poster, we examine ways in which an ASV system can handle this problem.

POSTER 234

NMR-Guided Refinement of Crystal Structures using 15N Chemical Shift Tensors.

Presenter: Ryan Toomey (Utah Valley University)

All Authors: Ryan Toomey (Utah Valley University); Luther Wang (Brigham Young University); Emily Heider (Utah Valley University); James Harper (Brigham Young University)

A new method using 15N NMR data refines crystal structures beyond the capabilities of traditional methods. Using six benchmark structures, the refinement improved agreement with NMR data without introducing significant errors. All structures show a statistically significant improvement in NMR fit over energy based refinements. Bond lengths, however, became slightly shorter, impacting the calculated 15N tensors. Further evaluation on tripeptides confirmed rapid convergence and high accuracy. The method's validity was corroborated by independent analysis and high agreement with experimentally determined structures. This approach offers significant improvements for crystal structure refinement.

POSTER 235

Discussion of Signal Averaging in Direct Imaging of Neuronal Activity (DIANA) Data Analysis

Presenter: Jae-Youn Keum (Sungkyunkwan University)

All Authors: Jae-Youn Keum (Sungkyunkwan University); Sophie Malaquin (CEA); Phan Tan Toi (Sungkyunkwan University); Cameron Héry (CEA); Eloïse Mougel (CEA); Celine Baligand (CEA); Julien Valette (CEA); Jang-Yeon Park (Sungkyunkwan University)

A novel fMRI method called DIANA (Direct Imaging of Neuronal Activity), which allows direct detection of neuronal activity and its propagation in somatosensory networks, was recently reported using anesthetized mice at 9.4 T with 5ms temporal resolution. Unfortunately, difficulty in replicating the DIANA signal has also been reported. While there may be several reasons worth discussing in this regard, here we discuss one of them specifically related to signal averaging in DIANA data analysis. This pertains to the proposed direct correlation between DIANA and neuronal activity which may profoundly affect the improvement of the sensitivity of DIANA responses through signal averaging.

POSTER 236

The BMRB archive of Protein, Nucleic Acid and Metabolite NMR Data

Presenter: Kumaran Baskaran (UCONN Health)

All Authors: Kumaran Baskaran (UCONN Health); Jonathan R. Wedell (UCONN Heath); Hongyang Yao (UCONN Health); Dimitri Maziuk (UCONN Health); Michael M. Gryk (UCONN Health); Hamid Eghbalnia (UConn Health); Jeffrey C. Hoch (UCONN Health)

The Biological Magnetic Resonance data Bank (BMRB: https://bmrb.io) serves the biomolecular NMR community by supporting a curated archive of primary and derived data and metadata linked to scientific investigations guided by the "FAIR Principles" (Findable, Accessible, Interoperable, and Reusable). BMRB seeks to empower scientists in their analysis of the structure, dynamics, and chemistry of biological systems. As of February 2024, the macromolecule archive contains over 11.4 million chemical shifts from 16297 entries. BMRB launched BMRbig (https://bmrbig.bmrb.io) in 2020 to accommodate time domain data and other diverse data (not just NMR data) beyond the types currently curated and annotated by BMRB. BMRB is supported by the US National Institute of General Medical Sciences under R24GM150793.

POSTER 237

Hostile Interference Rejection for NMR Signal Detection with Pseudorandom Phase Encoding

Presenter: Michael Malone (Los Alamos National Laboratory)

All Authors: Adam Altenhof (Los Alamos National Laboratory); Nicholas A. Dallmann (Los Alamos National Laboratory); Michael Malone (Los Alamos National Laboratory)

Using magnetic resonance in the field to do detection requires the ability to operate in the presence of uncontrollable, and even hostile, noise sources. We demonstrate a robust NMR signal detection scheme in the presence of an interferer with the same frequency as the NMR signal. By employing a pulse sequence that gives the NMR signal a pseudorandom phase we can correlate our signal to perform detection. The receiver operating characteristic (ROC) curve obtained with this new method against a CPMG scheme shows significant performance improvement without the need for special coils or multi-channel receivers.

POSTER 238

Time-domain processing of digitally filtered NMR data

Presenter: Peter Kiraly (JEOL UK)

All Authors: Peter Kiraly (JEOL UK); Ronald Crouch (JEOL USA); Vadim Zorin (JEOL UK); Paul Bowyer (JEOL UK)

Time correction of digitally filtered NMR data is critical to obtain high quality data when processing operations inherently need to be applied in the time domain. Here we present an implementation of an effective 'group delay' correction method which enables the obtainment of flat baselines when using solvent filter or backward linear prediction methods. It is compatible with data acquired by using any modern NMR spectrometers. The illustrative applications include removal of large unwanted solvent peaks simplifying the analysis of high dynamic range data and estimate of missing data points from the early part of the time domain data.

POSTER 239

PDBx/mmCIF Ecosystem for NMR Structures

Presenter: Hamid Eghbalnia (UConn Health)

All Authors: Hamid Eghbalnia (UConn Health); Kumaran Baskaran (UCONN Health); Jonathan Wedell (UConn Health); Hongyang Yao (UConn Health); Dimitri Maziuk (UConn Health); Michael R. Gryk (UConn Health); Jeffrey Hoch (UConn Health)

The Macromolecular Crystallographic Information File (mmCIF), also known as PDBx/mmCIF, is a standard text file format for representation and exchange of experimentally determined three-dimensional (3D) macromolecular structure data. It is the adopted standard for all data processing and annotation by the Worldwide Protein Data Bank (wwPDB, wwpdb.org). Because mmCIF can be easily extended to include representations that characterize unique structural features such as disorder, multiple conformers, and dynamic parameters, it is extremely well-suited for aligning data from NMR studies with atomic coordinates. PDB-Dev (https://pdb-dev.wwpdb.org) uses the mmCIF ecosystem to represent biological assemblies derived using integrative and hybrid methods. The dictionary and format are machine-readable, assuring that the wwPDB partners can more effectively represent, biocurate, validate and distribute structural biology data.

POSTER 240

NMR Studies of the CISS Effect

Presenter: Thalia Georgiou (UCLA)

All Authors: Thalia Georgiou (UCLA); Vladimiro Mujica (ASU); Solmar Varela (Institute for Materials Science and Max Bergmann Center of Biomaterials); Louis Bouchard (UCLA)

We have developed a theory that presents the first theoretical explanation for a finding reporting enantiospecific NMR responses in ss-CP experiments, by deriving an effective spin-Hamiltonian for valence electrons in DNA. We provide a foundation for the assertion that NMR possesses the capacity to discern enantiospecific responses through indirect coupling of nuclear spins mediated by conduction band electrons. DFT calculations of amino acids also confirm the dependence of J-couplings on the choice of enantiomer. In addition, both analytical (DNA) and numerical (DFT) models confirm the effects of the NMR shift on chirality. Our analysis shows that chiral discrimination of amino acids using NMR may be possible without a chiral solvent and proves that NMR parameters can be sensitive to chirality.

POSTER 241

Reconciling Theory and Experiment With Dynamics – NMR of α-Testosterone, a Case Study

Presenter: Alexander Elliott (Brigham Young University)

All Authors: Alexander Elliott (Brigham Young University); James Harper (Brigham Young University)

In the α-phase of testosterone, the carbon-13 experimental NMR data for a single atomic site (C5) of each crystallographically distinct molecule defy modeling via gas phase or plane wave methods. Specifically, the cluster method of Beran et al. shows 5 ppm difference in C5 isotropic shift values, compared to 2-3 ppm elsewhere.

To perform NMR-guided geometry optimization, candidate structures are generated via Monte-Carlo sampling of atom positions, and shieldings are calculated using the rPBE-D2* method. Comparisons between calculated shieldings and experimental shifts measure structure quality. In this presentation, we explore the limits of NMR-guided geometry optimization utilizing only isotropic shift values. Using data previously reported, we find NMR modeling is improved if disorder is included at the hydrogen bond.

POSTER 242

Digging into Decoherence in 1D-NUS: Patterns in Weighted Sampling

Presenter: David Rovnyak (Bucknell University)

All Authors: Lucille Cullen (Bucknell University); Henry B. Rovnyak (Purdue University)

Principles for designing non-uniform sampling (NUS) are well known, but implementing them remains challenging. Schedulers such as Poisson gap and quantile methods, can each result in strong 1D-NUS schedules, but can also produce poor schedules depending on seed (Poisson) or parameter (quantile) choices. To address this need, we recently proposed tools and heuristics to evaluate patterns in weighted NUS and used them to develop '1-click' 1D-NUS schedules by the quantile method, which enabled both sparser and more reliable schedules. In this work, we develop the underlying concepts of this approach by examining patterns in random weighted sampling, which suggests decoherence criteria for schedule design. A kernel density function is developed to illuminate the regions of schedules prone to patterned sampling.

After install of EventPilot App

You will be prompted to enter the event code: ENC24

Manage your schedule
Download the free app

Scan the QR code or enter the URL in your device browser to download

Download on the App Store

GET IT ON Google Play

For all other devices Web Version

Powered by ativsoftware.com EventPilot®