Vol. 72, No. 4

ISSN 0432-7136

BULLETIN DU GROUPEMENT

d'informations mutuelles

Groupement
AMPERE

SE CONNAÎTRE, S'ENTENDRE, S'ENTRAIDER

October to December 2023

No. 293

Office: ETH Zürich, Institute of Molecular Physical Science
8093 Zürich, Switzerland, www.ampere-society.org

Contents

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Special Editorial

Groupement AMPERE

Dear Reader,

Twelve years ago, there was an AMPERE event in Beijing, the 11th International Conference on Magnetic Resonance Microscopy. Lizhi Xiao, the conference chair quoted Confucius, a famous Chinese philosopher, "How happy we are to have friends from afar!" Indeed, as scientists we are privileged to make friends and meet colleagues from all over the world and across political divides.
This year, wars are raging at several locations. Currently we witness about 10 armed conflicts globally. Most attention in the news focuses on the cruel wars in Ukraine and Gaza. None of these conflicts has emerged suddenly. All of them have a history. Many tensions build up slowly under the cover of misguidance, neglect, or greed.
As internationally connected scientists we are in a unique and special position. Sir Paul Callaghan expressed our role and responsibility as scientists exceptionally well with his famous words in the Great Hall of the People in Beijing, the assembly hall of the Chinese ruling bodies, on August 17th, 2011, 10 minutes after he was asked by Lizhi Xiao to address the attendees at the conference dinner: "You are in a field where you understand how science and technology benefits humanity. But there is something more that science calls from us. Carl Sagan once said, 'Science is our candle in the dark'. It is what has enabled humanity to struggle out of a dark world indeed. And we have values in science. Those values are called upon by the world with its enormous problems and by the countries from which we come."
Let us speak up judiciously where and whenever we can.

Bernhard Blümich
Past President of the Groupement AMPERE

(1) https://www.ampere-society.org/books/11-icmrm2011.pdf
pp. 43-44

Portrait:

Peter Crowley

Why magnetic resonance and why NMR and MRI?
NMR spectroscopy is great for studying how proteins recognize and bind other molecules. Arguably, NMR is the most powerful method, providing both structural and thermodynamic data on protein – ligand interactions. It is fascinating to combine a protein with a partner molecule in an NMR tube and investigate binding. Whatever those molecules might get up to can be revealed by NMR, in combination with X-ray crystallography. And doing such experiments is far more fun (and more real) than purely computational methods.

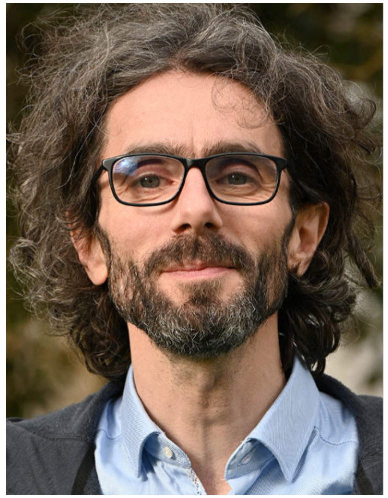
What is your favorite frequency?
I'd like to say 470.035 MHz because fluorine NMR is so simple. But in practice my favourite frequency is 600 MHz and the ^{1}H-^{15}N HSQC spectrum.

What do you still not understand?
This section would require several pages to do it justice. So, let's say no more.

Luckiest experiment you have ever done.
The luckiest experiment I have ever done was a simple and slightly counter-intuitive one. While studying the interactions of cationic cytochrome *c* with sulfonato-calix[8]arene, I observed that a 1:1 ratio resulted in almost complete loss of the ^{1}H-^{15}N HSQC spectrum. The lucky result was that titrating in excess calixarene restored the spectrum, albeit with significant chemical shift perturbations. Together with different crystal structures of cytochrome *c* - sulfonato-calix[8]arene assemblies, it was concluded that the macrocycle could switch on and off protein oligomerisation. If you would like to know more read about it here:
https://onlinelibrary.wiley.com/doi/full/10.1002/anie.201807490

What was the worst mistake you have made during your lab time?
The worst mistake I have made was trying to achieve perfection in an in-cell NMR project. Meanwhile, another laboratory published similar but better work. It took me too many years to realise that perfection is illusory.

Most memorable conference story.
This summer, I had the privilege of presenting at the Joint Conference on Calixarenes and Cucurbiturils in Tel Aviv-Yafo. It was a privilege both because of the conference and the location. I had the pleasure of meeting Julius Rebek who inspired me to write an essay on the origins of the *host* – *guest* terminology.
https://pubs.acs.org/doi/10.1021/acs.cgd.3c00985



With whom (historical person) would you like to meet?
Berzelius! One of the great chemists of the 19th century, he coined the word protein from *protos* – in recognition of their primary importance.

When do you get your best ideas?
I used to get my best ideas under conditions that I no longer enjoy. These days, they arrive sporadically, in the early hours as I emerge from sleep.

If you had just one month time for travelling - where would you go to?
One month of travelling would be great! It would definitely be a sea voyage, perhaps following the route George Orwell took to Myanmar. I would probably have to fly back.

Your idea of happiness.
Happiness is transient.

Position: Professor of protein chemistry, University of Galway
Homepage: www.universityofgalway.ie/science-engineering/staff-profiles/petercrowley/
Education: BSc Chemistry, University College Dublin, 1998
PhD Biophysics, Leiden University, 2002
Interests: Trees, Gardening, Food, Literature, Reggae

Call for nominations:

Raymond Andrew Prize 2024

In memory of Professor Dr. Raymond Andrew and to honor his pioneering work in the field of magnetic resonance, the AMPERE Group has founded the Raymond Andrew Prize. The prize is awarded to young scientists for an outstanding PhD thesis in magnetic resonance.

For the Raymond Andrew Prize 2024 the AMPERE Prize Committee is seeking your help in searching for qualified candidates who completed their dissertation during the period of 2022/2023. The prize will be presented during EUROMAR in Bilbao, Spain, June 30st to July 4th 2024.

You are kindly invited to submit nominations by e-mail to:
andrewprize@ampere-society.org

Nominations must be received by 15th February 2024 and should include the following documents:

- Nomination letter
- Curriculum vitae
- List of publications and presentations at conferences
- PhD thesis in PDF

The thesis should be written in English. In exceptional cases, the thesis may also be submitted in triplicate as a hardcopy to the AMPERE Secretariat. Submissions that arrive too late will automatically be transferred to the next year. The prize committee will reconsider excellent contributions for two years in a row.

For a list of past Andrew Prize winners see:
https://www.ampere-society.org/Awards.html

Report:

9th EFEPR Summer School on Fundamental theory and state-of-the-art applications of EPR Spectroscopy
Geneva, 3-9 September

Since the first School in 1999 in the Italian city of Caorle, the EFEPR Summer School earned recognition as a traditional core of European education in the field of EPR spectroscopy. Numerous research groups send their PhD and some Master students to the School to learn the state-of-the-art in the field. As for all social events, this tradition was forced to take a break during the Covid-19 pandemic. Following the wish of the European EPR community for a swift restart, the EFEPR decided in November 2022 in an online General Assembly to give it a go to organize the 9th EFEPR Summer School in Geneva, Switzerland, in September 2023, co-organized by Enrica Bordignon, University of Geneva, and Daniel Klose and Gunnar Jeschke, ETH Zurich, together with an international scientific board – backed up locally by a meticulously working blue-cladded team of brave helpers (known as "blue T-shirts").

As it is tradition on this school, the lecture program was dense, covering a wide range of EPR-topics and providing a formidable deep-dive from the basics of cw EPR, pulse EPR and quantum mechanics to current applications in Structural Biology and Catalysis, including the use of fast arbitrary waveform generators. Thus, the school catered to a wide range of interest groups and application fields from structural biology over coordination chemistry and catalysis to material science – and we thank all the 24 lecturers (42% female) for these excellent contributions!

The wide range and diversity are also reflected in the 112 participants (45% female), mostly PhD students from 21 countries on 4 continents, who showed their dedication in lively discussions at two evening poster sessions and in hands-on tutorial sessions.

These lab sessions, organized and scheduled according to each student's interests, included both real and virtual labs for the first time on our School – which allowed for more demonstrations via remote spectrometers for advanced tutorials such as DQC, ENDOR, HYSCORE, and RIDME.

A special lecture dedicated to the James Webb space telescope, given by a scientist from the "Observatoire de Genève", also met great interest, in particular because it covered the spectroscopy enabled by the telescope. After this evening lecture, participants enjoyed a traditional Swiss specialty: Chocolate fondue – to be added to the several kilos of chocolate distributed during the School! The dense scientific program was interrupted by the traditional relaxation day, which featured a boat tour across the idyllic Lake Geneva to the French village Yvoire. The sunny afternoon was rounded out by a picnic lunch, swimming, and a stroll through the charming medieval town.

On the last evening, we concluded our School with the poster awards for eight excellent young scientists, kindly sponsored by Groupement Ampere and the IES and the travel grant awards supported by Groupement Ampere. The last exchange of ideas was done via a lively forum, in which we addressed fundamental questions (what is SPIN?) and discussed the highlights of the School. We are happy that the participants embraced with great support the theme of sustainability which resonated with the ethos of this School (especially the vegetarian-only food, the absence of plastic bottles, the minimization of printed items, and the Swiss tap water).

Organizing such a lively event within such a short lead time would not have been possible without local support by the University of Geneva and donations from EPR-related and Swiss industries, including the Gold sponsors Bruker Biospin, Mettler+Toledo and Cryogenic.

We are already looking forward to the next EFEPR School 2025 kindly hosted at the University of Manchester, for updates see www.EFEPR.org.

Photos of the 9th EFEPR Summer School
The blue T-shirts: local organizing team.
The local organizers.
On the Boat towards Yvoire;
Four Ampère poster prizes:
Marvin Lenjer, Bartosz Mozgawa, Femke Reichardt, Maximilian Mayländer;
and four IES poster prizes:
Radhika Kataria, Julia Haak, Orit Nir-Arad, Sebastian Gorgon.
School group picture: 112 participants, 24 Lecturers and local helpers.

Poster Prize: EFEPR summer school

Marvin Lenjer

Exploring Spin Dynamics during Electron Spinlock Pulses

Marvin Lenjer[1,2*], Fabian Hecker[1], Nino Wili[3], Marina Bennati[1,2]

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[2]Department of Chemistry, Georg-August-University Göttingen, Göttingen, Germany
[3]Interdisciplinary Nanoscience Center, Aarhus University, Aarhus, Denmark
*marvin.lenjer@mpinat.mpg.de

Long microwave (MW) pulses are part of numerous pulsed EPR experiments including ELDOR detected NMR and cross-polarization ENDOR. Moreover, they can be used to lock transverse magnetization along the MW magnetic field in what is called a spinlock pulse. Under these conditions, spins are in the dressed state [1], which has possible advantages such as slower transverse relaxation.

However, understanding the spin dynamics during spinlocking is non-trivial because relaxation under MW irradiation plays a crucial, yet not fully understood role. Here, we use a commercial Bruker E680 W-band (94 GHz) EPR-spectrometer in combination with a SpinJet arbitrary waveform generator to analyse the effect of long MW pulses and the behaviour of dressed spins.

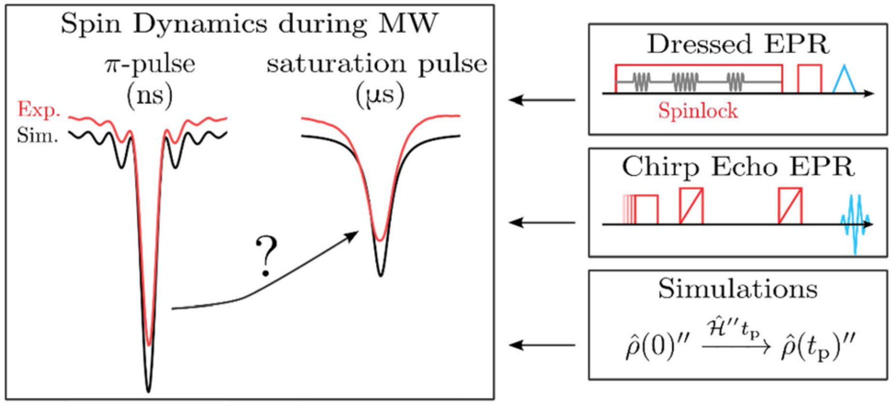

Figure 1: Methods for the analysis of spin dynamics during MW irradiation in order to understand the evolution of dressed spins.

Chirp echo EPR spectroscopy (CHEESY) allows us to analyse inversion and excitation

profiles of spinlock pulses [2] and ultimately, to observe the transition from a pulse with well-defined rotation properties to a spinlock or high turning angle pulse. Using phase modulation schemes, dressed state pulse sequences like dressed spin echoes are implemented [3]. The results suggest that spinlock under high-field EPR conditions achieves selective locking of resonant spins and show an increase of transverse relaxation time in the dressed state. Furthermore, we demonstrate the interchangeability of dressed and free state pulse sequence elements using modified dressed experiments (fig. c).

References:
[1] G. Jeschke, Chemical Physics Letters 1999, 301, 524-530
[2] N. Wili et al., Journal of Magnetic Resonance 2018, 289, 26-34
[3] N. Wili et al., Magnetic Resonance 2020, 1, 75-87

Poster Prize: EFEPR summer school

Maximilian Maylaender

Spin communication between light-induced triplet state and stable radical in different chromophore–radical systems

M. Mayländer[1*], T. Quintes[1], K. Kopp[2], O. Nolden[3], P. Gilch[3], O. Schiemann[2], A. Vargas Jentzsch[4], S. Richert[1]

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[2]Institute of Physical and Theoretical Chemistry, University of Bonn, Bonn, Germany
[3]Institute of Physical Chemistry II, Heinrich Heine University Düsseldorf, Düsseldorf, Germany
[4]SAMS Research Group, Université de Strasbourg, CNRS, Institut Charles Sadron, Strasbourg, France
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To identify novel materials for applications in molecular spintronics, a deeper understanding of the spin-spin interactions and their manipulation is crucial. To achieve this, we investigate molecular triplet-doublet systems which provide the advantage of systematic structural modifications [1,2]. They typically consist of a chromophore covalently linked to a radical. In such systems, the triplet state of the chromophore is accessible by light excitation via enhanced intersystem crossing (EISC). Following triplet formation, the magnitude of the exchange interaction between the two spin centres dictates the mechanism by which they interact [3]

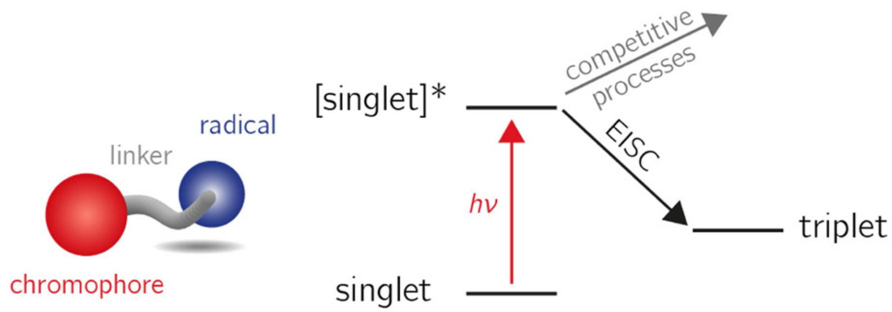

Figure 1: Through light excitation of the chromophore the excited singlet state is formed. Subsequently, the triplet state can be generated by enhanced intersystem crossing (EISC).

We use optical spectroscopic techniques, such as femtosecond UV-vis transient absorption spectroscopy to observe the differences in the ultrafast photochemical behaviour. To study the magnetic interactions between the two spin centres, we use transient EPR techniques. This work compares various chromophores, such as perylene diimides (PDIs) and boron-dipyrromethene (BODIPYs), attached via different linkers to nitroxide or trityl radicals with respect to the kinetics of the different photochemical processes, triplet formation yields and magnetic properties. In molecular systems with trityl as the stable radical, energy transfer between the excited chromophore and the radical limits the triplet yield. The length of the linker is shown to influence the rate and yield of the processes observed following light excitation as well as the nature of the interaction. These observations finally lead us to propose design guidelines for molecular systems comprising chromophores in which higher excited states can be efficiently sensitised in proximity of an unpaired electron spin.

References:
[1] S. Sanvito Chem. Soc. Rev. 2011, 40, 3336–3355.
[2] T. Quintes, M. Mayländer, S. Richert Nat. Rev. Chem. 2023, 7, 75–90.
[3] M. Mayländer, S. Chen, E. R. Lorenzo, M. R. Wasielewski, S. Richert J. Am. Chem. Soc. 2021, 143, 7050–7058.

Poster Prize: EFEPR summer school

Bartosz Mozgawa

EPR study and molecular modelling of transition metal active centres in oxides and zeolites – applications in catalysis

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Transition-metal-ion (TMI) exchanged zeolites and transition-metal oxides are among the most prominent catalysts in recent decades. Because of the paramagnetic nature of the active centres of both types of catalysts, EPR spectroscopy has been widely applied in studying their properties, including catalytic activity. This study aims to gain insight into the structural and electronic properties of TMI active sites and their role in selective catalytic reduction of NO_x using ammonia – NH_3-SCR. CW-EPR spectra of copper exchanged zeolites in different environments (H_2O, NH_3), were analysed to obtain information about the local coordination of the catalytic sites (Fig. 1a). Furthermore, molecular modelling has been performed using DFT calculations with VASP and ORCA to substantiate the analysis. From the latter, the characteristic values of the *g* and *A* tensors have been obtained and used to assign to specific locations of the active centres and structures of paramagnetic adducts (Fig. 1b).

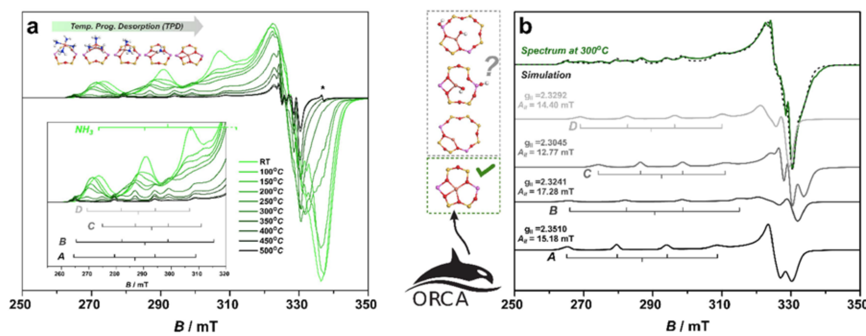

Figure 1: EPR spectra during temperature programed desorption of ammonia from Cu-SSZ-13 (a) and simulation deconvolution of selected spectrum, and assignment based on ORCA calculations (b).

The catalytic activity of the materials obtained was tested in heterogeneous reaction of the gas phase in NO_x SCR using ammonia over copper exchanged zeolites. In absence of oxygen NO is reduced by ammonia, while simultaneously depositing protons regenerating Brønsted acid sites, leading to reduction of copper, and thus a decrease in EPR signal (Fig. 2a). Remaining unreduced Cu^{2+} acts as reservoir for ammonia, which can be confirmed using isotopically labelled $^{15}NH_3$ (Fig. 2b).

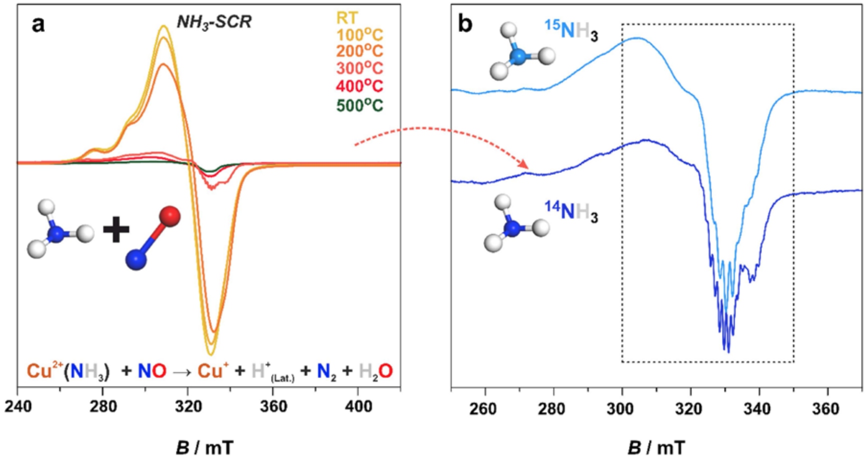

Figure 2: Reduction half cycle (RHC) of NH3-SCR of NOx (a) and isotopic labelling of residual ammonia-complexes (b).

Acknowledgements:

This work was financially supported by the National Science Centre Poland, grant Sonata Bis7 2017/26/E/ST4/00794 and National Centre for Research and Development in Poland, PNOX No. WPC1/PNOX/2019.

Poster Prize: EFEPR summer school

Fehmke Reichardt

Hyperfine spectroscopy to study radical transfer in E. Coli ribonucleotide reductase across the α/β interface

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[2]Department of Chemistry, Georg-August-University Göttingen, Tammannstr. 2, 37077 Göttingen, Germany.
*fehmke.reichardt@mpinat.mpg.de

E. coli ribonucleotide reductase (RNR) is a homodimeric enzyme ($\alpha_2\beta_2$) converting ribonucleotides to deoxyribonucleotides. The reaction is initiated by a long-range proton coupled electron transfer (PCET) from β to the active site in α (Figure 1A).[1] A yet unresolved step is the PCET across the α/β interface.
^{19}F electron–nuclear double resonance (ENDOR) studies by MEYER ET AL. using 3,5 F_2Y_{731}-α_2 revealed a flipped Y_{731}-conformation with an O–O distance of ~3 Å, suggesting a colinear PCET.[2] Alternatively, water-mediated PCET has been discussed, supported by the detection of 1st coordination sphere waters in ^{17}O ENDOR studies by HECKER ET AL..[3]

Despite the spectroscopic data provided, both hypotheses have distinct problems. While the use of fluorinated Y_{731} is a strong interference with the natural system, a water-mediated PCET requires a 2nd coordination sphere water to bridge the distance between the tyrosines (Figure 1B). Here, we present different approaches to pursue both theories. Labelling of α_2 mutants with ^{17}O tyrosine, for example, allows for detection of conformational changes via hyperfine spectroscopy with minimal perturbation of the natural system. Furthermore, we are exploring the upper distance limit of ^{17}O ENDOR with model systems (Figure 1C) to enable detection of 2nd coordination sphere water.

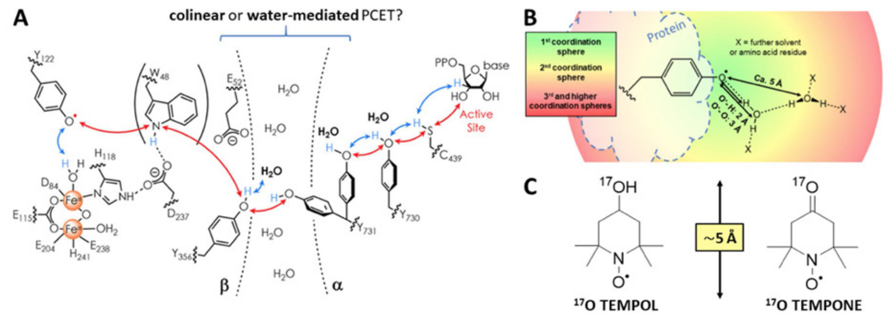


Figure 1: A: Proposed PCET in RNR. B: Illustration of coordination spheres. C: 17O model systems.

References:
[1] B. L. Greene et al., Annu Rev Biochem. 2020, 89, 45-75
[2] A. Meyer et al., J. Am. Chem. Soc. 2022, 144, 25, 11270–11282
[3] F. Hecker et al., J. Am. Chem. Soc. 2021, 143, 19, 7237–7241

Report:

17th ICMRM
Singapore, 27-31 August

The 17th ICMRM has been held from 27-31 August 2023 in Singapore.
The aim of the ICMRM is to promote recent advancements in high-resolution, spatially resolved magnetic resonance methods and applications. The first ICMRM was held in 1991 in Heidelberg and was originally known as the „Heidelberg Conference". Currently, ICMRM is organized biannually by the Division of Spatially Resolved Magnetic Resonance of the AMPERE Society.

Topics of interest included
- Research related to the application of spatially resolved magnetic resonance to a large variety of systems including solids, porous media, and biological tissues.
- Applications of magnetic resonance to engineering, biomedical and clinical sciences
- Molecular and cellular imaging
- Low field and mobile NMR
- Technological advances in magnetic resonance instruments
- Other exotic experiments

Awards
1. Erwin Hahn award recipient 2023: Henk Van As
2. Paul Callaghan Young Investigator Award went to Johanna Günther (University of Würzburg) for her presentation on ‚Chebyshev nodes-based fingerprinting of magnetic nanoparticles'
3. Poster competition Winner: Emanuel Bertizzolo (The University of Western Australia) ‚Low field NMR as an optimization tool for mechanical dewatering of anaerobic digestate'
4. Image Beauty Competition Winner: Maxime Yon (Lund University) ‚Double-rotation gradient waveforms and nonparametric distributions of frequency-dependent diffusion tensors'

Talks & Poster Presentations
1 Plenary: William (Bill) S. Price, Western Sydner University
2. Invited Talks:
- Le Roy Chong, Changi General Hospital, Singapore
- Daniel Clarke, Victoria University of Wellington
- Yang Xia, Oakland University
- Volker Behr, University of Wurzburg
- Ye Li, Shenzhen Institute of Advanced Technology, Chinese Academy of Sciences
- Maxim Zaitsev, University of Freiburg

- Thu Thao Le, National Heart Research Institute Singapore
- Konstantin Momot, Queensland University of Technology
- Pablo Prado, Livivos, Inc. 3. Educational Talks, 4
- Meghan Halse, University of York
- Michael Johns, University of Western Australia
- Hilary Fabich, ABQMR, USA
- Luisa Ciobanu, Neurophysics team at CEA/Neurospin
4. Orals: 43
5. Posters: 21

Statistics
Attendees: 96, 15 student helpers, 28% students, 22% female

Beauty Image Prize: ICMRM Singapore

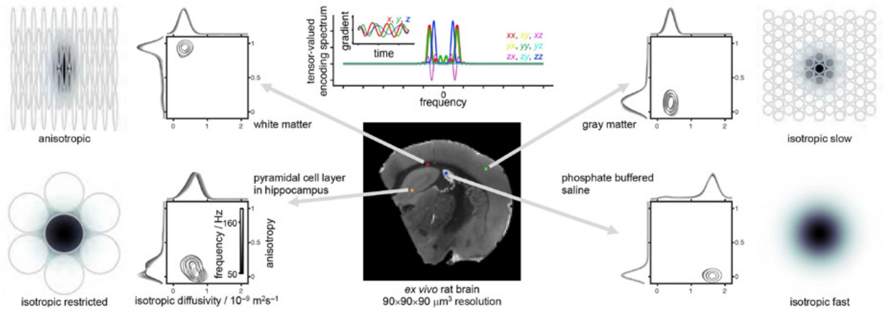
Maxime Yon

Double-rotation gradient waveforms and nonparametric distributions of frequency-dependent diffusion tensors

Omar Narvaez[1], Maxime Yon[1,2], Hong Jiang[2], Alejandra Sierra[1], D. Topgaard[2]

[1] A.I. Virtanen Institute for Molecular Sciences, University of Eastern Finland, Kuopio, Finland ,
[2] Department of Chemistry, Lund University, Lund, Sweden

MRI is the method of choice for noninvasive studies of micrometer-scale structures in biological tissues via their effects on the time/frequency-dependent ("restricted) and anisotropic self-diffusion of water. Traditional diffusion MRI relies on pulsed magnetic field gradients to encode the signal with information about translational motion in the direction of the gradient, which convolves fundamentally different aspects—such as bulk diffusivity, restriction, anisotropy, and flow—into a single effective observable lacking specificity to distinguish between biologically plausible microstructural scenarios [1]. To overcome this limitation, we introduce a formal analogy between measuring rotational correlation functions and interaction tensor anisotropies in NMR spectroscopy and investigating translational motion in MRI [2], which we utilize to convert data acquisition and analysis strategies from NMR of rotational dynamics in macromolecules [3] to MRI of diffusion in biological tissues, yielding model-independent quantitative metrics reporting on relevant microstructural properties with unprecedented specificity. In particular, we use “double-rotation” gradient waveforms [4] to explore both the frequency and anisotropy dimensions of the tensor-valued encoding spectrum (see Fig. 1) in addition to the *b*-value and direction of conventional diffusion tensor imaging, Monte Carlo data inversion [5] to convert the detected signal into nonparametric distributions of frequency-dependent diffusion tensors, and insights from “dynamics detectors” [6] to convert the noise-sensitive primary distributions into more robust projections and quantitative



metrics reporting on diffusivity, anisotropy,2 orientation, and restriction. The approach is demonstrated on 1 liquid crystals, yeast cells, and *ex vivo* rat brain as shown in Fig. 1.

Fig. 1. Diffusion encoding by double-rotation gradient waveform (top center) and frequency-dependent diffusion tensor distributions for selected voxels of an ex vivo rat brain. The multidimensional distributions are visualized as projections onto the 2D plane of isotropic diffusivity and normalized anisotropy for a series of frequencies (gray scale of contour lines), where restriction results in a shift of the distribution with frequency (bottom left). For each voxel, the underlying tissue structure is shown schematically with gray lines representing partially permeable cell membranes and “ink stains” illustrating water diffusion during the 10 ms timescale of diffusion encoding.

References:
[1] Topgaard, J. Magn. Reson. 306, 150 (2019). doi: 10.1016/j.jmr.2019.07.024;
[2]Narvaez, arXiv:2111.07827 (2021). doi: 10.48550/arXiv.2111.07827;
[3] Lipari, J. Am. Chem. Soc.104, 4546 (1982). doi: 10.1021/ja00381a009;
[4] Jiang, Magn. Reson. 4, 73 (2023). doi:10.5194/mr-4-73-2023;
[5] Prange, J. Magn. Reson. 196, 54 (2009). doi: 10.1016/j.jmr.2008.10.008
[6] Smith, Angew. Chem. Int. Ed. Engl. 56, 13590 (2017). doi: 10.1002/anie.201707316.

Poster Prize: ICMRM Singapore

Emanuel G. Bertizzolo

Low field NMR as an optimization tool for mechanical dewatering of anaerobic digestate

E. G. Bertizzolo[1,2], F. Tessele[2], M. L. Johns[1], E. O. Fridjonsson[1]

[1]The University of Western Australia, Perth, Australia
[2]Tessele Consultants, Perth, Australia

Introduction:
The circular economy plays a critical role in fulfilling sustainability demands worldwide [1]. In solids management, anaerobic digestion is being explored as it turns waste into valuable products. Its by-product, known as anaerobic digestate, is a high-potential bio-fertilizer [2]. However, a dewatering step including flocculation and mechanical separation is usually required to reduce volume and increase the efficiency of subsequent processing steps. In this work low field NMR was used as a technique to obtain information non-invasively from this dewatering step.

Methods:
Triplicates of anaerobic digestate from the red meat processing industry were flocculated using EM640CT (0.41% active content) from 0 to 5.25% v/v and then mechanically dewatered using IEA Cylinder Press “Romulus”. Samples were then analyzed via T_2 CPMG performed in an Oxford GeoSpec 12.7 MHz (Oxford Instruments) acquiring 25,000 echoes with an echo time of 300 μs and 4 scans on the flocculated solution and 10 scans on solids (also called cake) after mechanical dewatering and the acquired signal was regularized to obtain T2 relaxation time distributions [3].

Results and discussion:
Fig. 1 compile the main findings in this work so far. The overall visual assessment showed an increase in cakes’ physical properties with addition of flocculant and when comparing T2 distributions of sample 1 and 8 (0 and 5.25 % v/v respectively), the two longest populations shift to longer relaxation times (P1: 0.964 to 1.63 seconds, P2: 0.196 to 0.255 seconds) and an inversion in terms of signal fraction occurs (P1: 0.020 to 0.560, P2: 0.740 to 0.095). P3 relaxation time, on the other hand, hovers around 0.047+0.004 seconds and signal fraction 0.322+0.048. Given that lower T_2 relaxation water populations should correspond with proximity to solids, the results follow the models of different water populations in flocculated systems published in the literature [4]. With that in mind, when comparing T_2 distributions from the flocculated sample and the produced cake, the peak corresponding to the water close to the solids, P3, also kept stable throughout the samples with the exception when desired cakes were produced, which a sharp shift to shorter T_2 occurs (from 0.047 to 0.019 seconds for sample 8).

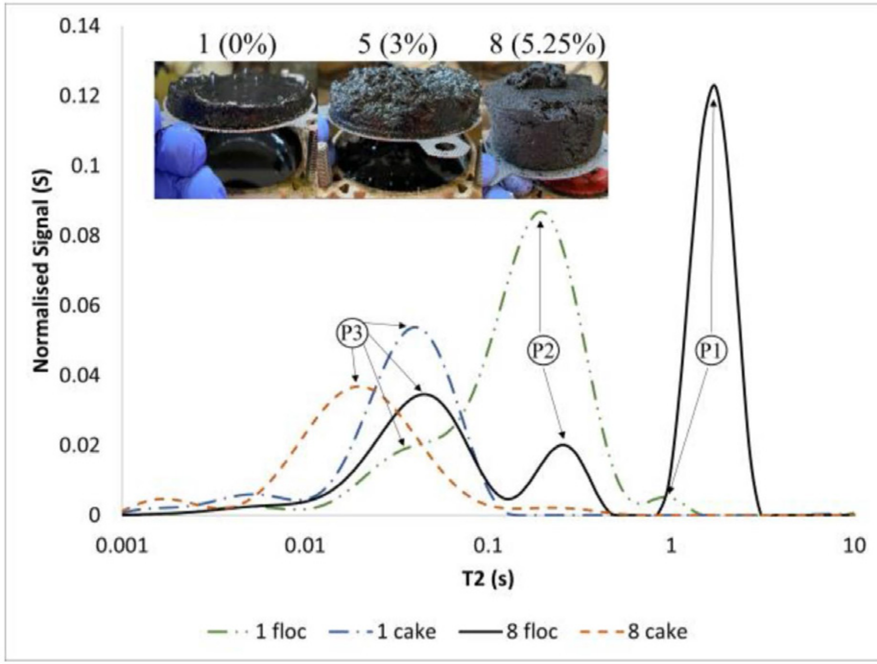


Fig. 1: Visual evidence of cakes after mechanical dewatering (inserted) and T2 distribution of both flocculated solution and resulted cake from sample 1 and 8.

Conclusion:
Low field T_2 relaxometry showed good performance tracking the dewatering process by providing quantitative information allowing identification of structural features on flocculation step which correlates with the resulted cake. Further studies are needed to establish predictors of a desired resulted cake after mechanical dewatering based on the outcomes from flocculation.

References:
[1] Tessele, Water e-Journal (2020). [2] Lukehurst, IEA Bioenergy Task 37 (2010). [3] Bristow, Water Research (2021). [4] Vesilind, Water Environment Research (1994).

From the AMPERE Café

Panel discussion “Applications of magnetic resonance: what we can and cannot do”

On November 23rd, we organized a panel discussion about the application of magnetic resonance spectroscopy in biology and materials science. Three seasoned experts shared their perspectives.
Anja Böckmann, Université de Lyon - bio solid-state NMR
Aaron Rossini, Iowa State University - materials solid-state NMR (with DNP)
Joern Werner, University of Southampton - bio liquid-state NMR

Some highlights from the conversation

- Interactions at conferences or a chat with the seminar speaker from outside the field of magnetic resonance are critical for selecting new and exciting chemical or biological questions.
- Remember the 3F rule (fun, feasible, fundable) when initiating a research project.
- In liquid-state NMR, sequences that were difficult to set up back in the day, are now used routinely. Solid-state NMR is developing along a similar path.
- Transient states and dynamics are the nature of biology. NMR is the most effective method to investigate them.
- For disordered materials and surfaces, NMR is the method of choice to investigate the local chemical environment.
- (MAS) NMR can locally probe if an Alphafold model is correct without first solving the entire structure.

Magnetic resonance pub quiz

On October 22nd, we hosted, for the second time, the magnetic resonance pub quiz. Ten teams competed. In the finale, Team *nmrlab@tifrh* (consisting of researchers from the TIFR Hyderabad) managed to beat Team *Hydrogen* (a team formed for the occasion with researchers from the University of Turin, NTU Singapore, Claude Bernard University Lyon 1, and Karlsruhe Institute of Technology). Congratulations, Team nmrlab@tifrh! The prize, a selection of fine Swiss chocolates, has been shipped.

To give you a flavor of the quiz, we share here a selection of 25 questions (and answers). Have fun!

Questions

1: Which famous physicist did not derive the “golden rule” that bears his name? Who derived it instead?

2: Charles Slichter writes in his text book that “The magical solution was found by the Wizard of Resonance, Erwin Hahn, and demonstrated by the Wizard and his Sorcerer’s Apprentice, Sven Hartmann.” He then cites a paper from 1962. What is he referring to?

3: Which stable isotope has nuclear spin 3?

4: Which Dutch physicist became famous for *almost* being the first to observe nuclear magnetic resonance?

5: In NMR and MRI, shimming is used to reduce inhomogeneities of the magnetic field across a sample volume. The process typically involves the careful adjustment of currents through a set of strategically placed coils. What is the origin of the word “shimming”?

6: What is the most cited paper in the Journal of Magnetic Resonance?

7: Which poisonous substance featured as a murder weapon in Agatha Christie’s novels and as a common test sample in the early days of two-dimensional liquid-state NMR?

8: What did George Feher, the person who invented ENDOR, do to supplement his research funding, apart from applying for government research grants?

9: What was Endor in the Star Wars movies?

10: Which numerical factor did Goudsmit and Uhlenbeck miss in their paper proposing the concept of spin? Who found it later?

11: What was John von Neumann notoriously bad at?

12: What is the electron spin Hilbert space dimension of two Gd^{3+} ions?

13: What is the strength of the Earth’s magnetic field in Berlin?

14: Quadrupolar overtone MAS NMR spectra can look different between Bruker and Varian spectrometers. Why?

15: Cambridge Isotope Laboratories separates ^{13}C-carbon monoxide from ^{12}C-carbon monoxide using large underground towers at a site in Xenia, OH. Of which distinct properties of these isotopomers do they make use in the process?

16: What is the origin of most helium on earth?

17: Which body part provided the very first human in-vivo MRI image?

18: In 1975, Jake Schaefer and coworkers reported the dipolar-decoupled ^{1}H-^{13}C cross polarization magic-angle spinning NMR spectrum of ivory, which they had machined into a rotor. Which particular circumstances led Jake Schaefer to choose ivory as a test sample?

19: How old were Uhlenbeck and Goudsmit when they introduced electron spin?

20: Nuclear spin isn't actually spin. What is it?

21: In 1896, the Dutch physicist Pieter Zeeman discovered what we now call the Zeeman effect. What did he observe in his first successful experiments in August/September of that year?

22: The Francis Bitter Magnet Laboratory is located in Cambridge, Massachusetts. It was established in 1960 and has since become famous for research in solid-state physics, magnet technology, nuclear fusion, NMR, and DNP. What was the purpose of the building before it became the Francis Bitter Magnet Laboratory?

23: Albert Overhauser first presented what we now know as the Overhauser effect at the APS meeting in April 1953. Which of the following Nobel laureates expressed skepticism? (a) Felix Bloch, (b) Edward Purcell, (c) Isidor Rabi, (d) Norman Ramsey.

24: What is the motto of the AMPERE society?

25: What is the name of the movie in which Minnie Driver tells Matt Damon that she can't go on a date with him, because she has to assign the proton spectrum of ibogamine?

Answers

1: **Enrico Fermi; Paul Dirac**
Dirac, P. A. M. *The Quantum Theory of Emission and Absorption of Radiation*, Proceedings of the Royal Society A. 114:767 (1927)

2: The **Hartmann-Hahn condition** for rapid cross-relaxation in the doubly rotating frame: $|\gamma_{\mathrm{I}} B_{\mathrm{eff,I}}| = |\gamma_{\mathrm{S}} B_{\mathrm{eff,S}}|$, or, on resonance, $|\gamma_{\mathrm{I}} B_{1,\mathrm{I}}| = |\gamma_{\mathrm{S}} B_{1,\mathrm{S}}|$

Hartmann, S. R. and Hahn, E. L. *Nuclear Double Resonance in the Rotating Frame*, Phys. Rev. 128:5 (1962)

3: ^{10}**B** (natural abundance about 20 %, γ = 4.58 MHz/T)

4: In 1936, **Cornelis Gorter** reported an unsuccessful attempt to observe nuclear magnetic resonance using a calorimetric method. He did not observe the expected increase in temperature upon irradiation of a magnetic dipole transition, because, in hindsight, the T_1 relaxation of the sample he had chosen was very long and he had saturated the transition. See J. H. van der Waals *Gorter's footprints on the trail that led to magnetic resonance*, Encyclopedia of Nuclear Magnetic Resonance, Vol. 1, p. 677 (1996)

5: Shimming received its name from **mechanical shims (thin plates of steel)** which are used to adjust position and coplanarity of the pole faces of an electromagnet. Improving field homogeneity with the help of steel plates is also referred to as passive shimming, as opposed to active shimming with coils with adjustable current.

6: *MLEV-17-based two-dimensional homonuclear magnetization transfer spectroscopy*, **Bax and Davis, 1985**. According to Web of Science it has 4490 citations (22 Sep 2022), Scopus 4353 (2 Oct 2022). The MLEV-17 mixing scheme is a modification of MLEV-16 composite pulse decoupling cycle. It is less sensitive to pulse imperfections and provides net magnetization transfer over a substantial bandwidth with only limited RF power. The apparent decay constant of spin-locked magnetization can be prolonged by up to a factor of two (compared to $T_{1\rho}$) by using this new type of mixing scheme.

7: **Strychnine** is a highly toxic, colorless, bitter, crystalline alkaloid used as a pesticide, particularly for killing small vertebrates such as birds and rodents. Characters poisoned with strychnine: Mr. Appleton in *The Coming of Mr Quin*, Miss Amelia Barrowby in *How Does Your Garden Grow?*, Lady Ariadne Grayle in *Death on the Nile* (short story), and Emily Inglethorp in *The Mysterious Affair at Styles.*

8: George Feher **played in poker tournaments in Las Vegas**. In an EPR Newsletter from 2003 (vol. 13, no 1-2), he speaks open-heartedly about his passion for the game. Feher, G. *Observation of Nuclear Magnetic Resonances via the Electron Spin Resonance Line*, Phys. Rev. 103:3 (1956).

9: Endor is a **moon** in the Star Wars universe. Endor is known for its endless forests and is the home world of the Ewok. It is the site of a pivotal battle in Return of the Jedi. Endor orbits a gas giant, which orbits two suns, Endor Prime I and Endor Prime II. Darth Vader's funeral was held on Endor.

10: A factor of **1/2**. It was found by **Llewellyn Thomas** (and now known as the Thomas Half) when he relativistically recalculated the precessional frequency of the doublet separation in the fine structure of the atom. Thomas, L. *The Motion of the Spinning Electron*, Nature 117 (1926).

11: **Driving.**

12: Gd^{3+} ions have $S = {}^{7}/_{2}$. The Hilbert space for the spin system consisting of two Gd^{3+} ions has dimension $(2S + 1)(2S + 1) =$ **64**.

13: In 2015, in Berlin: 49.5 µT –> **about 50 µT**. The exact strength of the Earth's magnetic field varies depending on geographic location and with time. Worldwide it ranges between 25 and 65 µT (0.25 and 0.65 G).

14: The rotors are spun in opposite directions. In the *Spinach* library (https://spindynamics.org/), see `examples/nmr_overtone/mas_glycine_1.m`.
Try changing the sign of the spinning rate.

15: CIL enriches carbon through cryogenic distillation of carbon monoxide. ^{13}C-carbon monoxide has a **boiling point** that is 0.1 K above that of ^{12}C-carbon monoxide. The boiling point of carbon monoxide at atmospheric pressure is -191.5 °C or 81.7 K.

16: **Radioactive decay** in minerals of uranium and thorium. Emitted alpha particles (helium nuclei, He^{2+}) combine with electrons as soon as they are stopped by the rock. Because helium is trapped in the subsurface under conditions that also trap natural gas, the greatest natural concentrations of helium on the planet are found in natural gas, from which most commercial helium is extracted.

17: The **finger** of Andrew Maudsley, who was Peter Mansfield's student in 1976.

18: **His father and grandfather were both professional and legendary billiard players**. He used the ivory of a **billiard ball**.

19: George Uhlenbeck (06.12.1900-31.10.1988), Samuel Goudsmit (11.07.1902-04.12.1978). They published their short letter, which posits intrinsic angular momentum for the electron, in November 1925 in Naturwissenschaften. Thus, **Uhlenbeck was 24 and Goudsmit was 23 years old**.

20: The **total angular momentum of the ground state**. For ^{17}O, for example, this is mostly orbital angular momentum.

21: A piece of asbestos containing NaCl is brought into a hydrogen/oxygen flame. With the help of a grating, the spectral lines are separated. **When the magnetic field is switched on, a broadening of the spectral lines** (specifically the D lines) **is observed**, by a factor of 2 to 3.

22: A **bakery** of the Ward (or Continental) Baking Company. Founded in 1849 by James Ward and his son, the Ward Baking Company is known for Wonderbread and the Twinkies snack cakes.

23: all four (**a, b, c, and d**)

24: **Se Connaître, S'Entendre, S'Entraider**, translated from French to English: to get to know each other, to listen to each other, to aid one another.

25: **Good Will Hunting** (1997). Ibogamine is a central-nervous-system stimulant alkaloid found in Tabernanthe iboga and Crepe Jasmine (Tabernaemontana divaricata). Ibogamine has been shown to reduce the self-administration of cocaine and morphine in rats.

Acknowledgement
The set of questions for the quizzes of the past two years came together during intermittent brain storm sessions between Guinevere Mathies and Ilya Kuprov. Individual questions were suggested by Quentin Stern, Sanjay Vinod-Kumar, Michal Leskes, Xander Barnes, and Arno Kentgens.

Your score
0-5: Novice. You either have recently started to work in the field of magnetic resonance or are just not very savvy.
6-10: All right, you have answered some questions correctly. But you need to step up your game.
11-15: Not bad. It looks like you know a thing or two about magnetic resonance.
16-20: This is a good score!
21-25: Can I do a PhD project in your group?

Guinevere Mathies, November 2023

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