Fifteenth Annual Symposium of the UK High-Field Solid-State NMR Facility

Tuesday 21st April 2026

Scarman Conference Centre, University of Warwick

UKRI Engineering and Physical Sciences Research Council
Biotechnology and Biological Sciences Research Council

Advantage West Midlands
www.advantagewm.co.uk

investing in your future
European Regional Development Fund
European Union

UK HIGH-FIELD SOLID-STATE NMR FACILITY

The UK High-Field Solid-State NMR Facility
Annual Symposium Programme
Tuesday 21st April 2026

10:30-11:00 Registration/Coffee

Morning Session (Chair: Prof. Frédéric Blanc, University of Liverpool)

11:00-11:05 Introduction

11:05-11:30 Prof. Mark Smith, University of Southampton, "Prof. Ray Dupree a UK Pioneer of Magnetic Resonance as a Probe of Materials – a personal perspective."

11:30-11:50 Dr. Roslie Thompson, University of Warwick, "Using 2D solid-state NMR to resolve the structure of cellulose in plant cell walls."

11:50-12:15 Dr Daniel Lee, University of Manchester, "Surface science using DNP-enhanced NMR spectroscopy."

12:15-12:30 Prof Steven Brown, University of Warwick, "Update on the UK High-Field Solid-State NMR National Research Facility"

12:30-13:15 Lunch

First Afternoon Session (Chair: Prof. Phil Williamson, University of Southampton)

13:15-13:40: Dr Katherine Stott, University of Cambridge, "Control of DNA condensation by ultra-low complexity linker histone tails"

13:40-14:00 Prof Yaroslav Khimyak, University of East Anglia, "Understanding of assembly of multi-component pharmaceutical solids using high-field in-situ CLASSIC NMR spectroscopy"

14:00-14:20 Malavika Manoj, St Andrew's University, "Investigating the aluminium environments in zeolites using NMR spectroscopy"

14:20-14:40 Dr Benjamin Gallant, University of Birmingham, "Atomic-level mechanisms of degradation in halide perovskite photovoltaics".

14:40-15:10 Coffee Break

Second Afternoon Session (Chair: Prof. Sharon Ashbrook, St Andrews University)

15.10-15:30 Kyle Watson, University of Liverpool, "Host-Guest Interactions of Biomass Mimic Guaiacol Revealed by Solid-State NMR in ^{17}O Enriched ZSM-5"

15:30-15:50 Dr Ieva Goldberga, University of Warwick, "Spin Fast, Pulse Fast: SOFAST and BEST at 150 kHz Magic Angle Spinning."

15:50-16:30 Prof. Clare Grey, Cambridge University, "Paramagnetic NMR: from early ^{89}Y studies at Warwick to battery materials and DNP"

16:30 End of symposium

Prof. Ray Dupree a UK Pioneer of Magnetic Resonance as a Probe of Materials – a personal perspective.

Mark Smith

Vice-Chancellor's Office and the Department of Chemistry, University of Southampton, United Kingdom.

Professor Ray Dupree made many significant contributions to the development and application of magnetic resonance across his sixty-year research career. An overview of his research career will be presented with some of his key works across that period highlighted. The examples picked will illustrate where Ray led the way. In the context of the National Research Facility for Solid-State NMR, for which Ray was a founding member of the Facility Executive, it is intended to demonstrate how many of those advances in his research career were inextricably linked with the availability of increasingly high magnetic fields. Some historical context is given of the advances of such magnetic fields over Ray's career.

Acknowledgments

MES thanks RD for his mentorship and help as he developed his own research career in solid-state NMR. EPSRC and its predecessor body SERC are thanked for their support over many decades of solid-state NMR infrastructure at Warwick and a wide range of research projects. HEFCE and the former Regional Development Agency, Advantage West Midlands are also thanked for their generous support of NMR infrastructure.

Using 2D solid-state NMR to resolve the structure of cellulose in plant cell walls.

Rosalie Cresswell[a]*, Parveen Kumar Deralia[b], Yoshihisa Yoshimi[b], Steven P. Brown[a], Paul Dupree[b], Ray Dupree[a]

[a] *The Department of Physics, University of Warwick, UK*
[b] *The Department of Biochemistry, University of Cambridge, UK*

* Rosalie.cresswell@warwick.ac.uk

Cellulose is the most abundant natural polymer on earth and its properties hold substantial interest as a sustainable alternative to petrochemicals for producing materials and biofuels.[1] The structure of crystalline cellulose fibrils has been widely debated over several decades;[2] however, due to these microfibrils having a width of ~3 nm, the resolution of X-ray diffraction techniques is poor. Thus, solid-state NMR is ideal for understanding cellulose structure as it does not require long-range order as is the case for diffraction techniques. Overlapping ^{13}C chemical shifts of cell wall components means that the resolution and thus the information obtained from a 1D ^{13}C MAS NMR spectrum is limited. By using ^{13}C labelled never-dried material, 2D MAS NMR experiments become feasible and have been used recently to reveal insights into the molecular structure of cell walls of a range of plants.[3-5]

Using a combination of standard and water-edited CP refocussed INADEQUATE and CP PDSD ^{13}C MAS NMR experiments at the NRF's high field NMR spectrometers, we found there are six major glucose environments common to the cellulose in the secondary cell walls of hardwoods, softwoods and grasses. These glucose environments were maintained when we made isolated holo-cellulose nanofibrils (hCNFs) from poplar wood by removing lignin and xylan, resulting in a simplified NMR spectrum. Water-edited experiments show that there are only two glucose environments in the fibril interior which have identical ^{13}C chemical shifts to cellulose Iβ (Fig. 1a).[6] This is contrary to previous work which described plant cellulose as a mixture of two cellulose I allomorphs[6] or alternatively its own distinct structure.[3] A glucose environment at ~89 ppm, previously assigned as an interior site, in fact resides on the surface of the microfibril (Fig. 1b and c).[6] This new insight into the cellulose structure will aid development of new biomaterials, the treatment of pulp, and the understanding of plant cell wall structure.

References

1. Foroughi, F., Ghomi, E. R., Dehaghi, F. M., et. al., *Materials,* 2021, **14**, 714
2. Atalla, R., Vanderhart, D., *Science*, 1984, **223**, 283-285
3. Wang, T., Yang, H., Kubicki, J. D., Hong, M., *Biomacromolecules.*, 2016, **17**, 2210-2222
4. Simmons, T.J., Mortimer, J. C., Bernardinelli, O. D, et. al., *Nat.Commun.*, 2016, **7**, 13902
5. Terrett, O. M., Lyczakowski, J. J., Yu, L., et. al., *Nat.Commun.*, 2019, **10**, 4978
6. Cresswell, R, Kumar, P. K., Yoshihisa, Y., *J. Am. Chem. Soc.*, 2025, **147**, 51, 47223–47236
7. Kirui, A., Ling, Z., Kang, X., et. al., *Cellulose,* 2019, **26**, 329

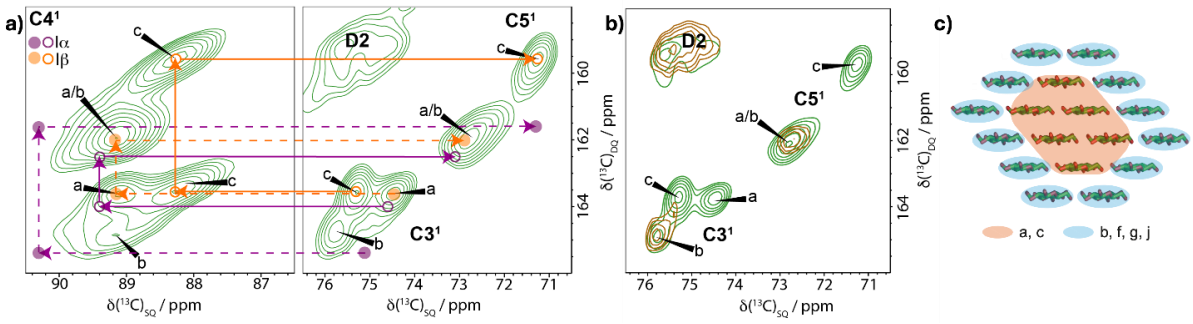

Figure 1: a) C4-C3/C5 of spectral domain 1 region of ^{13}C CP refocussed INADEQUATE of hCNFs of poplar stem compared to the ^{13}C NMR chemical shifts of cellulose I allomorphs. b) Comparison of the C3/C5 region of water edited (brown) and standard (green) ^{13}C CP refocussed INADEQUATE MAS NMR spectra of hCNFs of poplar stem. c) Example model habit of cellulose microfibril with 18-chains that is consistent with the MAS NMR spectra.[6]

Surface science using DNP-enhanced NMR spectroscopy.

Daniel Lee

Department of Chemical Engineering, University of Manchester, UK

Daniel.Lee@manchester.ac.uk

Dynamic nuclear polarisation (DNP) has transformed the scope of solid-state NMR spectroscopy by enabling orders-of-magnitude sensitivity enhancements, making it possible to probe surfaces, interfaces, and dilute species that were previously beyond experimental reach. In the UK, recent investments in DNP magic-angle spinning (MAS) NMR instrumentation have significantly strengthened the national infrastructure, complementing developments at the high-field NMR facilities and expanding opportunities for researchers across the physical, chemical, and biological sciences.

In this talk, I will outline the role of the UK DNP MAS NMR Facility in supporting the UK NMR and wider scientific community and discuss recent upgrades to the DNP infrastructure that enhance experimental capability and accessibility. I will then focus on applications of DNP-enhanced MAS NMR spectroscopy to problems in surface science. It will be illustrated how sensitivity enhancements enable the characterisation of surface species and interfacial structures, and how this can be linked to material properties and used for rational material design.

Control of DNA condensation by ultra-low complexity linker histone tails

Katherine Stott

Department of Chemistry, University of Cambridge, UK

Linker histones play an essential role in chromatin packaging by facilitating compaction of the 11-nm fibre of nucleosomal "beads on a string". The result is a heterogeneous condensed state with local properties that range from dynamic, irregular, and liquid-like, to stable and regular structures (30-nm fibres), which in turn impact chromatin-dependent activities at a fundamental level. The properties of the condensed state depend on the type of linker histone, particularly on the highly disordered C-terminal tail, which is the most variable region of the protein, both between species, and within the various subtypes and cell-type specific variants of a given organism. Our over-arching research question is how nature encodes function in proteins without stable structure. To answer this question for the linker histones, we have developed in vitro systems to model linker histone-condensed chromatin, which reveal that linker histones act as "liquid-like glue" to generate distinct states[1,2] that can be mapped directly back to full chromatin[3]. We use an integrative methodology combining NMR with other biophysical methods. Both solution- and solid-state NMR are essential for understanding the structures and transitions that bring about condensation. However, chemical shift assignment is especially challenging for the linker histone tails due to their low sequence complexity, necessitating the highest magnetic fields. We have so far leveraged our system to rationalise the distinct condensing properties of linker histone subtypes and variants across species that are encoded by the amino acid content and post-translational modifications of their C-terminal tails. With this we have uncovered a general set of molecular grammars that drive DNA condensation4. Some linker histones appear to retain their disorder in the condensed state, while others undergo structuring transitions. A "multi-state" NMR approach is necessary to understand the full picture.

References

1. Turner, A. L., Watson, M., Wilkins, O. G., Cato, L., Travers, A., Thomas, J. O. & Stott, K. Highly disordered histone H1-DNA model complexes and their condensates. Proc Natl Acad Sci U S A 115, 11964–11969 (2018). https://doi.org/10.1073/pnas.1805943115
2. Gibbs, E. B. & Kriwacki, R. W. Linker histones as liquid-like glue for chromatin. Proc Natl Acad Sci U S A 115, 11868–11870 (2018). https://doi.org/10.1073/PNAS.1816936115
3. Watson, M. & Stott, K. Disordered domains in chromatin-binding proteins. Essays Biochem 63, 147–156 (2019). https://doi.org/10.1042/EBC20180068
4. Watson, M., Sabirova, D., Hardy, M. C., Pan, Y., Carpentier, D. C. J., Yates, H., Wright, C. J., Chan, W. H., Destan, E. & Stott, K. A DNA condensation code for linker histones. Proc Natl Acad Sci U S A 121, (2024). https://doi.org/10.1073/pnas.2409167121

Understanding of assembly of multi-component pharmaceutical solids using high-field in-situ CLASSIC NMR spectroscopy

Anna Gołkowska,[2] Joseph Adjimani,[1] Maciej Nowak,[2] Karol Nartowski,[1, 2, ^] László Fábián,[1] Yaroslav Z. Khimyak[1*]

[1] *University of East Anglia, School of Chemistry, Pharmacy and Pharmacology, Norwich Research Park, NR4 7TJ Norwich, UK*

[2] *Wroclaw Medical University, Department of Drug Form Technology, Borowska 211A, 50-556 Wroclaw, Poland*

** E-mail: Y.Khimyak@uea.ac.uk, ^Deceased author*

Pharmaceutical cocrystals are multicomponent materials prone to exhibit polymorphism. The polymorphic outcome could be controlled using mechanochemical cocrystallisation techniques *via* the addition of solvents or polymers of different properties.[1,2] *In-situ* monitoring methods, such as synchrotron TRIS-PXRD, are used to gain knowledge of the mechanisms of such phase transitions. However, this methodology is limited in focussing exclusively on solid-state components of the reaction.

We have used high-field CLASSIC (*Combined Liquid- And Solid-State In-Situ Crystallisation*) NMR[3] as a versatile *in-situ* monitoring technique to gain a molecular-level understanding of crystallisation pathways during liquid- and polymer-assisted mechanochemical cocrystallisation processes. As models, cocrystals of theophylline and benzamide (TP:BZ 1:1) and metronidazole and gallic acid (MNZ:GAL 1:1) were selected due to their known polymorphism.[1,4] This methodology was also applied to investigate the crystallisation processes of cocrystals of nicotinamide and isonicotinamide with 3-hydroxybenzoic acid (3HBA), 2-hydroxybenzoic acid (2HBA), and 2,3-dihydroxybenzoic acid (2,3-DHBA), of varying stoichiometry. The selection of these compounds is driven by their isomeric relationships yet stark difference in hydrogen bonding patterns, providing a platform to investigate the influence of molecular structure on cocrystal formation.

These data enabled us to observe phase transitions of neat compounds and polymorphs of cocrystals forming *in-situ* when the starting physical mixtures were subjected to mechanochemical force arising from the spinning of the sample at the magic angle. In summary, CLASSIC NMR spectroscopy can be used successfully for *in-situ* monitoring and understanding the mechanism of solvent- or polymer-mediated mechanochemical reactions. We proved it to be an effective tool for following the solid-state phase evolution in time, comparable to synchrotron TRIS-PXRD.

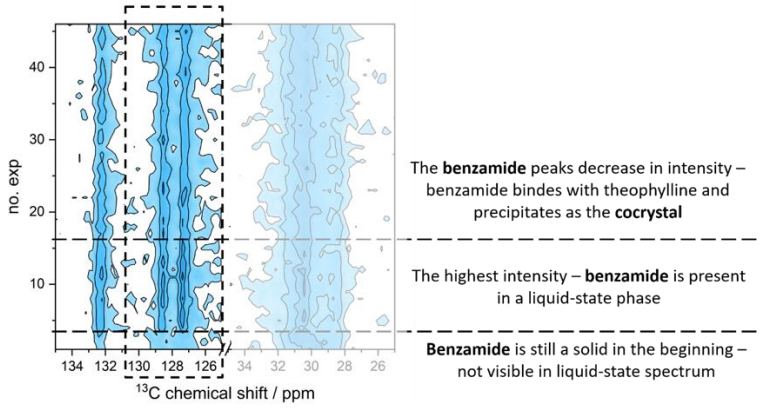

Figure 1: The phase behaviour of benzamide in TP:BZ system based on time-resolved $^{13}C\{^1H\}$ MAS NMR spectra.

References

1. Fischer, F. *et al*. *Cryst Growth Des*. 2016, 16, 3, 1701–1707.
2. Hasa, D. *et al*. *Cryst. Growth Des*. 2016, 16, 3, 1772–1779.
3. Hughes, C.E. et al. *Angew. Chem. Int. Ed.* 2014, 53: 8939-8943.
4. Dyba, A.J. et al. *Cryst. Growth Des*. 2023, 23, 11, 8241-8260

Investigating reversibility of aluminium coordination in zeolites

Malavika Manoj

Malavika Manoj[1], Edward J. Abram[2], Sharon E. Ashbrook[1], Paul A. Wright[1]

[1] *EaStCHEM School of Chemistry, University of St Andrews, St Andrews KY16 9ST, United Kingdom*
[2] *Johnson Matthey Davy Technologies Ltd, Billingham TS23 1LB, United Kingdom*
mm574@st-andrews.ac.uk

Aluminosilicate zeolites are important for acid-catalysed chemical conversions. Their acidity derives from the substitution of aluminium for silicon in their tetrahedrally coordinated framework and the associated presence of charge-balancing protons. Brønsted acidity arising from the bridging framework hydroxyl groups is well studied but the origin of Lewis acidity due to the presence of coordinatively unsaturated aluminium cations remains poorly understood. Here, different synthesis routes have been used to prepare zeolite frameworks with a range of Si/Al ratios which have been characterised using powder X-ray diffraction, scanning electron microscopy and solid-state NMR spectroscopy, including ^{27}Al, ^{29}Si and also ^{27}Al MQMAS NMR experiments. The Al environment in the acid forms of these zeolites is found to be a complex function of synthetic route, zeolite structure type and composition, and chemical manipulation.

While octahedral Al is generally considered to be extra-framework, recent work shows the possibility that this Al could be framework-associated.[1-4] A protocol was established for ammonium exchange and deammoniation on the small pore zeolite Rho, as shown in Figure 1, indicating tetrahedral and octahedral species. Reversible coordination change is confirmed. ^{27}Al multiple-quantum MAS NMR spectra of the H-forms show only tetrahedral and octahedral species and analysis show little or no loss of Al during cycles of ammonium exchange and deammoniation, while ^{29}Si MAS NMR spectra showed that the framework Si/Al ratio remained constant.

Extending the protocol to a wide range of zeolites with different framework types and compositions shows that the tendency of Al to become octahedral is dependent on structure type and post-synthetic treatment as well as the Al content. The consequences of this behaviour for zeolite stability and acidity will be discussed.

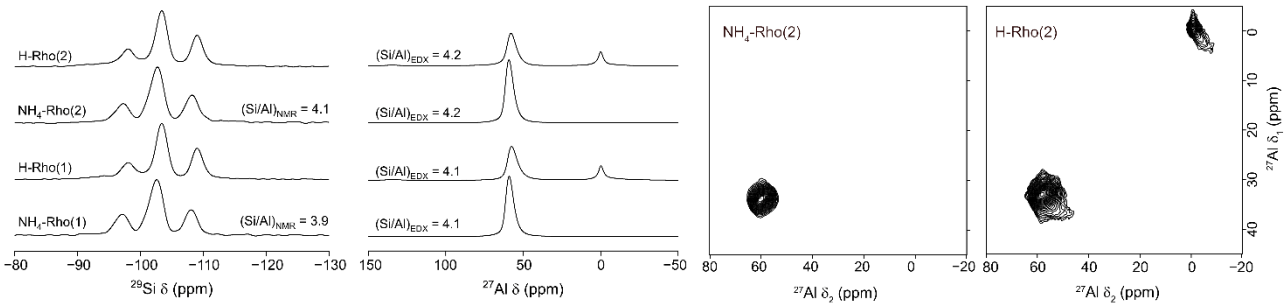

Figure 1. NMR spectra of zeolite Rho showing reversibility of tetrahedral-octahedral transformation during ammonium exchange and deammoniation cycles. (Left) ^{29}Si and ^{27}Al MAS NMR spectra of sequentially ammonium-exchanged and deammoniated zeolite Rho. (Right) MQMAS NMR spectra of (from left to right) NH_4- and H-forms of Rho in the second cycle.

References

1. Ravi, M., Sushkevich, V. L., van Bokhoven, J. A., Nat. Mater, 19, 1047-1056 (2020).
2. Ravi, M., Sushkevich, V. L., van Bokhoven, J. A., Chem. Sci., 12, 4094-4103 (2021).
3. Yakimov, A. V., Ravi, M., Verel, R., Sushkevich, V. L., van Bokhoven, J. A., Coperet, C., J. Am. Chem. Soc., 144, 10377-10385 (2022).
4. Jin, M., Ravi, M., Lei, C., Heard, C. J., Brivio, F., Tosner, Z., Grajciar, L., van Bokhoven, J. A., Nachtigall, P., Angew. Chem. Int. Ed., 62, e202306183 (2023)

Atomic-level mechanisms of degradation in halide perovskite photovoltaics

Benjamin Gallant

School of Chemistry, University of Birmingham, UK
Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Berlin, DE
b.gallant@bham.ac.uk | benjamin.gallant@helmholtz-berlin.de

Metal halide perovskites (ABX_3) have emerged as a promising class of thin film semiconductors for a range of optoelectronic applications, in particular photovoltaics. Their compatibility with solution-processing techniques, extraordinarily high tolerance of defects and soft, polycrystalline nature render them facile and inexpensive to fabricate.

However, halide perovskites are infamous for their phase and compositional instability in the face of environmental stimuli, in particular light and moisture. For example, perovskites rich in either formamidinium or cesium lead triiodide ($FAPbI_3$/$CsPbI_3$) are especially prone to photoactive phase instability, while mixed-ion compositions tend to rapidly de-mix *via* ion diffusion, which is unusually rapid in these materials. To make matters worse, in functional optoelectronic devices the enormous interfacial surface areas between perovskite layers and other passivation, charge transport, buffer or electrode layers act as further 'supercharged' regions for local degradation, with even nanometer-thin domains of undesirable phases capable of severely reducing device performance.
But there is hope!

Here I will discuss two key mechanisms that underpin halide perovskite degradation: (1) interaction with moisture, and (2) thermally-activated ion diffusion. Recently we have employed multinuclear NMR approaches to understand both these phenomena at the atomic level, including the use of surface-selective ^{17}O-enrichment to enable rapid identification of trace moisture-induced reaction products and variable-temperature quadrupolar NMR up to the halide perovskite melting point (~500 °C). Based on our findings, I will propose to strategies by which to minimise degradation occurring *via* either route.

Host-Guest Interactions of Biomass Mimic Guaiacol Revealed by Solid-State NMR Spectroscopy in ^{17}O Enriched ZSM-5

Kyle A. Watson,[a] Ming-Feng Hsieh,[b] Stephen Day,[c] Luke Tuxworth,[b] and Frédéric Blanc[a,d,e]

[a] *Department of Chemistry, University of Liverpool, Liverpool L69 7ZD, UK*
[b] *Johnson Matthey Technology Centre, Billingham TS23 1LB, UK*
[c] *Johnson Matthey Technology Centre, Sonning Common RG4 9NH, UK*
[d] *Stephenson Institute for Renewable Energy, University of Liverpool, Liverpool L69 7ZF, UK*
[e] *Leverhulme Research Centre for Functional Materials Design, University of Liverpool, Liverpool L7 3NY, UK*

High-resolution solid-state NMR spectroscopy provides a powerful approach for probing host-guest interactions in heterogeneous catalysts. Here, we showcase a suite of multinuclear NMR methods used to investigate the adsorption, confinement and reactivity of oxygenated biomass-derived molecules within aluminosilicate zeolites relevant to catalytic fast pyrolysis and bio-oil upgrading. Using guaiacol and others as bio-oil model compounds, ^{1}H and ^{13}C MAS NMR experiments reveal distinct in-pore and ex-pore species based on signal linewidth, chemical-shift dispersion, and mobility-dependent relaxation behaviour in both adsorbed and activated ZSM-5 zeolite via quantitative 1D and correlation measurements. Crucially, the ^{17}O isotopic enrichment of ZSM-5 has transformed zeolite framework oxygens into active probes of host-guest interactions. Two-dimensional ^{1}H-^{17}O dipolar heteronuclear multiple-quantum coherence (D-HMQC) NMR experiments were performed to selectively detect short-range heteronuclear dipolar couplings, providing sensitivity to the spatial proximity and confinement geometries of reactive species trapped within the channel intersections and sinusoidal/straight pores. Numerical simulations of the ^{1}H-^{17}O signal intensities and recoupling efficiencies provide estimates of dipolar coupling constants and hence insight into the spatial arrangement, restricted motion, and local binding environments of trapped guaiacol. Together, these results reveal distinct confinement regimes and differences in guest–framework hydrogen-bonding motifs, providing molecular level insight which can inform the rational design of next generation catalysts with faster activity, better selectivity and improved stability for catalytic upgrading.

Spin Fast, Pulse Fast: SOFAST and BEST at 150 kHz Magic Angle Spinning

Trent Franks, **Ieva Goldberga**, Jairah Lubay, Józef R. Lewandowski

Department of Chemistry, University of Warwick, UK

A key challenge in applying solid-state NMR to biological systems is its inherently low sensitivity. To achieve a sufficient signal-to-noise (SNR) ratio, multiple transients are typically required, with experimental time frequently extending over several days.[1] Various approaches have been introduced to boost sensitivity, including paramagnetic doping. Despite the gains resulting from reduced recycle delay, this methodology can lead to signal broadening, thereby reducing resolution. Furthermore, paramagnetic dopants complicate the dynamic studies of these biologically relevant materials. Consequently, there is a strong need to develop new NMR acquisition strategies in solid-state NMR that enhance sensitivity and reduce measurement times without compromising spectral quality or sample integrity.

Here, we demonstrate that methodologies employed in solution-state NMR, such as Band-selective Excitation Short-Transient (BEST) and band-selective Optimized Flip-Angle Short-Transient (SOFAST) approaches,[2,3] can also be applied to solid-state NMR under ultra-fast MAS conditions (150 kHz). These techniques utilize selective excitation to accelerate longitudinal relaxation, significantly reduce recycle delays, yielding substantial gains in SNR per unit time. Applied to crystalline GB1 and a GB1-IgG complex, these experiments provided improved time efficiency compared to conventional inverse cross-polarization (CP) sequences. Furthermore, band-selective excitation experiments show increased efficiency for aromatic protons compared to amide ones; notably, greater gains are observed for more complex systems, such as GB1-IgG, compared to a model crystalline GB1.
By integrating ultrafast MAS with solution-state-inspired pulse sequences and operating on microgram-scale samples, this approach offers a powerful approach for enhancing sensitivity in solid-state NMR. It enables detailed structural studies of complex biological systems in their native state, broadening the scope of what can be achieved with NMR in biomolecular research.

References:

[1]N.P. Wickramasinghe et al., Nat. Methods **6**, 215 (2009).
[2]P. Schanda and B. Brutscher, J. Am. Chem. Soc. **127**, 8014 (2005).
[3]P. Schanda, H. Van Melckebeke, and B. Brutscher, J. Am. Chem. Soc. **128**, 9042 (2006).

Paramagnetic NMR: from early ^{89}Y studies at Warwick to battery materials and DNP

Prof. Clare Grey

Department of Chemistry, Cambridge University, Cambridge, UK