Vol. 68, No. 2 ISSN 0432-7136

BULLETIN DU GROUPEMENT

d'informations mutuelles

AMPERE

SE CONNAÎTRE, S'ENTENDRE, S'ENTRAIDER

March to June 2019

No. 275

Office: ETH Zürich, Laboratory of Physical Chemistry
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Contents

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Editorial

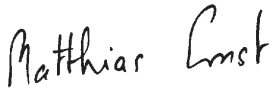
Dear members of the Groupement AMPERE,

this year's EUROMAR is approaching fast and many of you will be attending the conference in Berlin, Germany which is held together with ISMAR and the Fachgruppe Magnetische Resonanz of the German Chemical Society. We have a record attendance with well over 1000 people and a program with almost 200 talks in five parallel sessions and more than 500 posters. I am sure you will find many interesting contributions in your field of research and enjoy the possibility to meet colleagues and meet new people. Since all the participants become automatically members of the Groupement AMPERE, I would like to urge everybody to attend the General Assembly of the Groupement AMPERE on Tuesday at 6:30pm directly following the final plenary talk. Our society needs a broad basis and support to evolve and start new projects.

Besides the big annual EUROMAR conference, the smaller schools and conferences are at least as important. In this issue, you can read about the 16th International School Conference ‚Magnetic Resonance and it's Applications' Spinus 2019 (page 5) and about the AMPERE NMR school in Zakopane, Poland (page 16) which is organized every year since 1993 first by Jerzy Blicharski and now since many years by Stefan Jurga. It is a lot of work to set up the program and organize such an event every year. Thank you very much for investing so much into the education of students and the future of NMR.

Enjoy the summer time and I hope to see many of you at the EUROMAR in Berlin and especially at the AMPERE General Assembly on Tuesday evening.

Matthias Ernst
Secretary General Groupement AMPERE



Portrait: Prof. Sabine van Doorslaer

• why magnetic resonance and why EPR?
This is pure chance. Just like many other scientists, I ended up in this particular field out of a combination of inspirational teachers, ad hoc research possibilities and practical considerations. There were many moments in my education and academic career when I could have taken another direction. I am convinced they would have led me to equally fulfilling fields in science. However, having said this, I am very happy that I went for magnetic resonance, since it is a fascinating route and allows you to engage in many diverse interdisciplinary topics.

• what is your favorite frequency?
Q-band (35 GHz), since it combines the advantages of the lower and higher frequencies. Unfortunately, we do not have the research funding at the moment to buy such a spectrometer, and for that reason, my next best favorite remains X-band (9.5 GHz).

• what do you still not understand?
A better question would be: what do I understand? Every day I learn something new and find new questions.

• luckiest experiment you have ever done.
I was lucky in many experiments. What for instance looks in a paper like a deliberate annealing experiment, often just starts out by an instable temperature regulation during an experiment after which an unexpected effect is seen that is then found to be repeatable. Science would not be possible without the interplay between the occasional good luck (that puts you on a pink cloud for a while) and bad luck (that makes you curse, sweat and rethink matters).

• what was the worst mistake you have made during your lab time?
I made (and make) a lot of stupid mistakes, including forgetting to put the TWT on operate when setting up an overnight experiment, forgetting to properly store the raw data of a spectrum that I had obtained after long trying and accumulation, forgetting to switch back cables between experiments, forgetting to put a critical sample back in the fridge after an experiment, etc. The good thing is, you learn from every mistake you make, and it helps to prevent, recognize and quickly solve similar mistakes of your students.

• most memorable conference story
I have fond memories of many conferences, but one that I will always remember is the 4th EMARDIS conference in Sofia in 1995. I was in my last year of my PhD back then and, by chance, ended up sharing a room with Daniella Goldfarb (then already a professor). The bathroom had only occasionally running water which required some problem solving, which, in its own right, was already enough to make it a memorable event. However, the conference was also the one that started life-long friendships with Daniella, Gunnar Jeschke and Elio Giamello. And, I met many of the epic names in the field of EPR, like Yakov Lebedev and John Pilbrow. Due to the isolated and confined setting of the meeting, I could talk and interact with everyone, something I would never have dared at a big conference. It opened enormously my scientific horizon. I feel the same open atmosphere at our EFEPR schools, one of the reasons why these schools are so important.

• with whom (historical person) would you like to meet?
That is a very difficult question, since I can think of many fascinating people that I would have liked to meet, but let me name two extraordinary women: Hannah Arendt and Käte Kollwitz. Although Hannah was 39 years younger than Käte, they both lived throughout the two World Wars and the interbellum, and these turbulent times have very much influenced their work. Käte's art deals in an unsurpassed way with the theme of death. She was a very thoughtful person, who was not afraid to take a clear public stand, as she proved in the thirties when signing, alongside men like Einstein and Heinrich Mann, the urgent call for unity on the left side in order to defeat the extreme right in the elections. Hannah Arendt, in turn, has left us an incredible original philosophical work. I would like to meet them in Kaliningrad (the former Prussian city Königsberg) where both of them grew up, and hear their views on current European politics.

• when do you get your best ideas?
I always get my best ideas when talking to other people, even if the conversation is actually about something totally unrelated. In general, science is all about communication with peers. I am convinced that an attitude where we go more for competition than for collaboration is inevitably killing science.

• if you had just one month time for travelling - where would you go to?
I would travel in summer from Malmö to Luleå and take many (e-)books with me for the long evenings.

• your idea of happiness.
Our yearly street BBQ, when we put a long row of tables out on the street, lit candles, have a nice dinner with all our neighbors, talk through the summer night about our very different lives and watch the children grow up (and us get old).

Position: Full Professor, Biophysics and Biomedical Physics, University of Antwerp, Belgium

Awards:
Prize of "Jong KVCV" (KVCV = Royal Flemish Chemical Society) for the most excellent student in Chemistry at the University of Ghent (1991), Teaching award of the Faculty of Science, University of Antwerp (2014), Bruker Prize by the ESR group of the Royal Society of Chemistry (UK) (2018)

Homepage:
https://www.uantwerpen.be/nl/personeel/sabine-vandoorslaer/

Education:
BS+MS in Chemistry (1987-1991) - Ghent University, Belgium
BS+MS in Physics (1988-1992) Ghent University, Belgium
Ph.D. in Physics, Ghent University, Belgium (1992-1996)

Interests:
literature, history (especially history of the 19th-20th century), science philosophy and ethics, reading in general, having fun with family and friends, and of course science, more science and even more science.

Report: 16th International School-Conference ‚Magnetic Resonance and it's Applications' Spinus 2019

http://spinus.spb.ru

Since 2004 the Saint-Petersburg State University (SPSU) holds a series of the annual International School-Conference «Magnetic Resonance and its Applications. Spinus» (an AMPERE event since 2016). The 16th Meeting "Spinus-2019" was opened on Monday, April 1, and was closed on Friday, April 5, 2019. Venue: the hotel „Baltiets" in Repino on the beach of the Gulf of Finland, 30 km north-west from the center of Saint Petersburg (http://baltiets.ru/).

The goal of the School-Conference is to provide a platform to young scientists and students for the use of all aspects of magnetic resonance methods and techniques as well as computational and theoretical approaches for the solving of fundamental and applied problems in physics, chemistry, medicine and biology.
The scope of the Conference includes the following topics:
- Modern trends in NMR, ESR and NQR
- Magnetic resonance for fundamental science
- Magnetic resonance imaging
- Computer Modeling
- Earth's field NMR
- Magnetic resonance in industry
- Related areas

The official language of the "Spinus 2019" is English. Extended abstracts were published (see http://spinus.spb.ru/). Selected papers of participants will be published in a special issue of "Applied Magnetic Resonance".
Scientific adviser of the School-Conference "Spinus" is Professor Vladimir Chizhik (SPSU), Chairman of Organizing committee is Dr. Sci. Denis Markelov (SPSU).

The Meeting was attended by 154 participants (95 young participants: students of undergraduate, graduate, specialty and postgraduate studies; young scientists under 35 years old) from 16 countries (China, Cuba, Czech Republic, Egypt, England, Estonia, Finland, France, Germany, Italy, Sweden, Turkey, USA, Russia, Slovakia, Serbia).

The Meeting was opened by the talk of Prof. V. I. Chizhik (Saint Petersburg, Russia) "Peculiarities, possibilities, and areas of applications of quantitative analysis by NMR in magnetic field of the Earth".

During "Spinus 2019" there were 14 lectures, 31 oral (15-20') and 67 poster presentations. Besides, to young scientists with poster presentations the opportunity was given to make oral blitz-reports (5'), more than 30 such reports were made at the conference. Many „mature" participants noted that this experiment was successful: it allowed young scientists to demonstrate their level and gave them the confidence to participate at the future conferences with full oral reports.

Barbara and Uwe Eichhoff (Germany) founded prizes of 200 and 100 Euro, respectively, for the best oral and poster reports of students and postgraduate students. Nominees were selected by an international commission. The Organizing Committee has awarded:

The best oral report:
Sarah Schneider (Institute of Condensed Matter Physics, TU Darmstadt, Darmstadt, Germany)
"Ion and Molecule Transport in Aqueous Salt Solutions in Bulk and in Nanopores - a NMR study". (p. 8)
The best poster presentation:
Naira R. Khusnutdinova (Institute of Physics, Kazan Federal University, Kazan, Russia)
"Modeling of Dynamics and Calculation of Relaxation Parameters for Monomer of Bovine Insulin". (p. 10)

The social program of the Spinus 2019 consisted of a welcome party, a conference dinner and a bus sightseeing tour to Kronstadt (a port-town in Russia located on the island of Kotlin and the smaller islands of the Gulf of Finland and the dam adjacent to it).

Welcome to the next meeting "Spinus 2020"
Saint Petersburg, Repino, March 29 - April 4, 2020!

Best oral report: Spinus 2019

Sarah Schneider, Technical University Darmstadt

Ion and Molecule Transport in Aqueous Salt Solutions in Bulk and in Nanopores - a NMR study

Sarah Schneider, Christoph Säckel, Michael Vogel
Institute of Condensed Matter Physics, TU Darmstadt, 64289 Darmstadt, Germany
michael.vogel@physik.tu-darmstadt.de, www.fkp.tu-darmstadt.de/groups/ag_vogel

Introduction

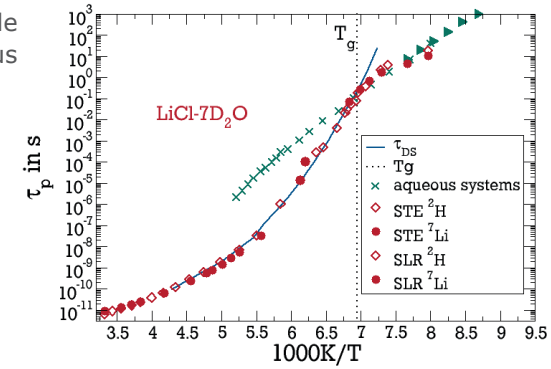
We analyze ion and molecule transport in aqueous salt solutions confined to nanopores as part of a project that aims to develop a new generation of nanosensors by combining biological and synthetic nanopores. While being highly selective and sensitive, biological ion channels lack the robustness for technological applications. Contrarily silica pores are well proven in industrial and clinical environments, but possess inferior capabilities, e.g. no selectivity. A hybrid system would combine the favorable properties of both fields.

Samples and Experiments

To optimize such pores, it is of large interest to understand the influence of the confinement on the T-dependent ion and molecule transport inside. We vary the pore parameters systematically and study their effects on the dynamics by NMR. Using ^{1}H and ^{2}H NMR we can selectively investigate water dynamics whereas ^{7}Li and ^{23}Na NMR analyze the local and long-range dynamics of ionic species. Samples of interest are LiC1-H_2O and LiC1- D_2O at various concentrations in the bulk and in MCM-41 with pore diameters of d=2.8- 3.8nm.

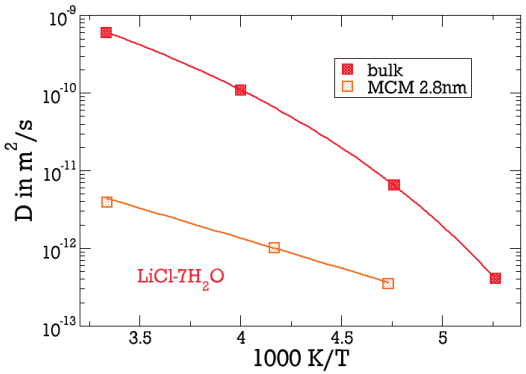
Bulk solutions [1]

Analysing the local ion and water dynamics in the bulk solutions reveals a slowdown with increasing salt concentration at ambient temperatures, while this difference vanishes upon cooling due to partial crystallization. Relaxation times and diffusion coefficients of water molecules agree with those of lithium ions in the weakly supercooled regime, indicating that the dynamics are strongly coupled. However, we see a decoupling in the strongly supercooled regime, most notably of the rotational motion of the water molecules, which does not follow the glassy slowdown of the studied salt solutions below ~145 K, but it rather resembles that in nanoscopic confinement, molecular solutions, and high-density amorphous ice at low temperatures. This common low temperature water dynamics is characterized by large-angle reorientation and Arrhenius temperature dependence.



Confinement

At a given concentration there is a slowdown in confinement with more heterogeneous dynamics. This can be explained by a slower layer at the pore walls and bulk-like dynamics in the pore center. Field-gradient NMR is applied to measure selfdiffusion. The extent of the effect and the relation between short- and long-range dynamics depend on the confinement properties, including filling degree and pore size, which also strongly affect a possible crystallization.



Acknowledgements
The authors thank the LOEWE project iNAPO funded by the Ministry of Higher Education, Research and the Arts (HMWK) of the Hessen state for financial support.

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Best poster presentation: Spinus 2019

Naira R. Khusnutdinova, Kazan Federal University

Modeling of dynamics and calculation of relaxation parameters for monomer of bovine insulin

Naira R. Khusnutdinova, Aidar R. Yulmetov
Institute of Physics, Kazan Federal University, Kazan, Russia
E-mail: nai.khus@yandex.ru

Introduction

The main effect of insulin is to reduce the concentration of glucose in the blood. If there is insulin disruption of work or secretion disorder one may suffer from diabetes mellitus (DM). It is classified into types: 1, 2, gestational DM, and other cases such as monogenic diabetes syndromes [1]. All patients with type 1 DM and many suffering from the other types have to take insulin injections. So, it is really important to study molecular properties of insulin. In this work computer model of bovine insulin monomer was made and were calculated parameters such as correlation function and cross-relaxation rate, compared with experiment.

Theory

The primary structure of insulin in different species varies, as does its importance in the regulation of carbohydrate metabolism. Closest to human is pig insulin, which differs with only one amino acid residue, next closest variant is bovine insulin – it differs by three amino acid residues: A1a8A–Thr8A, Val10A–Ile10A, A1a30B–Thr30B (former residues belong to the bovine form; latter – human). This difference does not alter the most conservative and important segments of insulin, A1–A3, A12–A17, and B8–B25 [2].

Synthesis of insulin begins with production of a longer peptide, then disulfide bridges are formed and a segment of the chain is removed. Mature insulin molecule consists of two chains called A (shorter) and B (longer). Six insulin molecules are associated in a hexamer (three symmetrical axes are visible). Molecules hold together histidine residues bound by zinc ions. The insulin is injected under the skin in the form of a hexamer, gradually breaking down into biologically active monomers entering the bloodstream [3].

The main quantity from 2D NOE experiments is the cross-relaxation rate σij describing how magnetization is transferred between spins i and j via dipolar coupling. Theory, relating relaxation rates from NOE experiments to correlation functions describing molecular motions has been reviewed in publications [4,5]:

$$\sigma_{ij} = \frac{\pi}{5}\gamma^4\hbar^2[6J_{ij}(2\omega) - J_{ij}(0)] \quad (1)$$

where ω – Larmor frequency, γ – gyromagnetic ratio of protons. J_{ij} – spectral densities, which characterize the modulation of dipolar coupling between nuclei with time.

While the spectral density is the Fourier transform of the correlation function of the dipole-dipole interaction [6], which may be calculated basing on coordinates of each nuclei.

$$C(t) = \frac{1}{5}\langle\frac{P_2(\hat{\mu}_{L,ij}(t)\hat{\mu}_{L,ij}(0))}{r_{ij}^3(t)r_{ij}^3(0)}\rangle \quad (2)$$

where $\hat{\mu}_{L,ij}$- unit vector in the direction of the inter-proton vector, $r_{ij}(0)$ – the distance of the two protons, P_2 – second Legendre polynomial.

Computer modelling

Studied molecule was modeled with GROMACs [7] package (fig.1). Monomer of bovine insulin was relaxed through an energy minimization, then placed in water. There were made equilibration conducted in two phases – NVT, NPT (constant Number of particles, Volume (or Pressure) and Temperature). After, trajectories of nuclei of this molecule in water were collected.

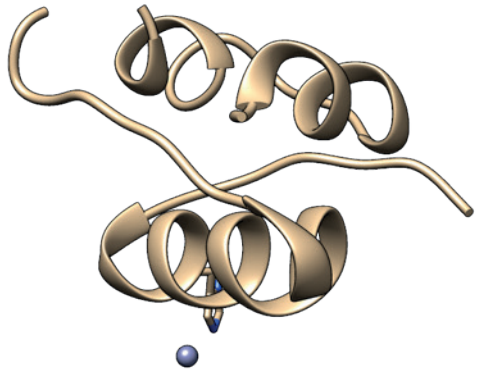

Figure 1. Monomer of bovine insulin

With MatLab package was made code based on GROMACs trajectories data. Example of such correlation function for IleA2 Ha -IleA2 HN is given below (fig.2):

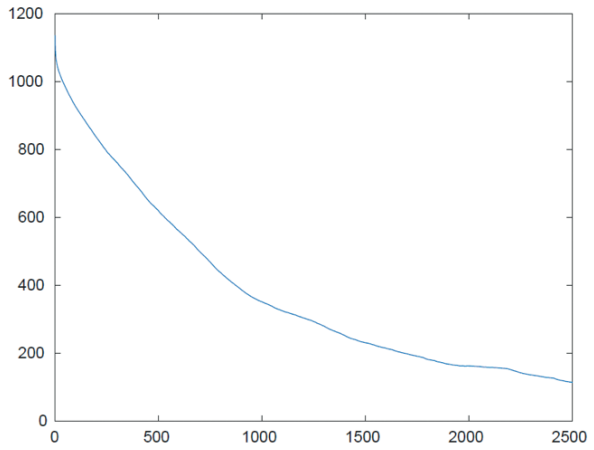

Figure 2. Correlation function

With these correlation functions were calculated cross-relaxation rates, which may be compared with experimental ones.

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First announcement

17th International School-Conference ‚Magnetic Resonance and it's Applications' Spinus 2020

29. March - 4. April
Saint Petersburg State University, Russia
http://spinus.spb.ru

Invitation
Welcome to the 17th International School-Conference "Magnetic resonance and its applications. Spinus" organized by Saint Petersburg State University from March 29 to April 4, 2020. The goal of "Spinus" is to provide a platform to young scientists for the use of all aspects of magnetic resonance methods and techniques, as well as computational and theoretical approaches, for the solving of fundamental and applied problems in physics, chemistry, medicine and biology. The number of participants is limited by 200 persons. The official language of the "Spinus" is English.

Scope
The scope of the Spinus Conference includes the following topics:
- Modern trends in NMR, ESR and NQR
- Magnetic resonance for fundamental science
- Magnetic resonance imaging
- Computer Modeling for magnetic resonance
- Earth's field NMR
- Magnetic resonance in industry
- Related areas

Extended abstracts will be published in the Book of Abstract.
Selected papers of participants will be published in a special issue of "Applied Magnetic Resonance".

Application to Spinus 2020
The application for Spinus 2020 will be available on the website: spinus.spb.ru by 1st of October, 2019.
If you were registered for Spinus 2018 or Spinus 2019, your account is still valid and in this case you can create an application for Spinus 2020 in your profile.

Conference Fee
The participation fee is 13‘000 RUB (≈175 €) for active participants and 6‘500 RUB (≈85 €) for young scientists (students and Ph.D. students) and accompanying persons. For Russian citizens (their participation is supported by Russian funds) participation fees are 4‘000 RUB and 2‘000 RUB, respectively. Participation fees include organization costs, visa support, conference materials, welcome-party, coffee breaks, transportation on the days of arrival and departure.
20% discount is available for early application and abstract uploading before December 3, 2019.

AMPERE discount for non-Russian citizens. If your annual Ampere Member fee has been paid (35 € for regular participant and 17,50 € for student) the registration fee will be reduced by this amount.

Abstract and Paper Submission
Abstracts up to 3 pages (including tables and figures) in the MS Word format, according to the conference template, should be uploaded to Spinus website application until the 1st of March, 2020.
The abstract template will be available on the Spinus website (on the 1st of October, 2019).

All accepted abstracts will be placed in Russian Science Citation Index and will be available on the resource www.elibrary.ru
In 2020 selected papers of the participants will be published in a special issue of “Applied Magnetic Resonance” with the standard reviewing process (the journal is indexed by Web of Science and Scopus). The journal website is http://www.springer.com/materials/journal/723

Location
The conference will take place at the hotel „Baltiets“ in Repino on the beach of the Gulf of Finland, 30 km north-west from the of St. Petersburg center (http://baltiets.ru/).
Estimated minimal cost for a one-day stay in the hotel (full board including a double room, i.e. 1‘800 RUB per person. It is possible to book a single room. The exact price will be announced in the beginning of 2020.


Organizing Committee
Chairman: Prof. Denis Markelov,
Vice-chairman: M. Sc. Alexandr Ievlev,
Committee members: Dr. Sci. Marina Shelyapina, Dr. Andrey Egorov, Dr. Andrey Komolkin, Dr. Pavel Kupriyanov, Dr. KonstantinTutukin,
M. Sc. Timofey Popov

Scientific adviser of the School-Conference “Spinus”
Professor Vladimir Chizhik

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Report:
AMPERE NMR School 2019

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Bernhard Blümich (Aachen), Germany, Janez Dolinšek (Ljubljana), Slovenia, Franz Fujara (Darmstadt), Germany, Stefan Jurga (Poznan), Poland, Wiktor Koźmiński (Warszawa), Poland, David Lurie (Aberdeen), UK, Alex MacKay (Vancouver), Canada, Beat Meier (Zurich), Switzerland, Shimon Vega (Rehovot), Israel.

Organizing Committee:
Stefan Jurga, Chair
Lidia Szutkowska, Executive Secretary,
Roksana Markiewicz, Jakub Jagielski, Adam Klimaszyk, Jacek Jenczyk,
Zbigniew Fojud, Kosma Szutkowski.

The AMPERE NMR School was held between 23rd and 29th June 2019 in Zakopane, Poland, situated in the High Tatra Mountains. The School had 94 participants from research institutions of 17 countries, representing 20 nationalities: Argentina, Canada, China, Spain, Estonia, Finland, France, Germany, India, Italy, Netherlands, Poland, Russia, Slovenia, Sweden, Switzerland, Turkey, Ukraine, United Kingdom and United States. The conference was organized by the NanoBioMedical Centre, and the Centre for European Integration of the Adam Mickiewicz University in Poznań, under the auspices of the Groupement AMPERE. It was focused on the basic and advanced NMR techniques and attracted young and experienced scientists from across the world, who had the opportunity to exchange knowledge and ideas on recent NMR and related research, to learn about new techniques and developments in this field and to establish new contacts and collaborations.

The programme of the School covered the following topics: NMR relaxometry, NMR diffusometry, Solid State NMR, NMR of quadrupolar nuclei, MRI and Field Cycling MRI, application of NMR in the area of biology, medicine and material science and technical aspects of NMR. In total it consisted of 21 lectures, given by:

Dr Witold Andrałojć
G-quadruplex nucleic acids studied by NMR spectroscopy
Institute of Bioorganic Chemistry, Polish Academy of Sciences, Poznań, Poland
Prof. Esteban Anoardo
Experimental issues on field-cycling NMR: critical aspects, physical and technical limits
National University of Cordoba, Cordoba, Spain
Prof. Bernhard Blümich
Mobile NMR: Principles and unusual applications
RWTH Aachen University, Aachen, Germany
Prof. Gerd Buntkowsky
Hyperpolarization with Parahydrogen
Technical University of Darmstadt, Darmstadt, Germany
Prof. Janez Dolinšek
NMR study of structural injection grouts for historical building repair
Jozef Stefan Institute and University of Ljubljana, Ljubljana, Slovenia

Prof. Matthias Ernst
Manipulating Proton-Driven Spin Diffusion under MAS solid-state NMR
ETH Zurich, Zurich, Switzerland
Prof. Fabien Ferrage
Relaxometry of biological macromolecules in solution
University of Sorbonne, Paris, France
Prof. Olli Gröhn
NMR/MRI in biomedical application – rotating frame relaxation MRI contrast
University of Eastern Finland, Kuopio, Finland
Prof. Wiktor Koźmiński
High dimensionality and high resolution NMR experiments for biomolecules
University of Warsaw, Warsaw, Poland
Prof. Danuta Kruk
Deep physics behind quadrupole peaks
University of Warmia & Mazury in Olsztyn, Olsztyn, Poland
Prof. Ilya Kuprov
Spatial dynamics in NMR and how to simulate it
University of Southampton, Southampton, United Kingdom
Prof. David Lurie
MRI basics and research on fast-field cycling MRI
University of Aberdeen, Aberdeen, United Kingdom
Prof. Alex MacKay
Many ways to separate spin reservoirs in brain
University of British Columbia, Vancouver, Canada
Prof. Beat Meier
Solid-state protein NMR: Resolution and sensivity for fast MAS experiments
ETH Zurich, Zurich, Switzerland
Prof. Dieter Michel
Nitric oxide – an interesting molecule for biomedical applications: and EPR and NMR study
University of Leipzig, Leipzig, Germany
Prof. Siegfried Stapf
NMR of wetting – how to rock it despite of ageing
Technical University of Ilmenau, Ilmenau, Germany
Prof. Janez Stepišnik
Molecular dynamics in simple liquids and their mixtures studied by NMR diffusion spectroscopy
University of Ljubljana, Jožef Stefan Institute, Ljubljana, Slovenia
Prof. Ville-Veikko Telkki
Multidimensional relaxation and diffusion measurements
University of Oulu, Oulu, Finland

Prof. Daniel Topgaard
Multidimensional diffusion MRI
Lund University, Lund, Sweden
Prof. Jadwiga Tritt-Goc
Molecular dynamics in cellulose-based gel and composites studied by NMR
Institute of Molecular Physics Polish Academy of Sciences, Poznań, Poland
Ziqing Wang
Interaction of Metabolites with Macromolecules Investigated by high-resolution NMR relaxometry
University of Sorbonne, Paris, France

The programme was enriched by additional tutorials. The first one was given by Prof. Ilya Kuprov, and was entitled "NMR Simulation Workshops", and focused on the treatment of spatial degrees of freedom (rotational and translational diffusion, hydrodynamics, spatial encoding, etc.) in magnetic resonance simulations. Moreover, 5 tutorials with online transmissions from the NMR laboratory of the NanoBioMedical Centre in Poznań were performed:
1. Two-dimensional NMR spectroscopy - Witold Andrałojć
2. NMR relaxometry - Zbigniew Fojud, Maria Dobies, Mariusz Jancelewicz
3. Solid-state NMR - Jacek Jenczyk
4. NMR diffusometry - Kosma Szutkowski
5. MRI: basic principles and application - Tomasz Zalewski

All participants were given the opportunity to present their research in a poster session which consisted of 65 presentations. The prizes were awarded to the authors of the three best posters:

First place (page 21)
Yijue Xu from University of Ottawa, Canada, for the poster entitled „Single-crystal NMR characterization of Halogen Bonds".

Second place (page 23)
Molly Wagner from University of Delaware, United States, for the poster entitled: „The kinetics of soap formation in oil paints and the effect of relative humidity: quantitative NMR measurements".

Third place (page 26)
Aditya Mishra from Swiss Federal Institute of Technology in Lausanne, Switzerland, for the poster entitled „High-Field Solid-State MAS NMR: A unique tool to look at the Atomic-Level microstructure of Lead Halide Perovskites for Optoelectronics".

The posters were evaluated by the members of the Poster Committee that consisted of Prof. Alex Mackay (chair), Prof. David Lurie, Prof. Ilya Kuprov, Prof. Bernhard Blümich, Prof. Matthias Ernst and Prof. Daniel Topgaard. All the winners received prizes accompanied by a certificate.

All abstracts of the oral presentations, tutorials and posters were published as printed proceedings (book of abstracts).

The social programme included "All together party", Dinner in the Regional Restaurant "Biały Potok" with the folk music of the Tatras region, excursions to Rusinowa Clearing and Dunajec River Rafting.
Participants also had the opportunity to listen to an organ recital performed by Prof. Dieter Michel from the University of Leipzig. The School received a lot of positive feedback based on survey conducted among its participants.

All the additional information about the AMPERE NMR School are presented on the website: school.home.amu.edu.pl or on Twitter: @AmpereNMR.

The next edition of the School will be held in Zakopane (Poland) from June 21st to 27th, 2020.

First Poster Prize, Ampere NMR School 2019

Yijue Xu, University of Ottawa, Canada

Single-crystal NMR characterization of Halogen Bonds

Yijue Xu, Bulat Gabidullin, and David L. Bryce
Department of Chemistry and Biomolecular Sciences & Centre for Catalysis Research and Innovation, University of Ottawa, Ottawa, Canada

Halogen bonding is a noncovalent interaction between the electrophilic region of a halogen, and an electron donor. This interaction is highly directional and comparable to hydrogen bonding, and has therefore gained an increasing amount of attention in different fields, such as catalysis, drug design etc.[1] Analysis of single-crystal NMR spectra, which can reveal the combined effects of quadrupolar coupling, magnetic shielding, and spin-spin coupling, allows for the measurement of the tensor magnitude and orientation for these anisotropic NMR interactions in the molecular frame.

Here, ^{17}O enriched triphenylphosphine oxide and three of its halogen-bonded cocrystals [2] featuring 1,4-diiodotetrafluorobenzene and 1,3,5-trifluoro-2,4,6-triiodobenzene as halogen bond donors have been characterized by ^{31}P and ^{17}O single-crystal NMR spectroscopy. ^{31}P chemical shift tensors, ^{17}O chemical shift tensors, ^{17}O quadrupolar coupling tensors, and ^{31}P-^{17}O indirect nuclear spin-spin (**J**) coupling tensors are reported for P=O···I halogen bonds. The angles between the direction of the unique component of the NMR tensors (chemical shift tensors and quadrupolar coupling tensors) and O···I halogen bond correlates with the deiviation in linearity of the P=O···I halogen bond. There is also clear decrease in anisotropy and an increase in asymmetry of the **J**(^{31}P,^{17}O) coupling tensors attributable to the formation of iodine-oxygen halogen bonds.

This work, encompassing the first ^{17}O single-crystal NMR studies of halogen bonds, demonstrates how NMR observables provide a useful means to characterize non-covalent interactions and also helps in our understanding of the correlation between the electronic structure of the halogen bond and NMR properties.

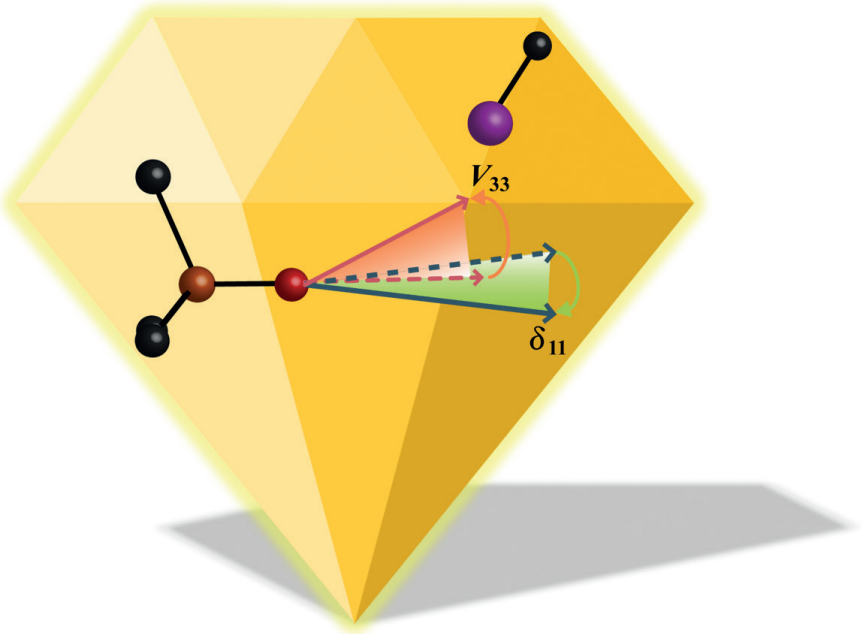

Figure 1. Schematic representation of CS and EFG tensor orientation changes upon halogen bonding between oxygen (red) and iodine (purple)..

Acknowledgements:
Dr. César Leroy, Dr. Eric Ye, and Dr. Glenn Facey are thanked for technical assistance with the DFS-pulse experiment.

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Second Poster Prize, Ampere NMR School 2019

Molly Wagner, University of Delaware, United States

The Kinetics of Soap Formation in Oil Paints and the Effect of Relative Humidity: Quantitative NMR Measurements

Anna Murphy[a], Yao Yaoa[b], Molly Wagner[a], Silvia A. Centeno[b], Valeria Di Tulliob[c], Guenther Neue[d], Jaclyn Catalano[e], Nicholas Zumbulyadisa[f], Cecil Dybowski[a]

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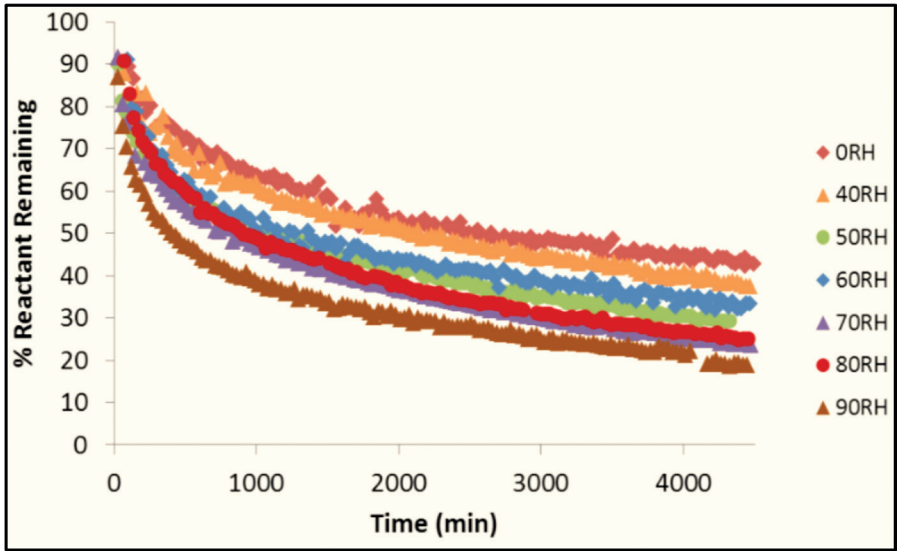
Traditional oil paints consist of pigment(s) in an oil binder. While there are many options for the oil binder, linseed oil has been and remains one of the most common, due to its high proportion of unsaturated fatty acids and therefore propensity for drying. These unsaturated fatty acids cross-polymerize as the paint dries, forming a complex matrix. The remaining fatty acids, primarily palmitic and stearic, are unsaturated, do not polymerize, and become "free" in the network. These free fatty acids (FFAs) can react with metal ions in inorganic pigments, such as those in zinc white or basic lead white, or in paint driers to produce heavy-metal carboxylates, colloquially known as soaps. The soaps contribute to the deterioration of the host media by consuming the colored pigment particles during formation then potentially manifesting as protrusions, hazy films, delamination, or cracks, or increasing paint transparency. Evidence of soap formation has been reported in works ranging from the 15th century to the present across numerous cultures. Due to the pervasiveness of oil paints, there is growing concern about the effects of soaps among art conservators worldwide.

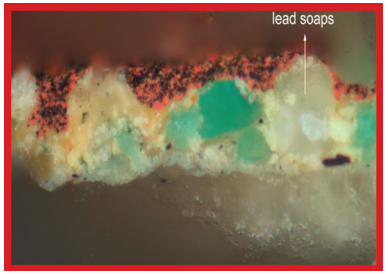
The formation of soaps is a multistep process which is still largely not understood. We propose the following steps: 1. FFA formation; 2. Movement of reactant(s), either FFAs, pigment ions, or both; 3. Soap formation; 4. Soap aggregation; 5. Irreversible damage becomes visible. We focused our studies on steps 2 and 3 in model paint films. Step 2 involves the relative movement of the fatty acids and pigment ions, usually by mutual diffusion. Our studies, based on single-sided NMR relaxometry of the diffusion of water or acetone, emphasize the importance of assisted diffusion in carrying the reactive species to contact.

To study the molecular dynamics of water and solvent molecules in the model oil paints, we measured the relaxation times T_1 and T_2 of hydrogen nuclei by single-sided-NMR, comparing the behavior at different relative humidity values (40 and 90% RH). To address step 3, we utilized solid-state NMR spectroscopy to monitor the progress of the overall reaction in model paint films when excess FFAs were introduced in the system, in this case ^{13}C-enriched palmitic acid, and the samples were exposed to different RH levels. The excess of FFAs is necessary to facilitate the reaction, allowing it to take place over a few days rather than years or centuries. The time dependence of the product concentration in these model systems is complex and varies with the exposure of the paints to water.

We propose a novel theoretical explanation of the kinetics of soap formation in a model paint system with high concentrations of free fatty acids that agrees with our experimental data, suggesting that the rate-limiting step for the process under these conditions is the diffusion of the reactants through the soap layer that forms around the pigment particles. We observed that the rate of the reaction varies with the RH, suggesting that the rate of formation of the soap layer and the diffusion through it depend on the amount of water in the paint film.



Fraction of palmitic acid remaining vs. time at different % relative humidities monitored by ^{13}C NMR.



Francesco Granacci, The Birth of Saint John the Baptist, ca. 1506-07 (above) and a cross section containing lead soaps protruding through the paint surface (below).

Third Poster Prize, Ampere NMR School 2019

Aditya Mishra , Ecole Polythechnique Fédéral de Lausanne, CH

High-Field Solid-State MAS NMR: A unique tool to look at the Atomic-Level microstructure of Lead Halide Perovskites for Optoelectronics

Aditya Mishra[1], Dominik J. Kubicki[1,2], Michael Grätzel[2], Lyndon Emsley[1]

[1]Laboratory of Magnetic Resonance, Institute of Chemical Sciences and Engineering, Ecole Polytechnique Fédérale de Lausanne (EPFL), 1015 Lausanne, Switzerland
[2]Laboratory of Photonics and Interfaces, Institute of Chemical Sciences and Engineering, Ecole Polytechnique Fédérale de Lausanne (EPFL), 1015 Lausanne, Switzerland

The emergence of lead halide perovskites (LHP) based solar cells has revolutionized the field of photovoltaics (PV). LHP are solution processable and allow unprecedented fabrication flexibility, while providing power conversion efficiencies greater than 24% within just a few years since the first report [1]. However, current best compositions still lacks in terms of long term stability, which is the biggest obstacle in their commercialization. The state-of-the-art perovskite compositions are multi-component organic-inorganic mixtures ($APbX_3$, A=Cs^+, Rb^+, K^+, $CH_3NH_3^+$, $CH(NH_2)_2^+$; X=I, Br, Cl) often additionally treated with small organic molecules to improve the optoelectronic performance on a trial-and-error basis [2].
Till very recently, the atomic-level mechanism of these improvements has been largely unknown. Here, we will discuss how the use of high-field MAS NMR has allowed the first quantitative studies of order, disorder, dynamics and phase segregation phenomena in these highly complex materials. We have applied ^{133}Cs, ^{87}Rb and ^{39}K MAS NMR at up to 21.1 T to show that, contrary to previous conviction, rubidium and potassium are not incorporated into lead halide perovskites, and confirmed it is the case for cesium [3,4]. We have developed a cryo-NMR methodology to obtain ^{13}C and ^{15}N CP spectra with unprecedented sensitivity, essential for the study of A-site cation mixing [5-8]. Further, we have shown that ^{14}N MAS NMR is a sensitive probe of the cubooctahedral symmetry, capable of capturing structural changes beyond the detection limit of XRD, while ^{2}H MAS NMR yields cation-specific information on dynamics in multi-cation materials [7,8]. The use of paramagnetic NMR strategies has allowed us to prove incorporation of transition metals and lanthanides into perovskites in a new unambiguous way [9,10]. In addition, we have used ^{1}H-^{1}H spin diffusion measurements to evidence atomic-level interaction between various organic passivation agents and the perovskite structure, leading to a new structural model [11]. We have applied many of these methods to thin films, providing structural information about materials directly used for optoelectronic device fabrication.

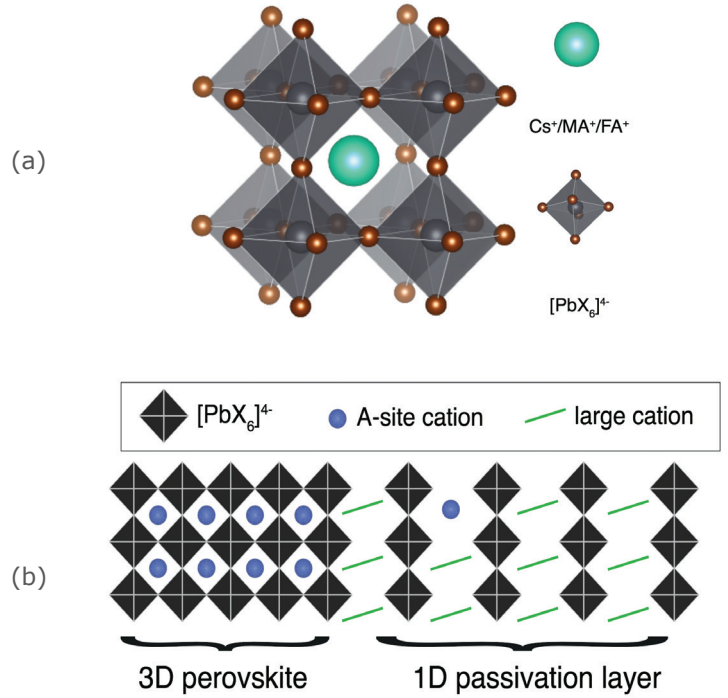


Figure (a) Perovskite ABX_3 structure (A=Cs^+, Rb^+, K^+, $CH_3NH_3^+$, $CH(NH_2)_2^+$; X=I, Br, Cl)
Figure (b) Perovskite with the passivation layer

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Future conferences

Ampere Events 2019

ISMAR / Euromar 2019	Berlin (Germany)	August 25-30 2019
XITH EFEPR Conference	Bratislava (Slovakia)	September 1-5 2019
11th Alpine Conference on Solid-State NMR	Chamonix (Switzerland)	September 15-19 2019
8th EFEPR School	Brno (Czech Republic	November 18-25 2019

non Ampere Event 2019

12th Australian and New Zealand Society for Magnetic Resonance (ANZMAG)	Pullman Bunker Bay Resort (Australia)	November 25-28 2019

Ampere Events 2020

17th International School-Conference ‚Magnetic resonance and its applications‘ Spinus 2020	Saint Petersburg (Russia)	March 29 to April 4 2020
MR Food 2020	Aarhus (Denmark)	June 1-4 2020
Ampere NMR School 2020	Zakopane (Poland)	June 21-27 2020
Euromar 2020	Bilbao (Spain)	July 5-9 2020
15th Magnetic Resonance in Porous Media	Tromsø (Norway)	August 24-28 2020
HYP20 + Dissolution and MAS DNP hands-on training at CMRN	Lyon (France)	August 30 to September 2 2020