Dear members of Groupement AMPERE,

hopefully you have already lost the weight gained during EUROMAR 2017 in Warsaw, Poland due to the plenty amounts of good food available in the coffee and lunch breaks and during the receptions by Bruker and JEOL. It was a scientifically opulent conference, too, where it was often difficult to select which of the parallel sessions to attend. Great thanks go to Wiktor Koźmiński and his team as well as to the International Scientific Committee for making this such a memorable event. Please have a look at the opening address by the President of Groupement AMPERE, Bernhard Blümich and at the report on the conference by Wiktor Koźmiński.

Earlier this year the 14th International Youth School-Conference „Magnetic resonance and its applications - Spinus-2017”, organized by Denis Markelov and Vladimir Chizhik, was taking place in St. Petersburg, Russia. This Bulletin issue presents the Award winners of the conference, Ekaterina S. Babicheva and Sergey Sokratilin. Likewise, the poster prize winners of EUROMAR 2017, Shyamsundar Adhikari, Walter Becker, and Christian Bengs are featured. Last but not least, the winner of this year’s Raymond Andrew Prize, Andrin Doll, describes his research in this issue.

We also continue our series of portraits of well-known scientists in magnetic resonance, this time with Beat H. Meier, Past President of Groupement AMPERE. Your suggestions on whom we should interview for one of the upcoming issues are welcome: admin@ampere-society.org. Enjoy the rest of the summer, unless you live in the Southern Hemisphere.

Gunnar Jeschke
Secretary General of Groupement AMPERE
Portrait: Prof. Beat Meier

• why magnetic resonance and why NMR and MRI?
  Because I started to understand quantum mechanics in Richard Ernst’s NMR lecture

• what is your favorite frequency?
The higher the better, for Larmor- and Rabi- and MAS frequencies

• what do you still not understand?
  How to properly interpret spin relaxation

• luckiest experiment you have ever done.
  My diplomar work: 2D exchange; everything worked immediately

• what was the worst mistake you have made during your lab time?
  Dropping a box with 2000 IBM punch cards to the ground, scrambling the lines of my program code.

• most memorable conference story
  Running down from the Lac Noir in Chamonix to make the evening session.

• with whom (historical person) would you like to meet?
  Felix Bloch

• when do you get your best ideas?
  Hiking in the mountains

• if you had just one month time for travelling - where would you go to?
  The Himalayas

• your idea of happiness.
  A scientific world without hype, impact indices and h factors but with collaboration and friendship.

Position:
Professor of Physical Chemistry
http://www.nmr.ethz.ch

Awards:
1992 Ruzicka Prize, ETH Zürich, Switzerland
2008 ISMAR Fellowship, ISMAR (International Society for Magnetic Resonance)
2010 Arthur D. Little Lecture, MIT, Cambridge, MA, USA
2014 Günther Laukien Prize, 55th ENC, Boston, MA, USA
2017 Member of the Academy Leopoldina

Education:
1974 - 1978 Undergraduate Studies at ETH Zurich, Department of Chemistry. Diplomar thesis under the direction of Prof. R.R. Ernst
1978 Dipl. Chem. ETH Zurich
1979 - 1984 Graduate studies at the Laboratory of Physical Chemistry (LPC), ETH Zurich under the direction of Prof. R. R. Ernst and Prof. A. Furrer. Teaching assistant
1984 - 1986 Postdoctoral Fellow at Los Alamos National Laboratory 1993
1994 - 1998 Full professor in Physical Chemistry at the University of Nijmegen (NL) and member of the board of the Nijmegen SON Research Center for Molecular Structure, Design and Synthesis and of the board of the National High-Field NMR facility
1998 - present Full professor in Laboratory of Physical Chemistry at the ETH Zurich

Interests:
Solid-state NMR, basics, hardware and application to proteins. Science and society. Traveling, hiking, skiing.
Since 2004 the Saint-Petersburg University holds a series of the annual International Youth School-Conference «Magnetic Resonance and its Applications. Spinus» (an AMPERE event since 2016). The 14th Meeting “Spinus-2017” was opened on Monday, April 24, and was closed on Friday, April 28, 2017. Venue: the hotel „Baltiets“ in Repino on the beach of the Gulf of Finland, 30 km northwest 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 Meeting was attended by 95 participants from 10 countries (Belgium, Cuba, Finland, France, Germany, Italy, Russia, Sweden, Turkey, USA).

Prof. V. I. Chizhik (Saint Petersburg, Russia) opened the meeting with his talk “A few details about Magnetic Resonance in Russia”.

During “Spinus 2017” there were 17 lectures, 36 oral (20-30’) and 35 poster presentations. The organizers published the Book of Abstracts. Selected papers (12 manuscripts) will be published in a special issue of the journal “Applied Magnetic Resonance”.

The Organizing Committee founded prizes (200+100 $) for the best oral and poster reports of students and postgraduate students. Nominees were selected by an international commission. The Organizing Committee awarded the best oral reports.

The winner:
Ekaterina S. Babicheva “Study of nanoemulsions of the hydrophobic phthalocyanine in pluronic aqueous solutions” (Moscow Institute of Physics and Technology) Abstract see page 10.

Laureates:
Sevastyan O. Rabdano “Reconstruction of particle size-distribution from conjunction of NMR and DLS diffusion data” (St. Petersburg State University)
Gleb L. Denisov “Paramagnetic terpyridine complexes of d-metals by methods of the magnetic resonance spectroscopy” (Nesmeyanov Institute of Organoelement Compounds of Russian Academy of Sciences, Moscow)

The best poster presentations
The winner:
Sergey V. Sokratilin “NMR relaxation and diffusion in aqueous solutions of fullerens and fullerenes with PVP and dextrine complexes.” (St. Petersburg State University) Abstract see page 14.

Laureates:
Daria L. Melnikova “Investigation of α–casein translational mobility by NMR methods” (Kazan Federal University)
Ksenia A. Levchuk “Methodology of intravital labeling of human mesenchymal stromal cells with superparamagnetic iron oxide nanoparticles” (Pokrovsky bank of stem cells, St. Petersburg)

The social program of the Spinus-2016 consisted of a welcome-party, conference dinner and excursion in the famous museum „The Hermitage“ (St. Petersburg).
First announcement

15th International Youth School-Conference
Magnetic resonance and its applications
Spinus 2018

Saint Petersburg State University, Saint Petersburg
1 – 6 April 2018

Invitation

Welcome to the 15th International Youth School-Conference „Magnetic resonance and its applications“ (Spinus 2018) organized by Saint Petersburg State University in April, 1–6, 2018. 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.

Scope

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-2018” is English. 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”.

Registration fee

The registration fee is 9300 rub. (≈150 €) for active participants and 4340 rub. (≈70 €) for young scientists (students and Ph.D. students) and accompanying person. For Russian citizens the fee is 3000 rub and 1500 rub, respectively.

Registration fees include organization costs, “Spinus 2018” materials, welcome-party, coffee breaks, transportation in the days of arrival and...
departure. Online registration will be available by September 1st, 2017, on the conference site: http://nmr.phys.spbu.ru/spinus

Location
“Spinus-2018” will take place at the hotel „Baltiets“ in Repino on the beach of the Gulf of Finland, 30 km north-west from the center of St. Petersburg (http://baltiets.ru/). Estimated minimal cost for a one-day stay in the hotel (full board including also swimming pool and sauna) is 3600 rub (≈ 52 €) for two persons in a double room, i.e. 1800 rub per person. The exact price will be known later (February 2018). It is possible to book a single room.

Abstract and paper submission
Abstracts up to 3 pages (including tables and figures) in MS Word format, according to the conference template, should be sent to the e-mail address spinus@nmr.phys.spbu.ru as an attachment (please, put the subject „Spinus-2018 abstract“) until the 1st of March, 2018. The abstract template will be available on the website.

All accepted abstracts will be placed in Russian Science Citation Index and will be available in the resource www.elibrary.ru

In 2018 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 site: http://www.springer.com/materials/journal/723
Articles prepared in accordance with the requirements of the journal should be sent to the e-mail address: spinus@nmr.phys.spbu.ru (please put the subject "Spinus-2017 AMR-paper"). Deadline for the manuscript submission: April 30. 2018.

Organizing committee
Chairman: Dr. Sci. Denis Markelov
Vice-chairman: Alexander Ievlev
Committee members: Dr. Andrey Egorov
Dr. Andrey Komolkin
Pavel Kupriyanov
Andrey Chudin
Dr. Konstantin Tutukin
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Scientific adviser of the School-Conference “Spinus”
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Ekaterina S. Babicheva
Study of pluronic nanoemulsions with the hydrophobic phthalocyanine

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The solubilization of hydrophobic compounds is an actual issue in different spheres of human activities. This problem becomes particularly important in pharmaceutical applications where the most effective and safe perspective compound could be inapplicable only due to its hydrophobicity. Solubilization of hydrophobic compound in surfactants aqueous solutions is one of the tools to resolve the problem, and understanding of solubilization mechanisms is a rather important task. NMR spectroscopy is a powerful technique for this purpose. It has been successfully applied on the intra- and intermolecular interactions in pluronic systems [1].

Phthalocyanines are a class of organic compounds widely used in different branches of science and technology, particularly as photosensitizers for photodynamic therapy of oncological diseases [2]. At the present work, we studied the solubilization of model phthalocyanine 1 in aqueous solution of polymeric non-ionic surfactant Pluronic F68 (Fig. 1).

![Fig. 1. Structures of pluronic F68 (a), model phthalocyanine 1 (b) and 3D structure of these molecules (c)](image)

It is known that pluronics tend to aggregate with increasing of concentration and/or temperature. For pluronic F68 it was found that its temperature-induced aggregation is accompanied by pronounced changes in its 1H-NMR spectra: the appearance of a new resonance signal of propylene oxide CH2-protons is observed. This phenomenon could be explained by conformational changing of propylene oxide fragments of the chain from gauche to anti, which was also confirmed by FT-Raman and FT-IR studies [3, 4]. In the work [2] this phenomenon was interpreted as micelle formation. Increasing of the pluronic concentration results in the same effect. We found that at 25°C the micelle formation of pluronic F68 (new signal appearing) begins at ~ 15% (Fig. 2). In our previous work we have shown that solubilization of phthalocyanine 1 does not affect the conformational changes of pluronic at high concentrations as detected by 1H-NMR spectra [5].

![Fig. 2. 1H NMR spectra of F68 in D2O at various concentrations](image)

The other transition observed by 1H-NMR spectroscopy is gelation. This process leads to increasing the chemical shift and broadening of the CH2 PEO signal. These characteristic changes were similar to those described for pluronic P104 in the work [2].

Using UV-Vis spectroscopy we found that dye 1 was slightly solubilized in aqueous solution starting from concentration of the surfactant lower than 0.1%, and its colloidal solubility (max possible concentration in colloid solution) sharply increase at concentration of pluronic 0.5-1%.
This range of concentrations is similar to those determined by pyrene solubilization methodic – 0.4-0.8%; this transition was attributed to micelle formation [6].

Thus, different methods allow observing changes in two different intervals of surfactant concentration and they could correspond to two different steps of micelle formation.

In order to gain information about particle size in F68 solutions we obtained temperature and concentration dependence of this parameter by means of dynamic light scattering method. We found that transitions observed by NMR spectroscopy correspond to threefold increase of particles radius (from 5 to 15 nm), but it was not possible to detect changes in particle size at low concentrations. It might be due to high lability of first micelle form.

Summarizing all collected information, the hypothesis about phase transitions in pluronic water solutions and their interaction with the hydrophobic phthalocyanine was proposed (Fig. 3). There are not 2 but 4 transitions in pluronic system. The first transition we can observe by significant increase of solubility of hydrophobic compounds. This phenomenon is the evidence of formation of small, highly hydrated micelles. Other transitions do not influence solubilization and correspond only to rearrangement of pluronic molecules. The third transition corresponds to increase of particle size by formation of weak gel consistency of big spherical micelles. We can observe the dehydration and fixation of these hydrophobic cores by NMR spectroscopy. The last transition affects the big micelles’ crown and, in NMR spectra, we can see the final fixation of gel structure.

References

Fig. 3. The hypothesis about phase transitions in pluronic solutions
Spinus 2017, best poster presentation

Sergey Sokratilin

NMR relaxation and diffusion in aqueous solutions of fullerenols and fullerenes with PVP and dextrine complexes.

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Introduction

Currently, there is a wide range of methods for the study of human internal tissues. One of the most promising methods is magnetic resonance imaging (MRI). To improve the accuracy of diagnostics by MRI so called “contrast agents” are used. Salt-chelates are usually used as a contrast agent, in particular rare-earth metals of the lanthanide group.

Fullerene is a molecular carbon compound in the shape of a truncated icosahedron. Carbon atoms in the fullerene molecules are arranged at the vertices of regular hexagons and pentagons that make up the surface of a sphere or ellipsoid. All atoms are equivalent, as evidenced by the spectrum of $^{13}$C, which comprises only one spectral line. Fullerol is a molecular compound, which is a fullerene, modified by OH - hydroxyl groups, that provide good solubility [1-4].

Endohedral metallofullerenes (EMF) with greater efficiency also can be used as a contrast agent. As contrast agents, the endohedral metallofullerenes were suggested to increase the efficiency and sensitivity of the method [5].

In this work we investigate the aqueous solutions of endohedral fullerenols with Fe atoms and fullerenes with PVP (polyvinylpyrrolidone) and dextrine complexes. The investigated solutions were of different fullerene concentrations (10 mM/l; 6 mM/l; 4 mM/l; 2 mM/l). This work is a continuation of work on the study of Fe and Gd ions inside fullerenols [6]. The choice of the object studied, namely Fe ions in aqueous solutions of fullerenols, is explained by the relevance of the work [7] to find effective relaxant for MRI.

Results and discussion

We made a series of measurements of relaxation rates for three different sets of samples. The first series of samples contained fullerenols with Fe, the second and third series contained endohedral metallofullerenes with Fe ions and their complexes with PVP and Dextrine. A series of measurements was carried out on five different spectrometers with different proton resonance frequency of 20 MHz, 90 MHz, 300 MHz, 400 MHz, 500 MHz at the temperature $T = 25^\circ C$.

Temperature dependence at 500 MHz in the temperature range of 295-360$^\circ$K was also obtained.
The dependence of the diffusion coefficient on solution concentration (C mM/L), 500 MHz were obtained.

Part of the studies was carried out at the Research Park of Saint Petersburg State University: Centre for Magnetic Resonance.

Euromar 2017, Opening Address by Bernhard Blümich

Dear Colleagues and friends,

Welcome to EUROMAR 2017 on behalf of the AMPERE Society. Thanks to the perfect organization by Wiktor Koźmiński and his team, this is our time to discuss magnetic resonance, meet old friends again from Europe and all over the world and make new friends.

It is the mission of the AMPERE Society to 'contribute to the progress of radiofrequency spectroscopy, magnetic resonance and related phenomena'. Founded in 1951 and having just passed the age of 65 it is by no means time for the AMPERE Society to retire. The AMPERE tree is growing strongly with new branches developing, each one standing for an AMPERE event like a specialized colloquium or a summer school, routing in the strong stem, which represents the EUROMAR conference (Figure).

The AMPERE Society was a stronghold of science in EUROPE's troubled postwar past, forming a bridge between East and West across the iron curtain. Groundbreaking discoveries were reported at AMPERE events. For example, Granell, the collaborator of Peter Mansfield at the time, reported 'NMR diffraction' at the first Specialized Colloque AMPERE in Krakow in 1973. This work along with that of Lauterbur was subsequently recognized with the Nobel Prize to Mansfield and Lauterbur as the seminal discoveries that have given us Magnetic Resonance Imaging and dramatically changed medical diagnostics.

Peter Mansfield passed away earlier this year, and so did Erwin Hahn late last year, the discoverer of the spin echo, a basic ingredient of modern MRI methods. We commemorate their legacies in our society, with our friendship, and the advances in magnetic resonance reported at this meeting.

The bridges that science builds across threats, restrictions, fences, walls, and manipulatively false information are by no means an issue of the past. In closing I therefore want to cite the words that our dear colleague Paul Callaghan had drafted within minutes before his speech addressing the participants of another AMPERE event, the International Conference on Magnetic Resonance Microscopy, in the Great Hall of the People in Beijing in 2011:
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.”

Bernhard Blümich
July 2017

Report on Euromar 2017

Warsaw, Poland July 2-6 2017

Euromar 2017, the largest European congress on magnetic resonance, has been organized in Warsaw, the capital and the largest city of Poland, in the prestigious Marriott Hotel. Warsaw Marriott Hotel is one of the most known hotels in Warsaw where history still makes itself present. Euromar 2017 gathered almost 560 participants, who met to exchange knowledge and the results of scientific research in the field of magnetic resonance, ranging from physics and chemistry to biology and medicine. Over hundred talks were given, including 13 plenary, 3 tutorial, 42 invited, and 63 talks selected from abstracts. The lectures were organized in plenary and parallel sessions. Additionally, over 300 posters were presented. Except for the speeches and workshops taking place during Euromar 2017, a very effective industry exhibition was also held. Our sponsors and exhibitors presented their latest products and innovations. Social events and get-togethers provided a less formal atmosphere to get to know each other, to share the experience and ideas, and to establish long-term fruitful collaborations. The goal of the conference was to provide a stimulating forum for sharing experience, exchanging ideas, and establishing fruitful collaborations. The conference web page: at https://euromar2017.org/, is still available.

The conference started from opening and first prize session. The Raymond Andrew prize to Andrin Doll (see page 22) was presented by Beat Meier; Varian Young Investigator Award for Christopher Jaroniec was presented by Lucio Frydman; and Richard Ernst Prize for Robert Griffin was presented by Lucia Banci.

The second prize session was held at the conference closing. The Journal of Magnetic Resonance
in Chemistry three prizes, for young scientists. In addition there was a number of poster awards, founded by JEOL (3), International EPR Society (3), and Suraj Manrao (10).

The social events started from the conference mixer Sunday evening. Bruker has organized Bruker night at Endorfina restaurant Monday evening, and JEOL founded the reception Tuesday evening. At the end of the conference the Gala Dinner with live music has been held in the Palace of Culture and Science.

Despite the main scientific program there was a number of satellite events organized by NMR hardware vendors: Bruker, JEOL, Magritec. In addition, workshops of EuroBioNMR EEIG, which is a new coordinated group of European NMR Research infrastructures, and Europol-ITN, an EU H2020 funded Initial Training project, were held successfully.

The exhibition and sponsorships formed an integral part of the conference. It provided a unique opportunity to launch successful cooperation between conference participants and exhibitors due to its arrangements. We would like to thank the sponsors of the conference for their generous support. In particular to NMR spectrometer vendors, our platinum partner – BRUKER, and the gold partner JEOL, the bronze partners Magritek, Merck, and Mestrelab, and numerous exhibitors.

The photo gallery from the event is published at https://euromar2017.org/gallery.

We have every expectation that Euromar 2017 was a memorable event that harnessed ideas and forged professional partnerships.

On behalf of the Scientific and Local Organizing Committees and Euromar Board of Trustees we would like to thank to all Participants, Speakers, Sponsors and Exhibitors for their participation at the EUROMAR 2017 2-6 July 2017 in Warsaw, Poland. We do believe that it was a profitable experience both for scientific and networking approach.

Wiktor Koźmiński
Warsaw, August 2017
Pulsed electron paramagnetic resonance (EPR) is a versatile technique to study specific interactions within a system containing unpaired electron spins. One limitation of the technique is that only a fraction of the entire EPR spectrum can be excited with the monochromatic excitation pulses applied at microwave frequencies. Especially for transition metal ions, this excited fraction can be rather small. As a consequence of this limitation, sensitivity is restricted in a number of relevant EPR experiments. Moreover, multi-dimensional correlation techniques that are well established in nuclear magnetic resonance (NMR) spectroscopy are not commonplace in pulsed EPR.

In order to excite a larger fraction of the EPR spectrum, this work introduces frequency-swept microwave pulses for increasing excitation bandwidth in EPR spectroscopy. Such pulses became available only in recent years thanks to fast arbitrary waveform generators. The focus of this work is on technical aspects as well as on experimental aspects.

Technically, implementation of frequency-swept pulses required construction of dedicated experimental setups. In particular, commercial X- and Q-band spectrometers were extended with incoherent frequency-swept pulses and a dedicated spectrometer providing competitive sensitivity with phase-coherent pulses at either X- or Q-band frequencies was constructed [1]. An important technical restriction when exciting electron spins with frequency-swept pulses is the limited bandwidth of the microwave resonator that is used to couple to the spins. Accordingly, a way to compensate such technical bandwidth limitations by adaptation of the pulse’s frequency sweep based on the adiabaticity criterion was developed [2]. This approach could be checked for self-consistency based on linear response theory [3] and the procedure was extended to pulses optimized for frequency-selective excitation [4]. Experimentally, these pulses were incorporated into a number of existing EPR pulse sequences. Significant sensitivity enhancement of distance measurements between a pair of electron spins was achieved by replacing the incoherent pump pulse in the four-pulse double electron-electron resonance (DEER) pulse sequence by an incoherent frequency-swept pulse. In particular, a three-fold improvement in X-band DEER modulation depth to 24% when pumping a spectrally broad Cu(II) center with a frequency-swept pulse instead of a monochromatic pulse was achieved [2]. For distance measurements between a pair of Gd(III) centers bearing a high spin of \( S = 7/2 \), similar modulation depths could be achieved at Q band by using two consecutive pulses that pump separate frequency windows [5].

Due to the multiple energy levels of the \( S = 7/2 \) spin state, coherence and population transfers within the spin multiplet were considered experimentally and with spin dynamics simulations [5–8]. In particular, an unwanted coherence transfer due to the pump pulse influenced DEER sensitivity and stimulated the development of a simple and fast procedure to optimize pulse parameters in situ [5]. With respect to population transfer, polarization from unobserved transitions could be transferred to the observed transition by means of frequency-swept pre-polarization pulses [6]. The resulting signal enhancement was up to a factor of three and allowed to determine Gd-Gd distances up to 8.6 nm by pre-polarized four-pulse DEER.

Frequency-swept pump pulses were also examined in systems containing three spins, where the large DEER modulation depth led to a more pronounced signature of three-spin correlations and alleviated its extraction from experimental data [9].

A general limitation when using frequency-swept pulses in DEER were interference effects related to the extended pulse duration, which could deteriorate data quality at short inter-spin distances. Accordingly, a novel interference-free pulse sequence (abbreviated as CIDME) that is suited for short distances was developed and verified by a Gd-Gd spacer with 2.1 nm inter-spin distance [10].

The home-built spectrometer allowed to perform correlation experiments by computing the Fourier-transform (FT) of transient spin echo signals. Thanks to the uniform excitation profile achieved with bandwidth-compensated pulses, the main impact of limited resonator bandwidth on FT spectra was only due to the detection process [3]. Accordingly, the net bandwidth of the FT EPR experiment was enhanced as compared to experiments performed with uncompensated pulses. As a result, FT EPR spectra ranging over several hundreds of MHz could be recorded. Three different types of two-dimensional correlation experiments were implemented, each correlating the FT EPR spectrum to either (i) longitudinal relaxation \( T_1 \), (ii) nuclear modulation, or (iii) dipolar modulation. Correspondence between FT EPR-correlated inversion recovery kinetics of nitroxides and the same kinetics observed with selective pulses required minimization of cross talk in FT EPR spectra due to proton modulations. In particular, the pronounced proton modulations at X-band...
frequencies induced changes on the order of $\pm 10\%$ in apparent relaxation times [3]. Furthermore, inversion recovery initiated by an adiabatic inversion pulse that inverts the entire spectrum reduced contributions of spectral diffusion between inverted and non-inverted spins. Consequently, longer relaxation times were observed upon an adiabatic inversion pulse than upon a monochromatic inversion pulse [2].

Nuclear modulations correlated to the EPR spectrum were studied at X and Q band for nitroxides and at X band for an oriented bispicolinate Cu(II) complex. Nitroxides at Q band revealed the proton combination frequency around 100 MHz, whereas the Cu(II) complex showed fundamental nuclear modulation frequencies up to 200 MHz related to the enriched $^{63}$Cu nucleus [11]. Such fast modulation frequencies could not be excited efficiently with monochromatic pulses due to limited excitation bandwidth.

Dipolar modulations correlated to the EPR spectrum were recorded at Q band using nitrooxide spin pairs [12]. The uniform excitation pulses were incorporated into an existing four-pulse sequence that refocuses the dipolar coupling by a solid echo (abbreviated as SIFTER) in a way that allowed to obtain FT EPR-correlated dipolar spectra. The resultant correlation pattern confirmed the expectation based on the inter-spin geometry of the spin pair. Previously, EPR-correlated dipolar spectra of nitroxides were not accessible by FT EPR techniques, especially not at Q band.

Here you find the direct download link to the entire thesis at the e-library of ETH Zurich

BIBLIOGRAPHY

On-chip capacitors for MACS NMR detectors

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Magic angle coil spinning (MACS), a highly sensitive, high resolution technique [1], features a micro resonator, inductively coupled to the probe coil, spinning simultaneously with the sample at the magic angle. A microcoil and a capacitor form the crux of these MACS resonators, which are carefully designed to resonate at the Larmor frequency of interest of a particular experiment. The first MACS detectors [1] employed hand-wound microcoils and soldered discrete capacitors. While the electrical characteristics of such a micro resonator have proven suitable for high performance NMR spectroscopy, there were obvious issues associated with the tediousness, robustness and reproducibility of manual manufacturing of microcoils, as well as with the mechanical balance of the entire device after soldering the capacitors. As an improvement to the fabrication process, micro fabrication techniques have been employed [2] to obtain MACS detectors with a higher degree of reproducibility. The pick-up coil of the micro resonator has been defined by automatic wire bonding [3], and the on-chip capacitors have been fabricated by a combination of various micro fabrication techniques such as lithography, etching and electroplating.

A discussion on the on-chip capacitors that have been integrated with the microcoil is presented. To begin with, the RF simulation of the quality factor offered by the two capacitor designs under discussion, namely, the interdigitated capacitor and the parallel plate capacitor shown in Figures 1a and 1b, has been performed. The results from the simulation in Figure 1c have been followed up with electrical characterization of the capacitors with respect to their phase angle as shown in the Figure 1d. Further, within the MACS arrangement, due to the relative motion between the metal and the magnetic field, eddy currents are induced in the coil and the capacitor which leads to the generation of heat and increases the sample temperature. Sample heating during spinning of the MACS detector degrades the spectral resolution in NMR experiments. The simulation results in Figure 2 shows that temperature gradient being developed across the on-chip capacitors is dependent on the thickness of the capacitor structures and spinning speed. The sample temperature rise has been evaluated for a 8 µm thick interdigitated capacitor structure using ethylene glycol [4] as an NMR temperature sensitive sample as shown in the Figure 2a and b. Therefore, it is concluded that the parallel plate capacitor is a better alternative to the interdigitated capacitor because it offers a better quality factor even at lower thicknesses which is vital in reducing the sample heating due to induced eddy currents.

Fig. 1 a) MACS detector (24 turn coil) with interdigitated capacitor; b) with parallel plate capacitor; c) RF simulation of the Q factor for interdigitated and parallel plate capacitor designs; d) Measurement of the phase angle, φ of the capacitor structures. \( Q = 1 / \cos \phi \).

Fig. 2 Simulation of the temperature gradient across the capacitor at different thicknesses and spinning speeds. Insets: a) Temperature increase in the sample at various spinning speeds measured using b) chemical shift difference in ethylene glycol.

Trimethylsilyl tag for the characterisation of protein-ligand interactions by NMR

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²University of Graz, Institute of Chemistry, Austria
³Monash Institute of Pharmaceutical Sciences, Monash University, Parkville, VIC 3052, Australia

Fragment-based drug discovery (FBDD) is a widely used approach in pharmaceutical and biotechnological companies. Nuclear magnetic resonance (NMR) spectroscopy is an important experimental method in FBDD, as it enables the screening of large compound libraries to identify weak binders in a high throughput manner. Limitations arise from requirements in protein size, amount and availability of isotopic labelling, especially in the absence of structural information.

Here we present a new tool for measurement of ligand binding affinity and characterisation of ligand binding site. The approach is based on a small molecule tag, trimethylsilyl methyl 2-iodoacetate (TMS), that reacts with the thiol group of a solvent-exposed cysteine residue. The TMS tag generates a narrow and intense singlet resonance in the 1D ¹H-NMR spectrum that can easily be detected without any isotope labelling. The chemical shift of the signal is near 0 ppm, where there are very few protein resonances. To demonstrate the potential of the TMS tag we used the Zika virus protease NS2B-NS3¹ and the human prolyl isomerase FK506 binding protein (FKBP)². Both proteins are established drug targets. In the case of NS2B-NS3 we mutated the natural cysteine residues (C80, C143) to serine and substituted V36 near the active site by a cysteine. In FKBP we mutated the natural cysteine residue (C22) to serine and mutated R18 to cysteine. Following ligation of the TMS probe to the cysteine residues we acquired 1D ¹H-NMR spectra with and without inhibitors. Changes in lineshape and chemical shift of the TMS signal upon titration of the Zika virus protease with an inhibitor (4-nitrophenyl 4-guanidinobenzoate hydrochloride) revealed a dissociation constant $K_d$ of about 200 µM. The same $K_d$ value was measured for TMS tags attached at different sites. The ligand-induced chemical shift changes in the TMS signal decreased in size with increasing distance of the TMS group from the active site. The FKBP inhibitor 4-hydroxy-N-(4-hydroxyphenyl)benzamide showed a $K_d$ value of about 165 µM. In both protein targets, the TMS signal provided very clear evidence for fast or slow ligand exchange. In the case of slow exchange, integration of the TMS signals in the free and ligand-bound protein was greatly facilitated by its location in a spectral region with few protein resonances.

In conclusion, the TMS tag provides an inexpensive and sensitive tool to assess ligand binding in target proteins without any isotope labelling using straightforward 1D ¹H-NMR spectra. As far as the chemical shift perturbation of the TMS signal is related to the distance from the ligand binding site, the tag also provides information about the approximate location of the ligand on the protein.

The continuing development of magnetic resonance theory and related fields allowed for the exploration and explanation of many challenging molecular systems in physics, chemistry and medicine. The theoretical description of a magnetic resonance experiment is often accompanied by computer simulations that are matched against the experimental outcome. Computer simulations are therefore an important tool in getting a better understanding of the system and in extracting as much useful information as possible. To this end several software packages are being developed that are able to simulate Nuclear Magnetic Resonance (NMR) and Electron Paramagnetic Resonance (EPR) experiments [1, 2].

We would like to present SpinDynamica, a simulation package that is heavily used within our group and others, employed as an important tool in the development of spin dynamical concepts [3, 4, 5, 6]. SpinDynamica is built around the core structure of Mathematica. This makes it useful for numerical but even more so for analytical calculations of magnetic resonance problems. SpinDynamica is subdivided into several lower level and higher level routines. Lower level routines include the possibility of constructing a personalised spin or operator bases, generation of the corresponding spin operator/superoperator representations, construction of relaxation superoperators and many helpful tools for the analysis and visualisation of Euler rotations. Spin angular momentum operators are internally stored as matrices, but the default output is symbolic, resembling standard conventions found in several NMR textbooks. This allows for a very natural interaction with the simulation environment and calculations that are usually performed on paper can be done within SpinDynamica. Higher level routines allow for the simulation of solution and solid-state 1D spectra and calculations of evolution trajectories of a given set of spin observables. We are currently working on releasing a new version of SpinDynamica, in which we hope to improve its numerical efficiency and to include the calculation of 2D spectra and chemical exchange effects.

Fig. 1 Optimisation and analysis of the $S_{2hM}$ pulse sequence using SpinDynamica. The optimum number of repetitions (A) and inter pulse delay (B) leading to maximal Singlet polarisation ($P_s$) were determined and then compared to the experimental results. The evolution of the singlet polarisation can be visualised by following the $x$, $y$ and $z$ components of the fictitious spin operators ($Q_i$) in the appropriate subspace as shown in C and D.
Minutes of the Meeting of the Ampere Bureau
in Warsaw, Poland, on July 4, 2017

Members Present:
B. Blümich, B. Meier, G. Jeschke, L. Frydman, S. Jurga, M. Ernst, A. Böckmann, C. Redfield

Excused:
J. Dolinšek, J. van Duynhoven, S. van Doorslaer

Agenda:
1. Approval of the minutes of the AMPERE Bureau meeting in Zürich
   March 16, 2017
3. Financial flow between EUROMAR and AMPERE
4. General rules for funding of AMPERE events
5. Preparation of the election of AMPERE Committee members
   by the General Assembly
6. Varia

At 13:00 hours G. Jeschke opened the meeting.

Ad 1. The minutes of the AMPERE Bureau meeting in Zürich, March 16, 2017, published in the AMPERE Bulletin 276, were approved unanimously.

Ad 2. G. Jeschke explained the current status of the finances of AMPERE (see page 34). The finances are stable and the Groupement AMPERE has a fortune of about CHF 50'000.- plus the endowment for the Andrew prize (120'000.- CHF). The rest of the money is owned by the subdivisions and only they can decide on the use of their money which is administered by AMPERE. The financial report will be presented to the AMPERE Committee and the General Assembly has to approve it. There was a discussion whether we should have a committee that audits the accounts before the report to the General Assembly. No decision was taken about this topic.

Ad 3. L. Frydman announced that Thomas Prisner (Frankfurt) was elected as the new chair of the EUROMAR Board of Trustees and will be a member of the AMPERE Bureau starting September 1.

A large part of the membership fees is generated through EUROMAR with about 600-700 participants. In the past years, about 10000.- Euro membership fees were generated every year through this. In return, AMPERE has provided student travel stipends (5000.- Euro) and sponsored one of the tutorial lectures (2000.- Euro) during the past EUROMAR conferences. The EUROMAR Board would like to have an earlier decision on the financial contributions of AMPERE to the yearly conference to make planning easier. The AMPERE Bureau decided to decide on the contribution for the next three conferences and leave it at the level that is described above. To reduce bank charges, the different contributions are often offset against each other but it is important to make the money flow transparent to the EUROMAR treasurer. It was also suggested to make it clearer to the participants that they become members of AMPERE and that this entitles them to discounts at various conferences.

Ad 4. In the last meeting in Zürich, the AMPERE Bureau decided to have general rules for a more even distribution of support to the various events of the subdivisions. B. Blümich and B.H. Meier proposed guidelines for the distribution of funds which were accepted with some changes (see ampere-society.org). For 2018, there will be a budget of 20000.- Euro with 7000.- Euro given to EUROMAR which leaves 13000.- Euro for distribution by the prize committee to other events. The new rules will be sent to the subdivision chairs. Proposals should be submitted by January 1, but if money is available, later proposals will also be considered.

Ad 5. At the General Assembly, we should propose a list of candidates for election to the AMPERE Committee. It was decided to propose a list with five candidates (Robert Konrat (AT), Ferenc Simon (HU), Georgios Spyroulias (GR), Eurico Cabrita (ES), and Daniel Topgaard (SE)) from countries that are currently not represented in the AMPERE Committee. This proposal will also be discussed in the AMPERE Committee meeting. There was a discussion how one could involve the national MR societies more in the activities of AMPERE and whether we should try to get the heads of the national societies into the AMPERE Committee. There was no consensus about this topic and it needs more discussion in the AMPERE Bureau before the next EUROMAR where new additions to the AMPERE Committee are required.

Ad 6. Varia: There were no topics to discuss under varia.

At 14:30 hours G. Jeschke closed the meeting and thanked all the present members for their time and effort.

Zürich, 4.7.2017
Matthias Ernst
### Balance of the Accounts of the Groupement Ampere and the Subdivisions

Period from March 15, 2016 to June 15, 2017

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<td>Prizes and Travel Grants</td>
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<td>Charges, Handling Costs (Deposits, Bank)</td>
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| Total fortune on June 15, 2017 | 223'065.47 | 32'687.93 | 1'524.39 | 46'126.88 |

### Balance March 15, 2016

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Minutes of the Meeting of the Ampere Committee
in Warsaw, Poland, on July 4, 2017

Members Present:

Excused:
J. Dolinšek, R. Boelens

Agenda:
1. Approval of the Agenda
2. Approval of the minutes of the AMPERE Committee meeting in Zurich, July 2, 2014
3. Financial report of the secretary general (G. Jeschke)
4. Preparation of the election of AMPERE Committee members by the General Assembly
5. General rules for funding of AMPERE events
6. Varia

At 19:00 hours G. Jeschke opened the meeting.

Ad 1. The agenda was approved unanimously.

Ad 2. The minutes of the AMPERE Committee meeting in Aarhus, July 5, 2016, published in the AMPERE Bulletin 263/264, were approved unanimously.

Ad 3. G. Jeschke gave an overview (page 34) over the finances of the Groupement AMPERE. The finances are good with little changes in the total amount since last year. The income is mostly from membership fees (about 50% from regular membership fees and 50% from conference attendance). The expenses are mostly the student travel grants for conferences and on the administrative side the printing of the Bulletin.

Ad 4. The AMPERE Bureau proposes the election of five additional members (Robert Konrat (AT), Ferenc Simon (HU), Georgios Spyroulias (GR), Eurico Cabrita (ES), and Daniel Topgaard (SE)) from countries that are currently not represented to the AMPERE Committee. There are currently 14 AMPERE Committee members (plus the members of the AMPERE Bureau) which were elected for a four year term at the last General Assembly. It is important to add new members on a yearly basis to ensure continuity in the Committee. G. Jeschke will contact them and check whether they are willing to stand for election at the General Assembly. The AMPERE Committee will be quite diverse with respect to countries and research fields represented.

Ad 5. The membership fees collected from Euromar represent a large block in the income of AMPERE. The past few years, this was about 11000.- Euro per year. On the other hand, AMPERE will finance student travel stipends (5000.- Euro) and a tutorial lecture (2000.- Euro) at the Euromar for the next three years.

Ad 6. The AMPERE Bureau decided to introduce new funding rules for AMPERE events and schools (see attachment). There will be an annual budget of 20000.- Euro for the support of events. From this amount 7000.- Euro are assigned for support of Euromar while the rest can be distributed to other events. To obtain support, a proposal has to be submitted to the AMPERE prize committee before January 1st.

Ad 7. Varia: Geoffrey Bodenhausen suggested to ask the organizers of the Chamonix meeting whether they are interested in a closer collaboration with AMPERE and would like to become a subdivision. There were some talks at the start of the Chamonix meeting series but then the organizers showed no interest. The president, B. Blümich, will discuss this issue with the new organizers of the Alpine Conference represented by Jean-Nicolas Dumez.

At 19:45 hours G. Jeschke closed the meeting and thanked all the present Committee members for their time and effort.

Warsaw, 4.7.2017
Matthias Ernst
Minutes of the Meeting of the General Assembly of the Groupement Ampere
in Warsaw, Poland, on July 5, 2017

Agenda:
1. Approval of the minutes of the AMPERE General Assembly in Aarhus, July 6, 2016
2. Financial report of the secretary general (G. Jeschke)
3. Election of the AMPERE Committee members
4. Varia

At 18:40 hours G. Jeschke opened the meeting.

Ad 1. The minutes of the AMPERE General Assembly in Aarhus, July 6, 2016, published in the AMPERE Bulletin 263/264, were approved unanimously.

Ad 2. G. Jeschke explained the finances of the Groupement AMPERE (page 34). The finances are stable with a slight increase in the fortune of the society. The biggest costs are the support of conferences through student travel grants (Euromar and AMPERE School in Zakopane) and the expenses for printing the Bulletin. The assembly approved the financial report unanimously with no abstentions.

Ad 3. The AMPERE Bureau and the AMPERE Committee propose four additional members for the AMPERE Committee from countries which are currently not represented (Robert Konrat (AT), Simon Ferenc (HU), Georgios Spyroulias (GR), and Daniel Topgaard (SE)) for a four year term. The new members of the AMPERE Committee were elected unanimously with no abstentions.

Ad 4. There were no points discussed under varia.

At 19:00 hours G. Jeschke closed the meeting and thanked all the present AMPERE members for their time and effort.

Warsaw, 5.7.2017
Matthias Ernst

Executive Officers and Honorary Members of the AMPERE Bureau

The AMPERE BUREAU includes the executive officers (which take the responsibility and the representation of the Groupement between the meeting of the committee), the honorary members of the Bureau and the organizers of forthcoming meetings.

Executive Officers 2016 - 2019

<table>
<thead>
<tr>
<th>Position</th>
<th>Name</th>
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<tbody>
<tr>
<td>President</td>
<td>Bernhard Blümich</td>
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<tr>
<td>Vice Presidents</td>
<td>Janez Dolinšek</td>
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<td>Anja Böckmann</td>
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<tr>
<td>Secretary General</td>
<td>Gunnar Jeschke</td>
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<td>Matthias Ernst</td>
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<td>EF-EPR Representative</td>
<td>Sabine van Doorslaer</td>
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<td>SRMR Representative</td>
<td>Melanie M. Britton</td>
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<td>MRPM Representative</td>
<td>Yi-Qiao Song</td>
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<td>MR-FOOD Representative</td>
<td>John van Duynhoven</td>
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<td>Hyperpolarisation Representative</td>
<td>Geoffrey Bodenhausen</td>
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<td>Lucio Frydman</td>
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<td>EUROMAR Treasurer</td>
<td>Christina Redfield</td>
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<td>Past President</td>
<td>Beat Meier</td>
</tr>
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<td>Honorary Member</td>
<td>Hans Wolfgang Spiess</td>
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<tr>
<td>Honorary Member</td>
<td>Stefan Jurga</td>
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</table>
Emeritus members

L. KIMTYS
Department of Physics, Vilnius University, Universiteto Str. 3, VILNIUS 2734, Lithuania

F. MILIA
NRC Demokritos, Physics Department, Aghia Paraskevi Attikis, GR - 15310 ATHENS, Greece
J. HENNEL, Inst. of Nucl. Phys. Uni. Radzikowskiego 152, PL - 31342 KRAKOW 23, Poland

Honorary members

R.R. ERNST
Laboratorium für Physikalische Chemie, ETH Zürich, CH-8093 ZURICH, Switzerland

J. JEENER
Université Libre - Plaine, CP 223, Blvd. du Triomphe, B - 1050 BRUXELLES, Belgium

K.A. MÜLLER
IBM Zurich Research Laboratory, Säumerstrasse 4, CH - 8803 RÜSCHLIKON, Switzerland

K. WUETHRICH
Inst. f. Molekularbiologie u. Biophysik, ETH Zürich, CH-8093 ZURICH, Switzerland

Guest members

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J.A. NORRIS
Dept. of Chemistry, University of Chicago, South Ellis Ave. CHICAGO IL 6037-1403, USA
Delegate of the International EPR Society

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C.P. SLICHTER
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Future conferences

Ampere Events 2017

10th Alpine Conference on Solid-State NMR
Chamonix Mont Blanc (France)
September 10-14 2017

Other Events 2017

SciX2017
Reno (USA)
October 8-13 2017

Ampere Events 2018

MRPM14 Magnetic Resonance in Porous Media
Gainesville (USA)
February 18-22 2018

Euromar 2018
Nantes (France)
July 1-5 2018

6th Symposium on Hyperpolarization
Southampton (UK)
September 2-6 2018

Ampere Biological Solid-State NMR School
Palma de Mallorca (Spain)
October 21-26 2018

FoodMR 2018
Brittany (France) 2018

Other Events 2018

SciX2018
Atlanta (USA)
October 21-26 2018

Ampere Events 2019

ISMAR / Euromar 2019
Berlin (Germany)
August 25-30 2019