Eleventh Annual Symposium of the UK High-Field solid-state NMR facility

Thursday March 31th, 2022

Scarman House, University of Warwick

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THE UK HIGH-FIELD SOLID-STATE NMR FACILITY
ANNUAL SYMPOSIUM PROGRAMME
THURSDAY MARCH 31TH, 2022

10:30 – 11:00	*Registration*

Morning Session
Chair: John Griffin

11:00 – 11.25	*Solid-State NMR Investigation of the Mixed-Metal Metal-Organic Framework (Al,Ga)-MIL-53* Zachary Davis, University of St Andrews
11:25 – 11:50	*New approaches to determining the atomic-level structure of halide perovskites for optoelectronics* Dr Dominik Kubicki, University of Warwick
11:50 – 12:15	*Understanding the Local Structure of Protective Alumina Coatings for Cathodes and the Coating-Cathode Interface* Dr Abby Haworth, Lancaster University
12:15 – 12:40	*Protein Choreography: Observing the local anisotropy of protein dynamics using solid-state NMR and Molecular Dynamics* Ben Tatman, University of Warwick
12:40 – 13:05	*The Bare Bones of Biomineralization: new insights into bone mineral composition, structure and formation* Prof. Melinda Duer, University of Cambridge
13:05	*Lunch*

Afternoon Session
Chair: Sharon Ashbrook

14:10 – 15:00	*Recent Developments in DNP Solid-State NMR Spectroscopy at High Magnetic Field and Fast Magic Angle Spinning* Dr Anne Lesage, École Normale Supériere de Lyon
15:00 – 15:25	*Showcasing Advanced NMR Approaches to Probe Li Ion Dynamics in Several Crystal Structures* Ben Duff, University of Liverpool
15:25	*Coffee Break*
16:00 – 16:25	*^{19}F magic-angle spinning NMR as a new tool to investigate the effect of fluorination on partitioning in cell membranes* Dr Philip Williamson, University of Southampton
16:25 – 16:50	*Solid-state NMR Applications in the Pharmaceutical Industry* Dr Andrew Tatton, GlaxoSmithKline
16:50	*Close of Meeting*

Zachary Davis, University of St Andrews**:**

Solid-State NMR Investigation of the Mixed-Metal Metal-Organic Framework (Al,Ga)-MIL-53

Metal-Organic Frameworks (MOFs) are important microporous materials with a range of applications in gas storage, catalysis and drug delivery.[1] This work utilises solid-state NMR to analyse changes in the breathing behaviour of mixed-metal (Al,Ga)-MIL-53 depending on the metal cation composition. These frameworks are synthesised using cost-effective routes such as a dry gel conversion method to enable ^{17}O enrichment, allowing for the acquisition of high-resolution spectra in a reasonable timeframe.[2–3] Additionally, exploration of post synthetic ion exchange reactions has been undertaken to better understand the cation distribution within these materials.

References

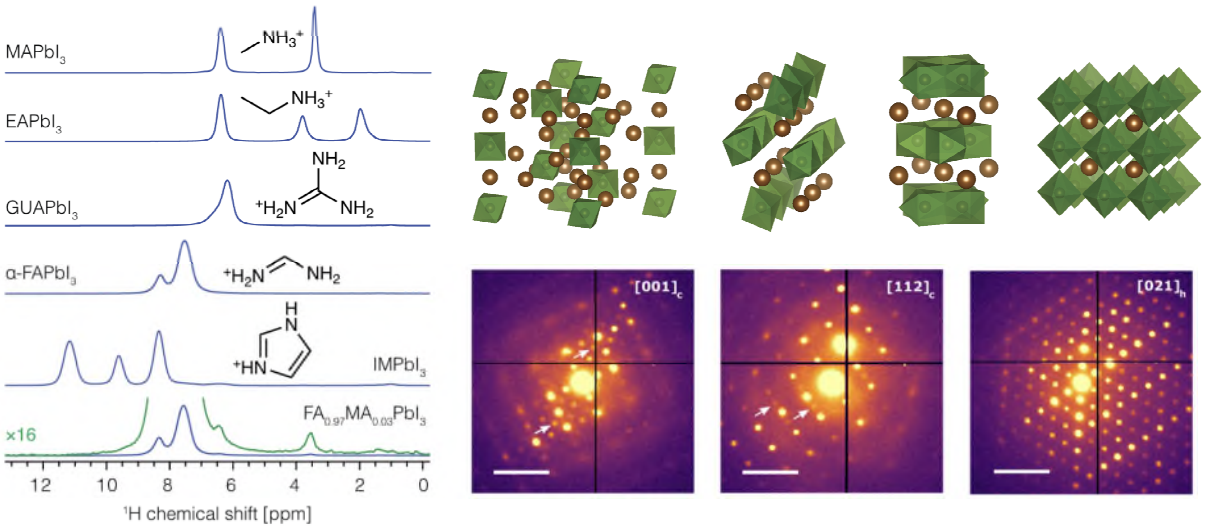
1. P. Wright, Microporous Framework Solids, RSC Publishing, Cambridge, 2008.
2. G. P. M. Bignami *et al.*, *Chem. Sci.*, 2018, **9**, 850.
3. C. Rice *et al.*, *Phys. Chem. Chem. Phys.*, 2020, **22**, 14514.

Dominik Kubicki, University of Warwick**:**

New approaches to determining the atomic-level structure of halide perovskites for optoelectronics

Determining the structure-property relationships at multiple length scales is one of the key tenets of rational design of new materials. While diffraction techniques offer insight into the long-range structure of solids, many properties are determined by local structure, which can be accessed using approaches based on, e.g., total scattering (PDF), XAFS, and magnetic resonance (NMR and ESR).

I will use the example of metal halide perovskites to discuss how we can determine the atomic-level structure of solids in an element-specific manner using solid-state NMR spectroscopy. The range of research problems includes quantifying dopant incorporation, phase segregation, decomposition pathways, passivation mechanisms, and structural dynamics.[1] I will also show how electron diffraction allows us to study structural phenomena inaccessible with X-rays.[2]



References

(1) Kubicki, D. J.; Stranks, S. D.; Grey, C. P.; Emsley, L. NMR Spectroscopy Probes Microstructure, Dynamics and Doping of Metal Halide Perovskites. *Nat. Rev. Chem.* 2021, **5**, 624–645.
(2) Doherty, T.; Nagane, S.; Kubicki, D. J.; Jung, Y. K.; Johnstone, D. N.; Iqbal, A.; Guo, D.; Frohna, K.; Danaie, M.; Tennyson, E. M.; MacPherson, S.; Abfalterer, A.; Anaya, M.; Chiang, Y.-H.; Crout, P.; Ruggeri, F. S.; Collins, S.; Grey, C. P.; Walsh, A.; Midgley, P.; Stranks, S. D. Stabilized Tilted-Octahedra Halide Perovskites Inhibit Local Formation of Performance-Limiting Phases. *Science* 2021, **374**, 1598–1605.
@DominikJKubicki http://kubickilab.wordpress.com/

Abby Haworth, Lancaster University**:**

Understanding the Local Structure of Protective Alumina Coatings for Cathodes and the Coating-Cathode Interface

Lithium nickel oxide ($LiNiO_2$ - LNO) has the potential to be used as a cathode material in batteries for the next-generation of electric vehicles. However, it suffers from capacity fade over multiple cycles.[1] Within the Faraday Institution-funded FutureCat project, our studies focus on extending the electrode's longevity via protective alumina coatings.[2] In order to improve and tailor these coatings, it is important to first fully understand the structure of the coating, in addition to any reactions, structural changes, or ion dynamics occurring at the coating-cathode interface. To probe this further, Al_2O_3 coatings have been studied using solid-state NMR spectroscopy. The latest ^{27}Al MAS NMR data, which will be presented here, provides insight into the local structure of the coatings and allows us to investigate the coating-cathode interface.

References

1. M. Bianchini, *et al. Angew. Chem. Int. Ed.* 2019, **58**, 10434-10458

2. S. G. Booth, *et al. APL Materials*, 2021, **9**, 109201

Ben Tatman, University of Warwick**:**

Protein Choreography: Observing the local anisotropy of protein dynamics using solid-state NMR and Molecular Dynamics

The local dynamics of proteins play a fundamentally important role with regards to their function. Using ultrafast magic-angle spinning and high-field solid-state NMR at variable temperatures it is possible to probe the anisotropy and thermodynamics of local protein motions over a range of timescales from picoseconds to microseconds. In this talk, I will introduce some of our recent work looking at the local motions within the beta-1 domain of Protein G using both solid-state NMR and Molecular Dynamics, and how access to the high-field 850 MHz and 1 GHz national facilities has assisted in this project.

Melinda Duer, University of Cambridge**:**

The Bare Bones of Biomineralization: new insights into bone mineral composition, structure and formation

The structure of bone and its mechanical properties are essential for our health and well-being. A key component of bone is the calcium phosphate mineral that forms in and around the underlying organic fibrils that form the bulk of bone tissue.

The formation and maintenance of bone mineral is highly complex, taking place in an equally complex physicochemical environment. Solid-state NMR spectroscopy has been key in understanding bone mineral structure and more recently, its formation.

In this talk, I will exemplify the latest insights into how bone mineral forms in vivo and discuss how solid-state NMR can be used to assess complex synthetic models of bone mineral, including the use of ^{43}Ca NMR which necessitates high field.

Anne Lesage, École Normale Supériere de Lyon**:**

Recent Developments in DNP Solid-State NMR Spectroscopy at High Magnetic Field and Fast Magic Angle Spinning

Over the last few decades, solid-state NMR has developed into an essential analytical tool to investigate the structure and dynamics of chemical and biological systems. While it can provide in many cases unprecedented insights into atomic-scale structures, solid-state NMR suffers from low sensitivity, which strongly limits its application fields. One possibility to increase the sensitivity of solid-state MAS NMR experiments is Dynamic Nuclear Polarization (DNP). DNP rely on a transfer of the large polarization of unpaired electrons to surrounding nuclei upon microwave irradiation. This technique, originally developed for low magnetic fields, has been shown to be applicable at high magnetic fields, opening new avenues to study molecular systems that were previously inaccessible to solid-state NMR studies.

In this presentation we will present some experimental developments in the field of heterogeneous catalysis and pharmaceutical research. In particular we will discuss results from high field (800 MHz) and fast MAS (~ 40 kHz) DNP NMR. New, highly efficient polarization sources suitable for high filed DNP will also be presented.

Ben Duff, University of Liverpool**:**

Showcasing Advanced NMR Approaches to Probe Li Ion Dynamics in Several Crystal Structures

Li-containing materials providing fast ion transport pathways are fundamental in Li solid electrolytes and the future of all-solid-state batteries. In this talk, I will introduce some of our recent work on the structural elucidation of potential solid electrolytes as well as the insights gained into the Li ion dynamics in these materials.

A range of variable temperature ^{6}Li and ^{7}Li NMR approaches such as ^{6}Li-^{6}Li EXSYs, ^{7}Li line narrowing and relaxometry were used in order to quantify Li ion dynamics as well as determine the

ion mobility pathways. The identification of factors limiting long range translational Li ion mobility in materials through the determination of Li ion correlation times via NMR, provide a framework for the further development of more highly conductive Li solid electrolytes.

Phil Williamson, University of Southampton**:**

^{19}F magic-angle spinning NMR as a new tool to investigate the effect of fluorination on partitioning in cell membranes

The lipophilicity of a compounds has been shown to be an excellent measure of the efficacy of orally administered drugs. It reflects a compounds ability to partition into the cell membrane, enabling it to cross the cell membrane and access its site of action within the cell. Fluorination is a much-utilized strategy to modify the biological and physical properties of candidate drugs but there have been few systematic studies to investigate how fluorination influences the partitioning of drugs into the lipid bilayer. Typical studies of lipophilicity report on the partitioning of a drug between water and octanol, however this fails to replicate the complex environment within cell membranes where the physicochemical properties of the lipids influence the spatial organisation and dynamics within the lipid bilayer. We have exploited the exquisite sensitivity of the ^{19}F chemical shift to local environment, together with ^{19}F MAS-NMR, to resolve the population of fluorinated drugs bound to the lipid bilayer from those in free solution. The absence of naturally occurring ^{19}F in the lipid bilayer or native cell membranes ensures that spectra are easy to interpret allowing reliable quantitative measurements of the drug partitioning to be made. This has enabled us to investigate how fluorination motifs influence drug partitioning and how this is influenced by the membrane composition.

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