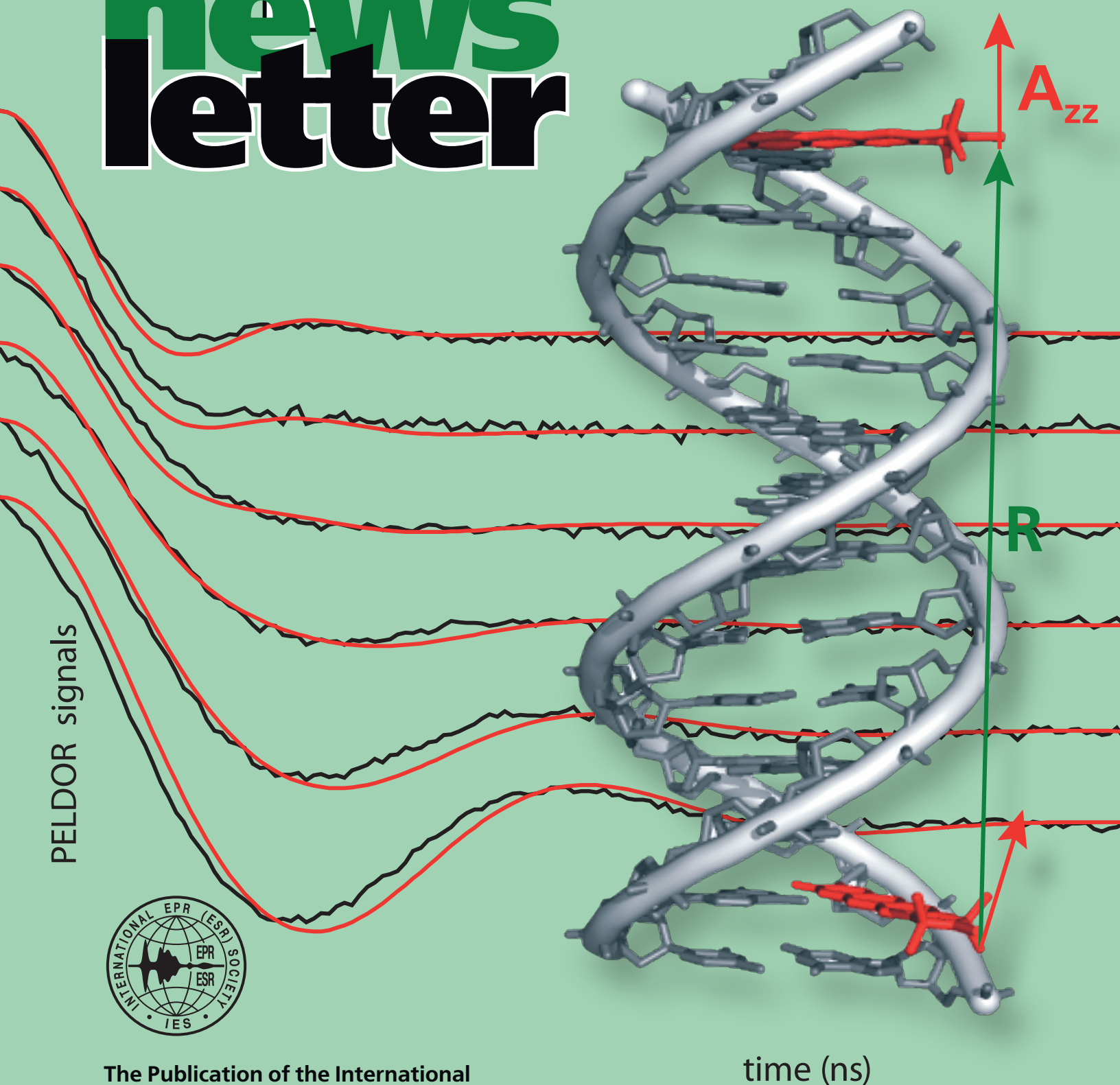


# epr news letter

2012  
volume 22 number 2



The Publication of the International  
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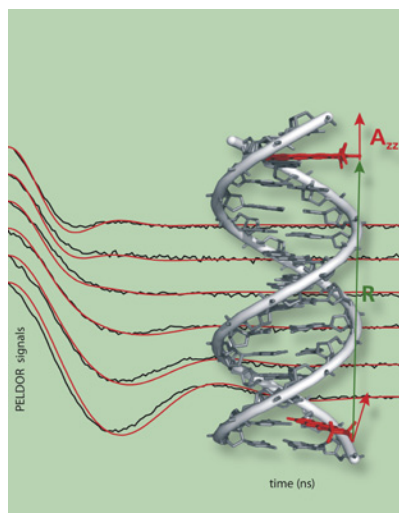
Please feel free to contact us with items (news, notices, technical notes, and comments) or ideas for the *EPR newsletter*.

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The cover picture illustrates aspects of research carried out by Thomas Prisner, recipient of the Bruker Prize 2011. It shows a series of orientation selective PELDOR experiments (with variable observer frequency) to unravel the conformational dynamics of double stranded DNA molecules. This was possible with a new type of spin label developed and synthesized by Prof. Sigurdsson (University of Iceland) which is rigidly incorporated into double stranded DNA molecules. The quantitative analysis of the PELDOR data obtained at X-band and G-band frequencies are consistent with a correlated twist-stretch motion of such DNA molecules. This work was published in *J. Am. Chem. Soc.*, 133 (34), 13375-13379 (2011) DOI: 10.1021/ja201244u.

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volume 22 number 2 2012

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Swiss Federal Institute of Technology Zurich



It is with deepest grief that we have to inform you that Professor Seigo Yamauchi, President of the IES, has passed away on September 26, 2012, due to a sudden illness. An obituary will follow in a forthcoming issue. We express our deepest sympathy to his family.

## Editorial

Dear colleagues,

I still cannot believe that Seigo-san has left this world. Just several days before his sudden death I corresponded with him. Seigo-san sent me his IES President's congratulations letter for the Zavoisky Awardee 2012, Prof. Richard Fessenden, to be announced during the Zavoisky Award ceremony. Within my mind I can visualize Seigo-san full of life and energy during his visit to Kazan for his Zavoisky Award 2011 and his noble reticence and dignity with which he received this highly deserved award. I was looking forward to meeting with him at the APES2012 conference in Beijing and discussing with him the EPR newsletter-related matters. But with sorrow I participated there in a session devoted to the commemoration of Seigo san. How come... life is so unpredictable...

Many magnetic resonance researchers all over the world collaborated with Seigo-san and could contribute to the understanding of his impact on the development of magnetic resonance to be featured in an obituary in a forthcoming issue of the newsletter. As for me, in the Autumn of 2002 I visited Seigo-san at the Tohoku University in Sendai to help him

with finalizing a special issue, "Spin Chemistry in Japan", of the journal *Applied Magnetic Resonance* (vol. 23, nos. 3-4, 2003 which is also available online at [link.springer.com/journal/723/23/3/page/1](http://link.springer.com/journal/723/23/3/page/1)). I enjoyed working with Seigo-san on the preparation of this special issue. A highly intelligent and modest man, he worked hard and was extremely exacting as to the quality of the submitted manuscripts.

I regret so much that we somewhat delayed the final touches to this issue and Seigo-san did not have an opportunity to read it. Seigo-san liked the *EPR newsletter* and highly valued its role in the life of the international magnetic resonance community. I feel he would have enjoyed this issue as well. It's my feeling that it is nicely balanced with the instructive material and information on a wider scope.

It was helpful that Bernard Gallez and Karsten Mäder contributed their success stories to the "IES Young Investigator Award Revisited" column (pp. 4-7). For sure, the young generation of the EPR researchers will be inspired by their input. It might be also good to reread previous relevant articles by Mark Newton (16/2-3, p. 8), Alex Smirnov (17/1, p. 4), Gunnar Jeschke (17/2-3, pp. 8-9), Eli Shkrob (18/1, p. 6), and Devkumar Mustafi (19/1-2, p. 22). Hopefully these articles will

The Executive has appointed new (acting) Presidents in place for the remainder of the current term 2012-2014, as follows:

**Professor Hitoshi Ohta:**

October 1, 2012 until June 30, 2013

**Professor Lawrence Berliner:**

July 1, 2013 until March 31, 2014

**Professor Klaus Möbius:**

April 1, 2014 until December 31, 2014

Please take note of this information for future reference. Accordingly, please send the nomination for the awards of IES for 2013 to Professor Hitoshi Ohta.

Updated information can be found at the IES website [www.ieprs.org](http://www.ieprs.org).

also stimulate other IES Young Investigator Awardees to contribute to this column. The young generation could also benefit from the good advice of Robert Kaptein given in his interview on the occasion of his Voevodsky Award 2012 (p. 3): "Follow your heart and don't be too much concerned with what you think may be good for your career", which is completely in line with the advice of Jim Bolton given in his interview on the occasion of his 75th birthday (pp. 10-11): "Follow your passion wherever it may take you. Be curious and alert for the unusual". Our congratulations to Robert and Jim!

Georg Gescheidt's didactic and flavorful adventure described in the "Another Passion" column (pp. 8-9) is a confirmation that men are the best cooks in the world and that physical chemists are the best cooks among men. Stephen Hill et al. did a good job adding a new facet to tips and advice on sample preparation (pp. 12-14). Keith Earle consequently deals with this topic in the "Tips and Techniques" column he edits together with David Budil. And of course, Malcolm Forbes' letter (pp. 14-15) telling about the activities of the Inter-American Photochemical Society is certainly of interest for our readership. Thanks a lot to all contributors to this issue.

Please note that the report about the IES Annual General Meeting 2012 will be published in the forthcoming issue *EPR newsletter* 22/3.

Laila Mosina

PS I look at this photo of Seigo-san picturing him with a soft smile on his lips and a glance into the infinite blue of the sky high over there. What does he see in the depths of the upper spheres?

# Robert Kaptein:

## An Interview on the Occasion of His Voevodsky Award 2012



EPR newsletter: *Dear Professor Kaptein, on behalf of the readers of the EPR newsletter we congratulate you on your Voevodsky Award 2012. We are most appreciative that you agreed to answer the questions of this interview. Why did you start towards your career in science?*

I have always been curious about how nature works. My father played a big role in this. He was a high-school chemistry teacher and he taught me chemistry in his class. He stimulated my interest in science.

*Who introduced you into magnetic resonance?*

It was not really a conscious decision to work in NMR. After my Bachelors exam in chemistry at the University of Leiden I was looking for a subject for my Masters that combined theory and experimental work. In the department of Prof. L.J.Oosterhoff there happened to be room for a student in the NMR group. I did not know what NMR was at that

time, but I happened to like it and stayed in this area all my life. I believe that although you may not know exactly what project you choose, you can still make it a success.

*What part of your research is most dear to your heart and why?*

Although there are several highlights in my work the most gratifying period for me was my PhD research on CIDNP. After the first publications on the discovery of the CIDNP phenomenon in 1967 it remained a mystery for about two years. To find the explanation in terms of the radical pair mechanism was a real "eureka" moment that I will never forget.

*What is the driving for you in your research?*

To understand nature is still my main motivation. My change to more biological applications of NMR in the 80's was also inspired by this. But science is also a social activity. To

be part of a large international scientific community and have many good friends there is an enjoyable aspect of working in research.

*What would you have done differently given the chance?*

With hindsight there are always many things that one might have done differently. However, I don't believe it is very useful to contemplate too much on this.

*What is your message to the younger generation of the magnetic resonance researchers?*

To young researchers I would say: follow your heart and don't be too much concerned with what you think may be good for your career. There is still so much to do in magnetic resonance. Pick a good problem and work hard to solve it. Your future will be bright!

### The Zavoisky Award 2012

**Richard W. Fessenden**

University Notre Dame, Notre Dame IN, USA

in recognition of a lifetime's work in magnetic resonance and, in particular, the laureate's fundamental studies of short-lived free radicals produced in liquids by high energy radiation

### Foreign Members of the Academy of Sciences of the Republic of Tatarstan

**Wolfgang Lubitz and Klaus Möbius** have been elected Foreign Members of the Academy of Sciences of the Republic of Tatarstan in recognition of their long-lasting and fruitful scientific cooperation with the academic institutions of Kazan, in particular with the EPR groups at the Zavoisky Physical-Technical Institute and the Kazan Federal University. In the official letter of the President of the Academy of Sciences of the Republic of Tatarstan, Professor Akhmet M. Mazgarov, the hope is expressed that this election will give a new impetus to the scientific and cultural collaboration between the Republic of Tatarstan and Germany.



Are you interested to become a member of the International EPR (ESR) Society? Please find the registration/information form for new/continuing members of the IES and non-credit-card payment instructions for individual members on this Web site: [www.epr-newsletter.ethz.ch/contact.html](http://www.epr-newsletter.ethz.ch/contact.html)

## Bernard Gallez

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I was asked several times to contribute to this “IES Young Investigator Award Revisited” column... And of course, although I like very much reading the *EPR Newsletter*, I found always a good (or bad...) reason to escape to my duties (grant period, exams period,...). But finally I am right now in front of my PC and I should start to fill this white page. An interesting, difficult and presumptuous task: “talking about myself” to the EPR community (a kind of psychoanalysis). A statement twelve years after receiving this award together with my friend Karsten Mäder for our contribution to the development of EPR applications in the field of Pharmacy and Pharmacology. It looks like an evaluation at the mid-term of my career (Where do I come from? Where am I going to?...).

How did I start into this EPR business? Well, by accident! I am a pharmacist and radiopharmacist by training. And, of course, I never heard about EPR during my education. Due to serious health problems during my childhood, I became enthusiastic about medical imaging and emerging non-invasive diagnostic procedures. When I started in 1988 a PhD thesis on the targeting of MRI contrast agents, it was impossible to anticipate that “Molecular Imaging” (this term did not exist at that time) would become so important and would revolutionize the diagnosis and the individualization of treatments. In brief, during my PhD, I was designing, synthesizing and characterizing nitroxides targeted to several different receptors present at the surface of the hepatocytes to build new MRI contrast agents. To understand the interesting relaxing NMR properties of my contrast agents, it was suggested that I use

this obscure technique called EPR. And I started molecular dynamic studies on a very old EPR system (without a PC associated, with only the trace slowly made by the ink pen). I worked together with a real EPR spectroscopist, René Debuyst who trained me in the field. At that time, I was (and I persist to be) rather concerned with the length of the equations and the inaccessible language commonly used by EPR scientists. However, after transformation into an operational tool, I started to think that EPR was an outstanding and powerful method that should become more popular in the biomedical community.

In 1993, I joined the laboratory of Hal Swartz at Dartmouth. It was a fantastic period, with outstanding post-docs (most of them created their own EPR centers later) with a profusion of ideas and many new applications in physiology, physiopathology, pharmacology, pharmaceutical technology, radiation biology, and toxicology. I discovered in vivo EPR and EPR oximetry, areas that I have continuously pursued during my research career.

Back in Belgium, I started to build a laboratory on two pillars: high-field MRI and EPR, including in vivo EPR and EPR Imaging (and clinical EPR will become available very soon!). The research areas that we are pursuing since that time mainly involve the characterization of the tumor microenvironment (oxygenation, hemodynamics, metabolism) in oncology. We are trying to develop innovative tools (EPR and MRI) to measure these crucial physiological parameters and new pharmacological interventions to change the tumor microenvironment in order to optimize anti-cancer treatments. Besides these

main topics, we are also working on other topics such as redox status, extracellular pH, dosimetry, etc... where, again, there is a clear synergy between MRI and EPR.

I certainly do not consider myself an EPR scientist. Only, in other scientific communities (MRI, pharmacy, oncology), other people are considering me an “EPR guy” (often meaning “he is doing strange things that we cannot understand”). From this permanent contact especially with the MRI community, we can learn a lot about the success/failure of other techniques. MRI became one of the most powerful imaging modalities, not only because of its intrinsic features and the extraordinary developments made by imaging physicists and engineers, but certainly because it was rendered popular to the medical community who can use it every day with minimal knowledge in MRI physics. I strongly believe that EPR is a treasure that should not be kept only in the hands of the scientists who are “initiated” into the EPR secret, but that the expansion of the field will strongly depend on our ability to render EPR as simple as possible for biomedical scientists. Over the years, the EPR group has grown in Brussels (presently, around 15 docs and post-docs). Besides our own developments, I am really happy that we succeeded in attracting external outstanding scientists who came to our lab because in vivo EPR was the only method able to solve their question. They did not know or did not care about the beauty of EPR physics, but simply acknowledged the unique capability of the EPR method. What is true for oximetry is certainly valid for many other areas like spin trapping, spin labeling, etc. We have some-  
how the responsibility of this development

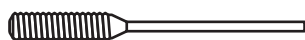
in our way to present ourselves outside, and in our capacity to develop and accept new ideas that will push the boundaries of EPR spectroscopy and imaging.

The fact that we are asked to write such a paper means that we are no more young investigators... and my final words will be for the new generation of young investigators. Do not forget to dream! Let your dreams come true! Science should not be severe! In my lab, although it is more and more difficult due to the overbooking of MR systems, we try to

keep the principle of the "Friday afternoon experiments"... where science-fiction may lead to unexpected fantastic (or of course silly...) results! I have in mind several examples. I am providing to you only one example as an appetizer. A group at University of Mons was working on the biomagnetism of bees, and asked us to carry out EPR studies on different parts of the bee. We had the stupid idea to put an intact bee in the EPR cavity and to make a high resolution image of one EPR signal that was present in the bee. We

obtained fantastic images (in 2 and 3 dimensions) of the bees and of the repartition of the melanin present in the bees. This was terribly funny... and finally led to the analysis of the melanin in melanoma tumors that were grown in mice, the first in vivo EPR image of an endogenous free radical (melanin) in melanoma, one PhD thesis on the application of EPR imaging to melanoma, EPR imaging studies on human melanomas, studies about the influence of pigmentation on the EPR image, etc... Keep young!

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# Turning the Spin on Tablets

## A Personal Interim Report

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One of the most frequently questions I have been asked in my life is the following: “Are you really a pharmacist?” The answer is of course, “Yes, I am.” I also remember very well when I attended the first all-German pharmacy PhD-student conference that I was introduced by the chairman (a professor of pharmaceutical chemistry) as somebody who “uses a strange method called electron spin resonance”. It was my first talk and I felt like a zoo animal. I tried to neglect the introduction, but it was very hard for me to concentrate on the talk and not to follow the emotions. Anyway, it did not stop me from working as a “stranger”.

I became interested in EPR as a pharmacy student in 1988, when I heard lessons of Reinhard Stößer, one of the EPR pioneers in East Germany. I asked him about the possibility of doing a diploma in his lab, because I found the method interesting and I wanted to do something related to pharmacy, but outside of the mainstream research of our institute. For my fortune, he agreed and I got the possibility to start EPR measurements on microemulsions on a Varian E4 (the output was a pen reader). The instrument was heavily occupied and located in the Institute of Physicochemistry in Berlin, Bunsenstrasse, where Nernst postulated the third law of thermodynamics. The Reichstag, separated by the wall, was very close and there was a border gate for boats very close to the Institute. In many cases, I was measuring during the night and it was a strange and scary mixture of noises coming from the old institute and the border control with their German shepherd’s. The

Stößer group was composed of people from very different fields (ceramics, food chemistry, photochemistry, organic and inorganic chemistry...). Due to the small space, one got educated during the measurements due to the close proximity of the discussion room. I liked the work very much and I continued to work on the EPR/pharmacy interface as a PhD student. The work was on biodegradable polymers and biological applications of EPR and I was happy to start also with EPR imaging experiments together with Thomas Herrling. Due to the lack of low-frequency EPR spectrometers, we started in vivo EPR at X-band measuring nitroxide concentrations after transdermal or pulmonic administration. During this time, I attended my first EPR conferences. I got in contact with Hideo Utsumi (an EPR pharmacist!) and also with Hal Swartz. Both of them stimulated me very much to continue on my way. I visited the EPR lab of Hal Swartz at Dartmouth in Hanover, NH in 1993 for three months. I returned soon. The German Academic Exchange Service (DAAD) granted me a Postdoc stay in Hal’s lab in 1994/1995. This stay was probably the most research intensive and productive time and the reason to be nominated for the young investigator award. But I would also like to stress, that the same time was also a time of joy in my life. We worked in the lab, but we did also enjoy soccer, volleyball, hiking in the white and green mountains, concerts, cinema (Dartmouth is small, but offers a lot of culture) and also the local breweries. I completed my habilitation with a grant from the German research foundation

(DFG) in Berlin. I was very optimistic to get a permanent position at the Institute – until the closure of pharmacy at the Humboldt University was decided. Our first son was born and I had to look very quickly how to avoid unemployment. I was lucky to get a position in Marburg in the group of Thomas Kissel – one of the very best groups in drug delivery – which was unfortunately almost 450 km far away from Berlin where my wife had a permanent position. After one year of shuttling between Marburg and Berlin, I got a temporary position at the Free University in 1998. I switched from polymers to solid lipid nanoparticles and there was also some space to continue with EPR, although it was certainly not the main research focus. In 1999 I got the IES Young Investigator Award together with Bernard, and I thought to myself, it is a nice recognition, but maybe you will not be able to continue on this field.

The main concern was the job and I was applying for several permanent academic positions in the field of drug delivery – without any success until I had three offers simultaneously early 2000. Two associate professorships – but not well equipped – and an offer from the Pharma industry in Basel, Switzerland. Although I had only an academic carrier in my mind until 1999, I decided to switch to industry. I was very surprised to discover an EPR book in the lab library, where all other books were devoted to pharmaceuticals. Was it a test or joke? No, it wasn’t. Two group members (a physicist and a chemist) had an EPR background from the ETH Zürich or the USA. Although they never told me, I as-



sume that my EPR research was an advantage to get the job offer. However, there was no EPR spectrometer and the main tasks were very far from EPR. I was paid to develop new drug delivery systems and to focus on product based research. I did some work on NMR spectroscopy of formulations and also some in vivo MRI of floating tablets and their food interaction. In the fall of 2002 I got an offer for a Full Professorship in Pharmaceutics from the Martin-Luther-University in Halle. I did apply in 1998 and this position was actually out of my mind due to the long time which had passed. Because I was still very much attracted by the degree of research freedom, I accepted the offer. Since 2003, I have been a Full Professor of Pharmaceutics. I am very happy to combine the development of drug delivery systems with EPR, but we also work – in addition to formulation processes, particle sizing etc. – on Benchtop-MRI (BT-MRI) and multispectral Optical Imaging.

What are my feelings about the current status of EPR? I feel myself not really as an EPR scientist, but as an EPR user. I started my

carrier in Pharmacy as “somebody who uses a strange method”. Until now, many people in the drug delivery field perceive EPR as a “complex and difficult method”. However, EPR has also demonstrated its power as a unique research tool. For example, EPR was the first method to show that the pH value in clinically used biodegradable implants can drop to pH values as low as pH 2. More recently, EPR was the first method to quantify the detailed mechanism and kinetics of in situ forming implants in vitro and vivo. The work was awarded the “Phoenix” award of Pharma research, which honors the best publication in Pharmaceutics from Germany, Austria and Switzerland. This is certainly a stimulus to continue. I am also optimistic about the perspectives of EPR due to the development of new probes. I anticipate a growth and broader application of EPR, especially in preclinical in vivo research.

I lived my scientific life so far in almost two completely separated worlds: the EPR

and Drug Delivery (Pharmaceutics) world. I am very grateful that I had the possibility to get to know and also to cooperate with some of the leading scientists in each field. In the EPR field, I am especially obliged to Reinhard Stößer, Hal Swartz and Hideo Utsumi, which stimulated and supported me at the early stage of my career. I am grateful to all my group members, but especially to Hendrik Metz and Sabine Kempe, which run the major part of the EPR work in our group.

Coming back to the introduction and the “strange method” – It is a pleasure for me to be invited to give a talk at the annual meeting of the analytical group of the German Pharmaceutical Society this year. EPR and pharmaceutics – probably not as strange as 22 years ago...



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# Molecules, Thermodynamics and Cooking

## Georg Gescheidt

Institute for Physical and Theoretical Chemistry, Graz University of Technology, Graz Austria

Most of us like decent food, some of us even enjoy it.

Many of us have to teach with all its joys and frustrations. To me it has always been rather unsatisfying that a high number of students somehow learn thermodynamics and kinetics but it is hard to convince them that these subjects are actually crucial for all fields of molecular sciences. Astonishingly, many chemistry students in the early stages of their education are not even confident how they heat up foodstuff using microwaves.

Accordingly, at some point, I decided to show students that the knowledge of thermodynamic basics could be rather helpful for facilitating everyday matters like cooking. Here, the developments of the molecular kitchen, being rather popular for the last decade, appeared to be very helpful. So, I started developing a lecture on the physicochemical and molecular basics of cooking. There is a whole bunch of books around on this subject. I found the books by Peter Barham and Harold McGee particularly helpful [1, 2]. Whereas Barham concentrates on (simple) physical-chemical background, McGee offers lexical knowledge on almost any sort of foodstuff. Moreover, review articles help in following more recent developments [3–5] also illustrating the history of molecular gastronomy.

Preparing the new lecture opened a new world to me: Rather simple knowledge on thermodynamics, colloids etc. combined with cooking provides a lot of fun. Obviously the theory had to be tested with well-described and newly developed recipes (partly demonstrated in the lectures). Since then, the family, friends and my research group have become regular victims of my cooking adventures.

Here I will give you just two simple examples, which may serve as teasers for you to check more literature and to develop your own ideas and experience in this field.



One of the classics is preparing ice cream using liquid nitrogen. It is really possible to produce any kind of ice cream ranging from some smooth chocolate to a perfect sorbet. My coworkers like preparing fruity sorbets. You can either buy fresh fruit or frozen one, cut it into small pieces and blend it. Then add sugar syrup (easily produced by mixing identical weights of water and sugar and heating it) until you like the taste of the mixture (it should be relatively sweet). For fruit sorbets, the addition of some lemon juice always helps intensifying the taste. Now you can put the blended mixture into a metal container,

protect your hands and eyes, take a wooden spoon and stir it. A second person then slowly adds liquid nitrogen (Figure 1). Always make sure that the sorbet does not crystallize at the surface of the container, and if this happens just warm it up again a bit and re-cool it; rapid cooling helps since the mixture does not have sufficient time to crystallize. Moreover, you have to stir sufficiently fast. After just a short period of exercise, you will be able to produce any kind of ice cream you desire (coffee, vegetables...).

A frequently asked question is: How can I prepare a perfect steak? Again, the knowledge on molecular structure and phase transitions enables you to achieve a pleasant result. The strength of meat is substantially caused by the lamellar structure of the collagen fibers, which is based on protein triple helices. Heating to a temperature of ca. 55 °C causes that water molecules are inserted in the triple helix and, accordingly, the helix is widened, losing its strength. As a consequence, the meat gets nicely chewable, i.e., soft (Figure 2).

Too high a temperature, however, leads to the evaporation of the water molecules out of the helix and the peptides are irreversibly glued together, a very unpleasant tough situation. So obtaining soft meat affords only very low temperatures. Keeping the temperature constant between 55 and 65 °C does not change



Figure 1. Ice cream production.

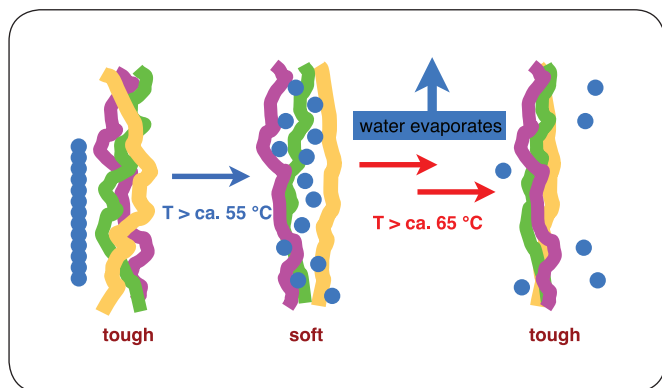


Figure 2. Gelation of meat at 55 °C and consequences of too much heat.

the soft quality of the meat since there is no phase transition in that temperature range. Unfortunately, the situation is not as simple. One aspect is that bacteria have to be destroyed (ca. 70 °C), the other that we want the meat to taste good as well, which is caused by the Maillard reaction starting at temperatures above 140 °C. Putting it all together, the meat has to be shortly heated to 70 °C, then kept at 55–65 °C and finally, the surface has to be heated to 140–160 °C to provide the

pieces or mince it, then the triple-helices will be cut short and the result becomes edible (chewable).

These were just two examples out of very many. Once you become addicted to combining physics, molecular basics and some knowledge about foodstuffs you can build up a rather wide range of simply achievable, interesting, and pleasant dishes.

Today's molecular gastronomy takes advantage of several ingredients, which are also used

taste (this can be done at the beginning or at the end).

Additionally (or alternatively) the triple helix can also be decomposed enzymatically by marinades or by injecting fresh pineapple juice (for ca. 15–20 min, longer periods lead to a meat gel – not very pleasant).

What can you do if the meat gets terribly tough? Cut it in small

industrially (emulgators, stabilizers etc.). From my point of view, though, natural ingredients offer a broad range of possibilities and the use of synthetic additives is not necessary. It is much more interesting to find out what can be done with natural resources.

To summarize, the impact of teaching experience led me to discover alternative ways to explain chemical and physico-chemical phenomena and to develop a lot of fun with preparing menus. So cooking became a combination of (re)searching and playing with results of a wide palette of qualities. I am continuing to explore.

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4. H. This: *Molecular Gastronomy*. Angewandte Chemie (International Edition) 2002, **41**, 83–88.
5. H. This: *Molecular Gastronomy, a Scientific Look at Cooking*. Accounts of Chemical Research 2009, **42**, 575–583.



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# Jim Bolton:

## An Interview on the Occasion of His 75th Birthday

EPR newsletter: *Dear Professor Bolton, on behalf of the readers of the EPR newsletter we congratulate you on your 75th birthday. We are most appreciative that you agreed to answer the questions of this interview. Why did you start towards your career in science?*

I have always been fascinated with how things work – I must have stressed my parents by always asking “Why?”. When I was in High School, my Chemistry teacher inspired me a great deal, and from that moment I had a ‘passion’ for Chemistry and was firmly committed to becoming a Chemistry Professor.

*Who introduced you into magnetic resonance?*

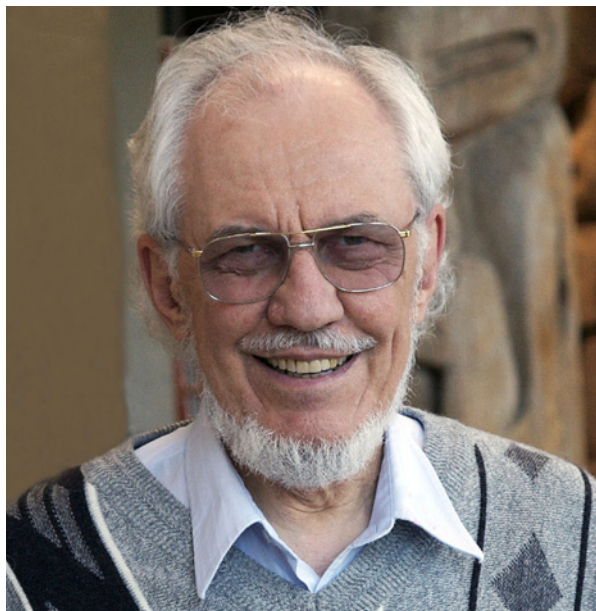
I was interested in Theoretical Chemistry, so I corresponded with Prof. Longuet-Higgins, who directed the Theoretical Chemistry group at the University of Cambridge. When I arrived in Cambridge and met Prof. Longuet-Higgins, he suggested that I work with a new lecturer in his group, Alan Carrington, who was interested in EPR to study the behavior of free radicals. He suggested that I set up a lab and use their newly acquired Varian EPR spectrometer.

*Could you please give us an historical perspective of your contributions to EPR?*

My research involved studying radical ions of aromatic compounds, such as toluene, xylenes, etc. This provided hyperfine splittings that reflected the unpaired electron density on the adjacent carbon. We found that these agreed very well with molecular orbital (MO) calculations. In particular, we showed that methyl substituents could split the degeneracy of the benzene anion MOs allowing either the *antisymmetric* or *symmetric* orbital to lie lowest.

The EPR spectrum of the durosemiquione anion exhibited a remarkable linewidth alternation caused by restricted rotation of the hydroxyl group. We required extensive calculations to simulate the effect. The university had set up one of the first computer centers in the world (the memory was only 2 KB!), which I used for my calculations.

I then joined Prof. George Fraenkel at Columbia University as a Postdoctoral Fellow. He had built an EPR spectrometer with very high resolution and sensitivity. Here I studied polycyclic aromatic anion and cation radicals. MO theory predicted



that the spin densities in the highest bonding molecular orbital should be the same as those in the lowest antibonding molecular orbital (the ‘pairing theorem’). We analyzed very-high resolution EPR spectra of the anion and cation radicals of anthracene and were even able to determine some of the  $^{13}\text{C}$  hyperfine splittings, but the  $^{13}\text{C}$  hyperfine splitting in the 9-position eluded us. Using  $^{13}\text{C}$  labeled at the 9-position we obtained a complete set of  $^{13}\text{C}$  hyperfine splittings, which were almost the same between the anion and cation. This provided experimental evidence to support the pairing theorem.

My first faculty position was at the University of Minnesota, where an older faculty member, John Wertz, was already doing EPR research. For the next several years, I did EPR research, and published many papers studying a variety of chemical problems, such as the nature of fluorine hyperfine splittings.

In 1967, I was beginning to feel a bit bored with the research I was doing (it did not have very much practical application.). I had a chance meeting with Albert Frenkel from the Botany Department, who was doing research with photosynthetic bacteria. When I told him about my EPR research, he was quite interested, so I invited him to bring some bacteria to my lab. So he and his graduate student, Connie Cost, came with a purple bacteria suspension, which we put into my spectrometer. In the dark, there was no signal. But when I turned on a flashlight, there was a very big signal. We were astonished; this totally changed the focus of my research. I suddenly had a passion to find out what was causing the generation of radicals in the bacteria. This was the beginning of my transformation to become a photochemist.

If I was to do research into the primary mechanisms of photosynthesis, I not only had to learn a lot about biology of plants and bacteria, I also had to learn about the basics of photochemistry. I used a sabbatical leave to work with Prof. Rod Clayton at Cornell University. I was attracted to him because he was a physicist, who had decided to study the photochemical reactions in photosynthesis, so his background and motivation were similar. So I applied EPR spectroscopy

<sup>1</sup> Jim Bolton was born in 1937 in Swift Current, Canada and received his BA and MA from the University of Saskatchewan and Ph.D. from the University of Cambridge. He is currently an Adjunct Professor in the Department of Civil and Environmental Engineering at the University of Alberta and President of a consulting company, Bolton Photosciences Inc.

<sup>2</sup> In the interests of saving space, no references have been given. Dr. Bolton would be happy to provide them – contact him at [jbolton@boltonuv.com](mailto:jbolton@boltonuv.com).

to show that the bacterial EPR signal arose from a special reaction center protein that Rod Clayton and his group had just been able to extract from photosynthetic bacteria. We were the first to determine the quantum yield (1.0) for the generation of the EPR signal in photosynthetic bacteria.

In 1970, I moved to the University of Western Ontario in London, Canada where I joined the newly formed Photochemistry Unit. I carried on doing research on photosynthesis, such as determining the quantum yield for the EPR signals in chloroplasts. But gradually my interests shifted away from EPR to other aspects of photosynthesis, including modeling of the reaction center of photosynthesis. Today, my research does not involve EPR but is heavily oriented to environmental photochemistry, such as the application of ultraviolet to disinfect and/or purify water and air.

*What is the story beyond your textbook "Electron Paramagnetic Resonance, Elementary Theory and Practical Examples", 2nd edn., by John Weil and James Bolton, Wiley, New York, 2007?*

In 1966, the American Chemical Society asked John Wertz and I to organize a workshop on Electron Spin Resonance (as EPR was

called at that time). We put together a set of lecture notes and problems for the Workshop. We decided to expand them into a book. So "Electron Spin Resonance, Elementary Theory and Practical Applications" was published by McGraw Hill in 1972. From the beginning, we adopted the premise that learning is best achieved by problem solving, so each chapter was accompanied by a set of problems at the end. This book rapidly became the 'standard EPR textbook'.

When our book went out of print, John Wertz and I decided to organize a new edition and asked John Weil to join us as a co-author. By this time John Wertz's health was not good, and my interests had shifted away from EPR, so John Weil took on the heaviest load in the new 1994 edition.

Sadly, John Wertz passed away in 1997, and John Weil felt that a new edition was necessary, so I helped him to prepare the 2007 edition, although I did not contribute much to its scientific content. Sadly John Weil passed away in 2011, so I am the only remaining living author of this textbook. If anyone would like to take on the task of preparing a new edition to keep the textbook up-to-date, I would be happy to cooperate.

*What would you have done differently given the chance?*

Nothing really. I have been blessed with good luck and fascinating research projects throughout my life.

*What is your message to the younger generation of the magnetic resonance researchers?*

Follow your passion wherever it may take you. Be curious and alert for the unusual. If you discover something that is very interesting but outside your current research interests, have the courage to take some time to explore the new opportunity – no telling where it will take you! I have found that I have had the most energy and excitement every time I changed research directions. Finally, remember that you could not have got where you are without the support of family, students and friends.

So I end this interview with warm thanks for the very significant support I have had from my mentors, my many graduate students, post-doctoral fellows and research associates, and, in particular my first wife, Wilma (sadly she passed away in 2000), and my second wife, Ingrid.

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# High-Frequency and High-Field EPR/ESR in Tallahassee, FL

## Tips and advice on sample preparation

Jurek Krzystek, Andrew Ozarowski,  
Johan van Tol, Junjie Liu, Stephen Hill

National High Magnetic Field Laboratory,  
1800 E. Paul Dirac Drive, Tallahassee, FL 32310

The EMR Program of the National High Magnetic Field Laboratory offers several high-frequency EPR/ESR (HFEP) spectrometers to users from all over the world upon receipt of a two page proposal (see: <https://users.magnet.fsu.edu> and [www.magnet.fsu.edu/usershub/scientificdivisions/emr/index.html](http://www.magnet.fsu.edu/usershub/scientificdivisions/emr/index.html)). These instruments differ radically from conventional (e.g., X-band) setups in their design and operation. Consequently, samples must be prepared differently as well. Moreover, sample requirements vary from one spectrometer to another. Since many of our potential users are not aware of these requirements, we have prepared the following summary. In doing so, we were inspired by Ralph Weber *et al.*, who published an excellent sample “primer” for users of the Bruker BioSpin facility in Billerica one of the previous issues of the *EPR newsletter* (Vol. 21 (2), 2011).

### The Superconducting Magnet-Based Broad-Band EPR Homodyne Instrument and the Resistive Magnet-Based Spectrometer

These two spectrometers share the same basic transmission design, with no resonator [1, 2]. The frequency range is 26 GHz – 1 THz. A sample is positioned inside the waveguide in the center of the magnet in vessels of maximum outer diameter of 8 mm (Fig. 1). Teflon cups of that diameter are most often used as sample containers, as are gelatin caps (size ‘00’), Fisher brand vials 03-338-1A, or similar (Fig. 2). The sample height may be up to 10 mm. To avoid torquing in high magnetic field, compressed powder pellets of the compound under study mixed with KBr may be prepared, or *n*-eicosane used as an immobilizing agent. Powder samples must be very well ground as the finite crystal size effects are much stronger in HFEP than in X- or Q-band. The amount of sample depends strongly on the system being studied. For transition metal complexes this may vary from single mg to 200 mg or more. It is advisable to contact the authors of this article prior to bringing or sending samples to Tallahassee.

Air sensitive samples can be sealed in 6–8 mm OD quartz tubes, which should be made as short as possible, no longer than 50 mm (Fig. 3). This arrangement works up to about 300 GHz; above that frequency quartz becomes strongly absorbing. Usage of X-band quartz tubes is not recommended. An alternative way of handling air-sensitive samples is to ship them to us pre-loaded in Fisher brand vials 03-338-1A, which in turn are sealed in glass vials, and placed on dry ice, or in liquid nitrogen to slow decomposition.

Frozen solutions in various solvents, including water, have been successfully measured; also air-sensitive samples using the method described above. Volumes of up to ca. 500  $\mu$ L can be handled. The

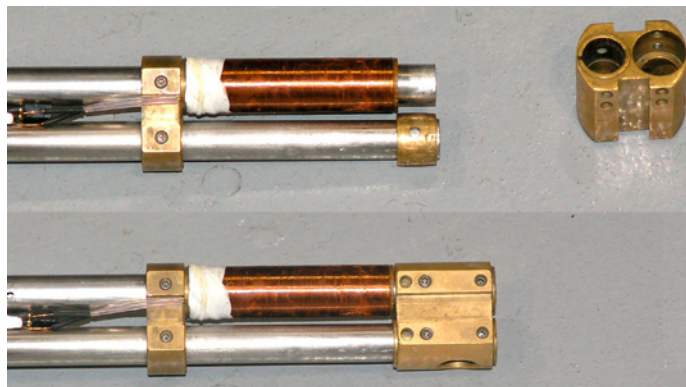


Fig. 1. Bottom part of the non-resonant transmission probe associated with the homodyne superconducting magnet-based spectrometer. The sample container is placed in the middle of the modulation coil on a proper support. The brass part on the right contains a V-shaped mirror reflecting the sub-terahertz wave beam back to the detector.



Fig. 2. Typical sample containers for non-air sensitive powder or solution samples in the homodyne transmission spectrometers. From left to right: Fisher brand vial 03-338-1A; a homebuilt teflon container integrated with the support placing the sample at the right height; a teflon support for a sample cup; a 400  $\mu$ L-volume teflon cup; a ‘00’ size gel cap. The outside diameter of all these containers is 8 mm.

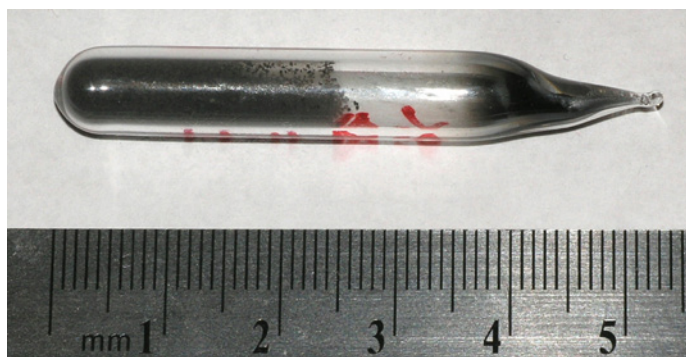


Fig. 3. Flame-sealed quartz ampoule used for air-sensitive samples in the homodyne transmission spectrometer. Outside diameter, 8 mm.

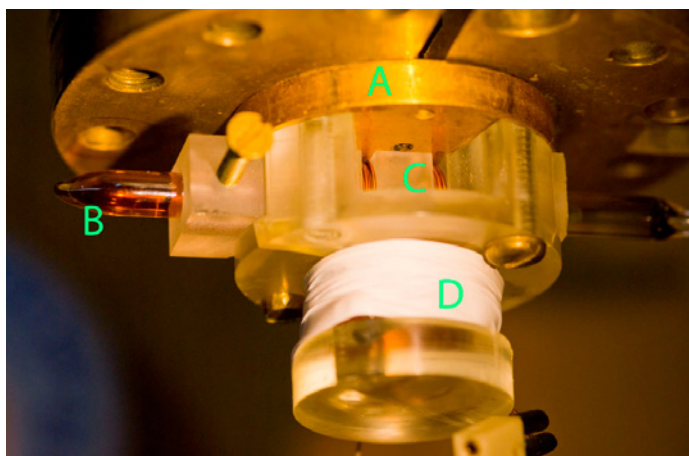


Fig. 4. ENDOR sample holder for 4 mm tubes at 120, 240, and 336 GHz in the heterodyne quasi-optical spectrometer. A: Bottom flange of the corrugated horn; B: 4 mm sealed sample tube; C: active sample space with ENDOR coils. D: modulation coil.



Fig. 5. Single-crystal sample rotator for the heterodyne quasi-optical spectrometer. The 2 mm rod fits sideways into the modulation coil (right).

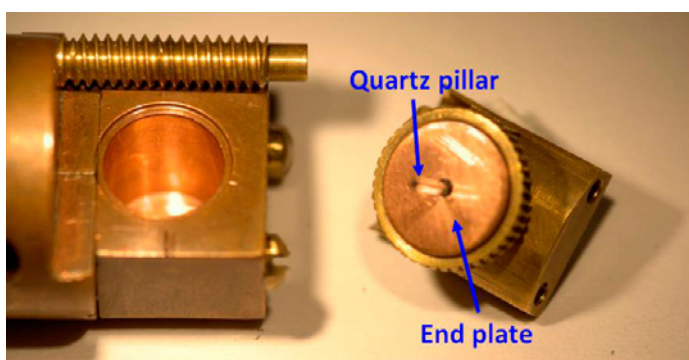


Fig. 6. The rotating resonant cavity for the heterodyne HFEP spectrometers in the Hill lab. Part of the cavity (the end plate) has been disassembled in order to show the inside of the resonator. The sample can be mounted on either the end plate (diameter ~8.4 mm) or the quartz pillar (diameter ~0.7 mm). The end plate, as well as the quartz pillar, can then be rotated via an externally controlled worm drive.

minimal concentration strongly depends on the spin system under investigation. While for low-spin ( $S = 1/2$ ) species concentrations of a fraction of 1 mM can be sufficient, in broad-line metal complexes the concentration needs to be at least in the 10–100 mM range.

Single crystal spectra at a fixed orientation can be recorded in these spectrometers (see below for oriented crystal measurements). A variable-temperature cryostat provides temperatures in the 3–300 K range in the case of the superconducting-based instrument, or 1.4–77 K for the resistive magnet-based setup.

#### The Multifrequency Quasi-Optical Heterodyne CW/Pulsed EPR/ENDOR Spectrometer

This spectrometer operates at 120, 240, and 336 GHz; the sample requirements for EPR are similar at all three frequencies, at least for measurements without a resonator [3]. The instrument is permanently associated with a 12.5 T superconducting magnet. The temperature range is 1.2–300 K. The typical sample space for powders, frozen solutions, or crystalline samples is 3.5 mm (1/8 inch) diameter and about 6 mm depth, but the latter is variable from 0.5 to 10 mm. Typically for powder and frozen solutions, a small polyethylene cup with inside dimensions of 4 mm diameter and 3 mm height is used, with a volume of around 35  $\mu\text{L}$ .

Because the detection method and sensitivity rely on ‘induction mode’ detection, where the on-resonance circularly polarized EPR absorption induces a cross-polar signal, symmetric samples give best results.

For sealed (air sensitive) samples, a 4 mm EPR quartz sample tube can be mounted horizontally. The maximum tube length should be less than 60 mm, and sample should be present in the center of the tube (Fig. 4). If it is necessary to make the seal further from the sample, a tube with a 90 degree bend can be used as well.

Samples for ENDOR measurements tend to be a little smaller, but different ENDOR coils with different sizes can be used.

The optimal sample dimensions for pulsed EPR/ENDOR measurements tend to be a little smaller, typically of the order of 2–3 mm diameter and 1–2 mm height in order to have the sample in the highest  $B_1$  region, and to limit (electric dipolar) absorption in the sample, which can be appreciable at high frequencies. Holders and RF coils can be tailored to a specific sample.

For measurements on single crystals, a single axis mechanical rotator is available. Crystal dimensions should be of the order of 2 mm or smaller (Fig. 5).

In some cases, e.g. for liquid aqueous solutions, a Fabry-Perot resonator is the best option. Sample volumes are then much smaller. 0.25, 0.15, and 0.1 mm ID capillaries give the best results at 120, 240, and 336 mm, respectively. The capillaries are mounted horizontally and should have a length of 40–50 mm. Small single crystals (~0.1 mm dimensions) can be also be mounted and rotated in the Fabry-Perot resonator. Furthermore, uniform flat samples, such as thin films on a substrate, or semiconductor wafers can give good results in the Fabry-Perot resonator.

#### The heterodyne HFEP spectrometers in the Hill lab

The spectrometers in the Hill lab are optimized for oriented single-crystal c.w. EPR measurements spanning the frequency range from 50 GHz to 1 THz, from room temperature down to as low as 500 mK (1.25 K is routine). Resonant cavities (see Fig. 6) are used at lower frequencies (< 400 GHz) [4, 5], while a simple transmission configuration is used at the higher frequencies. Spectrometers and probes can either be associated with a 15 T vertical superconducting magnet, a 7 T horizontal split-pair magnet, or a 9/5/1 T vector magnet, where the latter two systems enable *in-situ* two-axis crystal rotations. In addition, all spectrometers are compatible with the

## Tips & techniques

dc resistive magnets at the NHMFL with a highest field of 45 T, including single-axis crystal rotation capabilities. The possibility of performing two-axis oriented single-crystal EPR studies under hydrostatic pressures of up to 3 GPa also exists [6]. The sizes of samples needed for ambient-pressure single-crystal EPR studies varies from about a millimeter to just a few hundred microns in terms of the average linear dimension; the minimum sample volume of course depends on several factors such as the spin density, the sharpness of the EPR spectrum, and the frequency range where data are desired. As a general rule, higher frequency measurements require larger crystals, while high-pressure studies are limited to sub-millimeter sized crystals. X-ray structures should be available before single-crystal measurements are performed. If at all possible, users are encouraged to send crystals that have been face-indexed, though this is not required. Preliminary magnetic measurements are also very useful, e.g., dc susceptibility measurements are strongly recommended in order to facilitate planning and interpretation of single-crystal EPR measurements. For compounds that co-crystallize with structural solvent molecules, it is suggested that the crystals should be shipped in the solvent in order to avoid evaporation and degradation in sample quality. For compounds containing particularly volatile solvents, as well as materials that may be air-sensitive, protocols have been developed for rapid sample loading (within <1 min [7]). Finally, potential us-

ers are encouraged to discuss plans with members of the Hill group prior to submission of a proposal.

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## Readers corner

Dear EPR Colleagues,

I was recently elected Vice President (2012–2014) and President-Elect (2014–2016) of the Inter-American Photochemical Society (I-APS) ([www.i-aps.org](http://www.i-aps.org)). I am very grateful to the editor for an opportunity to introduce this very broad group of scientists to the EPR community, and tell you a little bit about their activities.

The I-APS was established in 1976 to promote and disseminate knowledge and encourage development of photochemistry and allied subjects throughout the North, Central, and South America. The Society complemented similar organizations in Europe and Japan (now, Pan-Asian). Within 14 months of its founding, the Society counted 460 members, and today is 600 strong. The mission of the I-APS is the promotion of cooperation between American photochemists in universities, research centers, and industry through fostering national and international contacts. In addition, the organization and encouragement of educational activities, workshops, and symposia in photochemistry at all levels is a high priority for the Society. Many fruitful collaborations between North American scientists and their cousins to the south have originated and grown through I-APS activities.



The I-APS has a broad scope, using the interdisciplinary nature of photochemistry to bridge the traditional areas of chemistry, including theory, photophysics, photobiochemistry, photobiology, photovoltaics, photocatalysis, and spectroscopy, as well as related areas such as EPR. Indeed, you will see in the membership roster many established researchers from the "spin chemistry" community, such as Mike Wasielewski, Jim McCusker, Nick Turro and myself. The society's long-standing links to the EPR and spin chemistry communities are exemplified by the G. L. Closs Student Award (see more below), which is named for my late advisor and honors

a scientist who built strong bridges between the magnetic resonance and photochemistry fields throughout his outstanding career.

Bringing together scientists from diverse fields has the potential to aid in finding solutions to global problems that involve photochemistry, such as the conversion of solar energy to electricity or chemical energy, environmental remediation using sunlight, and understanding the reactivity of greenhouse gases and pollutants when they interact with light in the atmosphere. The photophysical properties of molecules and materials are central to industrial processes, such as opto-electronic devices, sensors, and high-density data storage, among others. Magnetic field effects, spintronics, catalysis by coordination compounds, and characterization of excited triplet states are all highly relevant sub-specialties of organic, inorganic and physical chemistry that forge links between EPR spectroscopy and the light-induced behavior of molecules and materials.

The most significant contributions to the photochemistry field are recognized by a series of prestigious awards sponsored by the Society, including the I-APS Award in Photochemistry and the Young Investigator Award. In addition, the Cilento Award recognizes the accomplishments of South American scientists and the Gerhard Closs Student Award



recognizes outstanding research in photochemistry by graduate students. The Society also partners with the European and Asian and Oceanic Photochemistry Societies in awarding the Porter Medal every two years. A distinguished group of 18 photochemists have been selected as I-APS Fellows for their lifetime contributions to the field. Our most recently established award, the Hammond Award, is given for lifelong achievement and contributions to the photochemical sciences. Prof. Nicholas J. Turro of Columbia University was selected as the recipient of the inaugural 2012 Hammond Award. Nick is well known to the EPR community as a pioneer of supramolecular “spin chemistry” research and is a most deserving first recipient of this award.

In the 36 years since its founding, I-APS has sponsored 22 conferences that bring together photochemists and photophysicists, from throughout the Americas at various sites in North and South America, for 3-4 days of intensive discussions of the latest research advances in the field. Recent South American meetings were held in Bahia, Brazil and in Mendoza, Argentina.

Together with current I-APS President Claudia Turro, I invite you to participate this year’s Winter Meeting in Sarasota Florida (at the Lido Beach Resort), which takes place January 2–5, 2013. This meeting features presentations by all the 2011 and 2012 I-APS award winners, as well as by Prof. Thomas J. Meyer, who was awarded the 2012 Porter

Medal. We expect a vibrant meeting with significant student participation.

I also ask that you and your students consider an I-APS membership of your own, for very reasonable annual fees (student fees are kept very low to encourage their participation in the Society). Students also pay reduced fees for the annual meeting and are eligible for travel awards to the winter conference. More information about I-APS can be found at our website: [www.i-aps.org](http://www.i-aps.org).

I hope to see you Sarasota this winter!

With kindest regards,  
Malcolm D. E. Forbes  
Vice President, I-APS, 2012-14  
Chapel Hill, North Carolina  
September 24, 2012

## Conference reports

### 45th Annual International Meeting of the ESR Spectroscopy Group of the Royal Society of Chemistry

The 45th Annual International Meeting of the ESR Spectroscopy Group of the Royal Society of Chemistry (RSC) was held at The University of Manchester 25–29th March 2012. The conference was attended by 95 delegates from 15 countries and was held at

the Chancellors Hotel & Conference Centre. The conference was hosted by Profs. David Collison and Eric McInnes with magnificent support from Prof. Richard Winpenny, Dr. Kathy England, Cassandra Kenny, Sarah Evans and Gill Smith, and the smooth running was ensured by the help of the post-graduate and -doctoral researchers from the Manchester EPR and molecular magnetism groups. The successful organisation even extended to the

weather, with not a single cloud spotted in a gloriously sunny week!

The scientific highlight was the 2012 Bruker Prize Lecture, delivered by Prof. Kev Salikhov of the Zavoisky Physical-Technical Institute of the Russian Academy of Sciences. His excellent lecture entitled “Quantum computing on electron spins using the pulse EPR spectroscopy methodology” highlighted some of the opportunities and challenges in this excit-



The 2012 conference delegates in the grounds of the Chancellors Conference Centre. Photographs courtesy of Dr. Art Heiss (Bruker).

## Conference reports

ing area. Prof. Robert Bittl (Freie Universität Berlin) introduced Prof. Salikhov's lecture and highlighted the many achievements of his long and varied career in the development and applications of EPR spectroscopy.

The conference opened with a keynote lecture from Prof. Richard Winpenny (University of Manchester) on "EPR studies of rings and dimers of rings", and further keynotes were given by Profs. Walter Köckenberger (University of Nottingham; "From electron-nuclear spin pairs to the electron spin interaction with the bulk nuclei: a closer look at dynamic nuclear polarisation"), Heinz-Jürgen Steinhoff (University of Osnabrück; "Structure and conformational dynamics of nucleic acids and membrane protein complexes studied by site-directed spin labelling"), and Takeji Takui (Osaka City University; "New aspects of nitroxides and open-shell graphene fragments chemistry: from quantum computers to energy conversion materials"). Invited lectures were given by Dr. Gavin Morley (University of Warwick; "Quantum control of hybrid nuclear-electronic qubits"), Dr. Arzhang Ardavan (University of Oxford; "Quantum information processing with molecular nanomagnets"), Prof. Georg Gescheidt (Technische

Universität Graz; "Probing (anti)-oxidative effects with time-resolved EPR and CIDNP"), Prof. Steffen Glaser (Technische Universität München; "Optimal control of spin dynamics in magnetic resonance"), Dr. Christiane Timmel (University of Oxford; "A multifrequency approach: using RF and microwave fields to unravel the recombination kinetics of a photoinduced radical pair") and Prof. Robert Bittl (Freie Universität Berlin; "EPR in bioenergetics and photovoltaic research").

The 2012 JEOL Student Lecture Prize competition was won by Alice Bowen of the University of Oxford, with an outstanding lecture on the development on DEER methods entitled "Utilizing the TWT linear region: double electron-electron (DEER) with multiple pulse excitation pulses and dead-time free DEER". Other lectures in this excellent session were given by Ilia Kaminker (Weizmann Institute of Science; "A novel triple resonance correlation sequence for resolving and assigning signals in ELDOR-detected NMR spectra"), Tomasz Mazur (Jagiellonian University; "EPR/HYSCORE and DFT study of nickel adducts with O<sub>2</sub>, CO and NO molecules engaged in zeolites"), Christoph Meier (Freie Universität Berlin; "Microcrystalline silicon: orientation

dependence of light induced EDMR signals") and Gunnar Reginsson (University of St Andrews; "Trityl: a new spin label for nanometre distance measurements").

On the free afternoon, over twenty of the delegates visited the EPSRC UK National EPR Spectroscopy Facility & Service in the Photon Science Institute ([www.epr.chemistry.manchester.ac.uk](http://www.epr.chemistry.manchester.ac.uk)), and were given a guided tour by Floriana Tuna, Stephen Sproules and Daniel Sells. The conference dinner was held in the imposing setting of the dining room of Wooton Hall, where the Chair of the ESR Spectroscopy Group (Mark Newton, Warwick) oversaw the presentation of the Student Prize Talk to Alice Bowen, by Peter Meadows of JEOL (UK)

The conference was a great success and the RSC Committee would like to thank Bruker and JEOL for their continued sponsorship and support of this meeting. The 46th International Meeting of the RSC ESR Group will be held at the University of Warwick from Sunday 7th to Thursday 11th April 2012. Information about the 2012 meeting, previous meetings and the RSC ESR group can be found at the website [esr-group.org](http://esr-group.org).

Eric McInnes

## EUROMAR 2012 Dublin, Ireland, July 1–5, 2012

This year's EUROMAR meeting took place in the University College Dublin, with Prof. Dr. Chandralal Hewage as the main organizer. The meeting was preceded by the XeMat2012 meeting (27–29 June) and the

COST Spin Hyperpolarisation meeting (29 June – 1 July), which attracted many scientists interested in Xe-NMR and DNP to the meeting. The EUROMAR conferences originate from the grouping of the European experimental NMR conference, the AMPERE congress and the NMR Discussion Group Meeting of the Royal Society of Chemistry to offer a communication platform for new methodological

approaches, innovations and applications in the field of magnetic resonance. With more than 600 participants, the EUROMAR2012 meeting confirmed that EUROMAR is by now the main meeting of the European magnetic resonance community.

The conference started with a Sunday tutorial session on *Current trends in metabolomics* (L. Brennan), *Quadrupole NMR in solids* (P. ►



Grandinetti) and *The inner workings of NMR probes – How to get more from CPMAS* (K. Zilm). Before the price ceremony, the participants took a moment to pay tribute to two important magnetic resonance colleagues that sadly passed away in past months. Beat Meier highlighted the achievements of Robert Blinc, president of the Groupement AMPERE from 1990 to 1996, who died in September 2011 at the age of 78, and Andrew Coy gave a personal view on his friend Paul Callaghan who lost his fight against cancer in March 2012, only 63 years of age. Paul was meant to be one of the plenary speakers at the conference...

During the prize ceremony, Galia Debelouchina (Princeton, USA) received the Raymond Andrew Prize of the Groupement AMPERE and Lyndon Emsley (Lyon, France) was honoured with the AMPERE prize. The evening session was finalized by a keynote lecture of Robert Tycko on *Biomolecular solid state NMR*. Despite the fact that the subsequent welcome mixer was not supported by the weather gods, it offered a good opportunity to mix with friends and colleagues. In fact, some of the vendors had foreseen rain and had turned it to their profit by handing out umbrellas to those visiting their stands.

In the following four days, 86 talks (12 plenary, 32 invited and 32 promoted talks) were given. Two plenary talks and two parallel sessions were fully dedicated to EPR, while additional DNP- and EPR-related talks could also be found in other sessions. Every day, a poster session was held, allowing the participants to enjoy the roughly 400 posters that were on display. Many young scientists were present at the meeting, also thanks to the grants

given by the EUROMAR committee to PhD students and young postdocs. Furthermore, during the conference, three MRC awards were awarded to young scientists sponsored by John Wiley & Sons, selected on the basis of their abstracts.

The coffee breaks in the poster area and the hospitality suites of Bruker and Agilent held in the O'Reilly hall, allowed for time to discuss science (and other important things in life) in a relaxed atmosphere (sometime even with live music). On Thursday evening, the plenary lecture hall underwent a metamorphosis and became the site for the conference banquet, with traditional Irish folk music and Irish river dancing. The (professional) dancers picked out a few people from the banquet and made them join the dancing. I leave it to your imagination how that looked to the observers standing at a safe distance from the dance floor...

Chandralal Hewage and his colleagues of the University College in Dublin took on a great challenge when agreeing to organise the EUROMAR meeting, since they were the first that could no longer benefit from the EMAR funding that provided a safety net for the previous organisers. They did a splendid job in making the conference a success on all levels.

Next year, the EUROMAR meeting will be organized in Hersonissos (Crete) and the fact that the local organisers have put a HYSOCORE spectrum (and not an NMR spectrum!) on their announcement flyer indicates that the organizers want the EPR field to be strongly represented in the meeting. I therefore hope to see you next year in Crete with many EPR spectra.

Sabine Van Doorslaer

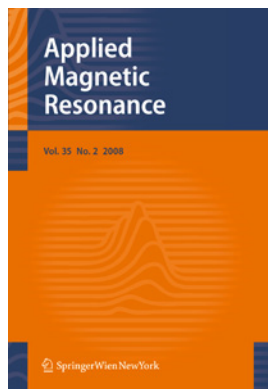
University College Dublin (Ireland), hosts of EUROMAR 2012, has a large campus in the suburbs of Dublin, Belfield with a nice pond with swans and fountains in its center. The scope of this meeting was traditionally focused on the topical problems of NMR and DNP, and also included many plenary EPR lectures.

First of all, I would like to say many thanks to the organizers of the meeting for their support and the opportunity to participate in the conference with other young postdoctoral researchers and graduate students. As for me, this conference also provided an opportunity to meet with my friends and colleagues.

It is no secret and everybody realizes that the most important aspect in the work of a researcher is that the results of the research are in demand. To find a practical application of his method or object means to find finance. In my opinion, medicine has become the most popular area of EPR and NMR research. In this field, EPR and NMR methods demonstrate their power. The most well-known topics of research in medicine are still cancer, the early diagnostics of which is extremely important for successful treatment, and also Alzheimer and similar diseases. NMR, which has long been used in various forms from tomography to conventional spectroscopy, was represented by a large number of reports concerning solid-state NMR. I was impressed by the reports on the diagnostics of urine and blood metabolites. Professor Jeremy Nicholson from Imperial College London, who presented a plenary lecture, carried out a very successful research in this field. In general, metabolo-



# The Different Magnetic Resonance Communities Join Forces for Progress in DNP\*



Special Issue on DNP of Applied Magnetic Resonance  
 Edited by Vadim Atsarkin and Walter Köckenberger  
 Springer-Verlag 2012

This special issue of *Applied Magnetic Resonance* is the result of an attempt to show

the very broad range of current research activities revolving around the central theme of dynamic nuclear polarization (DNP) and the impact DNP is currently making in many different scientific disciplines. It is fascinating to see how an idea that is almost as old as the whole field of nuclear magnetic resonance (NMR) is now revolutionizing the way people think about the maximal sensitivity that can be achieved in NMR experiments. Nowadays, scientists try to produce fully polarized nuclear spin systems for use as highly sensitive probes in medical diagnostic imaging or to

generate high spin polarization on surfaces to investigate conformation of molecules in the boundary layers. In particular, two developments have tremendously stimulated the recent progress in DNP research. First, through the continuous and persistent efforts of Prof. R Griffin at the Massachusetts Institute of Technology, Boston US, robust hardware and experimental strategies are now available for solid-state magic angle spinning NMR experiments in conjunction with DNP. The second development was the use of low-temperature DNP followed by a fast rise in temperature to produce solutions containing highly polarized spin systems. This technique is now commonly referred to as dissolution DNP and has been conceived and developed by Prof. K. Golman and Prof. J.-H. Ardenkjaer-Larsen in Malmö, Sweden. The impact that these two major achievements are currently generating on the design of novel strategies for NMR spectroscopy and MR imaging and on increasing the range of applications cannot

be rated highly enough. On the other hand, it is clear that the idea of using the electrons to generate highly polarized nuclear spin ensembles had been developed many years back, in the early days of magnetic resonance, by a number of pioneers in magnetic resonance such as Albert Overhauser, Anatole Abragam, Maurice Goldman and others. As a great loss for the scientific community, both Albert Overhauser and Anatole Abragam passed away in 2011 and for this reason we should remind ourselves about the solid theoretical foundations that were laid out by these two great scientists and on which we are building today. The first article in this special issue is written by Charles Slichter and devoted to the memory of Albert Overhauser. We would also like to remind in this special issue of the progress made in understanding the fundamental principles of DNP during the 60s and 70s of the last century. The second paper tries to summarize these historical achievements.

The special issue contains articles about DNP theory, hardware and experimental strategies, as well as applications of a wide variety, ranging from materials sciences to biomedical diagnostics. The articles originate from an interesting mixture of different disciplines and as such truly reflect the current state of the DNP community. One important achievement of the ongoing DNP research efforts has already become apparent: the hunt for better sensitivity and more exciting applications has unified many branches of magnetic resonance. Previously, very little

\* Reproduced from *Appl. Magn. Reson.* (2012) 43:1–2, DOI 10.1007/s00723-012-0368-z with permission from Springer-Verlag.

## Conference reports

mics has been a subject of a lot of reports in the NMR sections.

In the EPR section, a very interesting report was presented by Professor Sabine Van Doorslaer with an allegorical comparison of cartoon characters: big, powerful and clumsy Obelix – NMR and small, fast Asterix – EPR. I would emphasize that several reports in the EPR and NMR sections were concerned with the quantum chemical calculations, mainly DFT calculations of the EPR and NMR parameters and the structure of objects. A relevant talk about the structural characterization of various samples from small molecular to large molecular crystals supported by quantum chemical calculations was given by Professor Lyndon Emsley, the AMPERE Prize 2012 winner. Reports on the ab initio calculations were also presented. Poster sessions with about 500

posters demonstrated a large variety of NMR and EPR research in diverse fields.

And finally many thanks to traditional sponsors of this conference: Bruker, JEOL and Agilent Technologies, and to somewhat not so large heavyweights such as Cambridge Isotope laboratories, Sigma Aldrich, Cryogenics Limited and many others companies. We enjoyed a nice party with drinks and food they provided. One night a party was especially memorable because of the final football game of Spain vs. Italy in the Euro 2012 Cup. This game was displayed on a large screen. Scientists are also human beings and they consider nothing that is human alien to them.

The next EUROMAR conference will be held in Crete, Greece from June 30 to July 5, 2013. I strongly recommend it to all.

Alexey Kononov

## notices of Meetings

↑ **SCM 2013** ↓  
 Bad Hofgastein

**13th International Symposium on Spin and Magnetic Field Effects in Chemistry and Related Phenomena (SCM2013)**  
 Bad Hofgastein, Austria, April 22–26, 2013

The meeting is organized by Peter Hore and Stefan Weber together with the international scientific committee. The conference is an open meeting for all aspects of spin related phenomena and also for all groups worldwide. Due to the excellent facilities we have booked for you, the conference can accept a maximum number of 120 participants. The poster space is also practically unlimited (about 100).

cross talk and exchange took place between the electron paramagnetic resonance community, the NMR solid-state and liquid-state spectroscopy communities and the magnetic resonance imaging community but it is currently interesting to observe how these com-

munities moved together to join forces for achieving even better progress in DNP.

We very much hope that this special issue will serve the DNP community, and also any other scientist who wants to enter this field, as a source for interesting, ideas and infor-

mation. Certainly for us as Guest Editors the task of putting together this issue has been highly enjoyable and rewarding.

Vadim Atsarkin  
Walter Köckenberger

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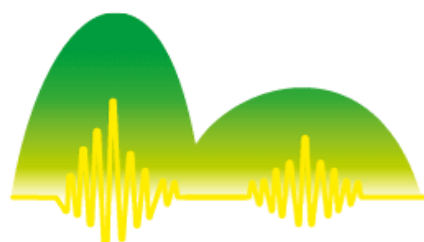
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Registration will open in November and can be reached via the conference web page:

[www.cecp.at/index.php/scm2013](http://www.cecp.at/index.php/scm2013)

E-mail: [SCM2013@cecp.at](mailto:SCM2013@cecp.at)



18th ISMAR Meeting  
14th NMR Users Meeting  
Vth Iberoamerican NMR Meeting  
Biomedical Imaging Symposium  
Rio de Janeiro, Brazil, May 19–24, 2013

Web site: [www.ismar2013.net](http://www.ismar2013.net)

E-mail: [ismar2013@arem.org.br](mailto:ismar2013@arem.org.br)

## Scientific Program:

Anatole Abragam Prize lecture, sponsored by Bruker. • ISMAR Prize lecture, sponsored by Cambridge Isotope Laboratories (CIL). • Paul Calagham Plenary lecture, sponsored by Magritek.

Ten plenary lectures, 96 invited lecturers and 56 oral presentations selected from submitted abstracts are programmed for the event, covering the topics listed below:

Biomolecular Nuclear Magnetic Resonance (NMR) • Solid-state NMR/Physics/Exotica/Instrumentation • Liquid Methods/Pharma/Small Molecules (Special session on NMR applied to Natural Products and Medicinal Chemistry) • In vivo/Magnetic Resonance Imaging (MRI)/In cell • Electron Paramagnetic Resonance (EPR)/Electron Nuclear Double Resonance (ENDOR)/Dynamic Nuclear Polarization (DNP) • Workshop NMR on Porous Media • Hot Topics session



**Erratum:** Custom Design of Microwave Probes for EPR Using Numerical Analysis Techniques by Jason Sidabras EPR newsletter 22/1 (2012, pp. 11–13

Two equations in the article “Custom Design of Microwave Probes for EPR Using Numerical Analysis Techniques” by Jason W. Sidabras were missing a  $\mu 10^4$  for a unit conversion from Amps/meter to Gauss. The efficiency parameter,  $\Lambda$ , is

$$\Lambda = \frac{\mu 10^4 \max(H_r)}{P_1^{1/2}} \text{ [G/W}^{1/2}\text{]},$$

and the average efficiency parameter over the sample,  $\Lambda_{\text{ave}}$ , is

$$\Lambda_{\text{ave}} = \frac{\mu 10^4}{P_1^{1/2} V_s} \int H_r dV_s \text{ [G/W}^{1/2}\text{]}.$$

### POSITIONS



#### Research Positions – Advanced EPR of Biochemical and Chemical Systems

Several research positions (doctoral and postdoc level) are presently available in the Biophysical Chemistry Department of the Max Planck Institute for Chemical Energy Conversion in Mülheim/Ruhr, Germany. We are looking for highly motivated young scientists in the field of Electron Paramagnetic Resonance who are interested in studying biochemical and chemical systems related to the topic of the institute.

#### *In-house projects:*

- Photosynthetic systems (reaction centers, water oxidation);
- Hydrogenase enzymes and related model systems;
- Radical enzymes and protein maquettes.

#### *Collaborative projects:*

- EPR instrumental developments dedicated to EPR studies of (single) protein micro crystals;
- Advanced EPR investigation of highly reactive chemical intermediates and their weakly bound intermolecular complexes.

Our lab is equipped with 10 modern EPR spectrometers covering the frequency range from 2 to 244 GHz capable of the complete repertoire of CW EPR and pulse techniques (ENDOR/TRIPLE, ELDOR, ESEEM) in combination with laser excitation and freeze quench techniques. More details can be found on our website: [www.cec.mpg.de](http://www.cec.mpg.de).

Candidates should have project relevant knowledge and be trained in Magnetic Resonance Spectroscopy, preferably in EPR. Candidates with an interest in EPR instrumental development and microwave engineering are specifically encouraged to apply.

Please send your application including CV and the scope of scientific interests to:

Prof. Wolfgang Lubitz

Max Planck Institute for Chemical Energy Conversion, Stiftstrasse 34-36, 45470 Mülheim an der Ruhr, Germany

e-mail: [wolfgang.lubitz@cec.mpg.de](mailto:wolfgang.lubitz@cec.mpg.de)

#### Faculty position wanted

As an experienced theoretical and experimental physicist, I desire to take my career to the next level through a faculty position. I'd like to focus my research on EPR method development. The marvels of digital revolution, fast arbitrary waveform generators and digitizers,

together with novel data processing methods are about to reshape EPR spectroscopy and imaging. I've been working on a number of projects with the common goal to enhance sensitivity and functionality of CW, rapid scan and pulse EPR. Some of these methods are being implemented into standard commercial instruments and software. I look forward to starting a lab where students from a variety of disciplines: electrical and mechanical engineering, math, biology and chemistry, can contribute to new developments in the methodology and applications of EPR.

Mark Tseytlin (Tseitlin in publications), Research Scientist at the University of Denver. Portfolio: <http://portfolio.du.edu/mtseytli>; e-mail: [mark.tseytlin@du.edu](mailto:mark.tseytlin@du.edu)

#### Post doctoral/Ph.D positions available

Two post doctoral (or Ph.D) positions are available in the laboratory of Prof. Daniella Goldfarb at the department of Chemical Physics, Weizmann Institute, Rehovot, Israel.

The research focus is development of pulse EPR methodology, including distance measurements using standard and new spin labels, and applications of pulse EPR to biological systems.

The positions require background in Magnetic Resonance, and/or Biochemistry/Structural Biology.

Information about the groups and the Weizmann Institute can be found at [www.weizmann.ac.il/chemphys/EPR\\_group](http://www.weizmann.ac.il/chemphys/EPR_group) and [www.weizmann.ac.il](http://www.weizmann.ac.il).

Interested candidates should contact Daniella Goldfarb ([daniella.goldfarb@weizmann.ac.il](mailto:daniella.goldfarb@weizmann.ac.il)) for further information.

For serious suitable candidates the possibility of a funded visit to the lab will be offered prior to final decisions.

#### Bruker BioSpin Corp

Bruker BioSpin Corp is looking for a highly motivated individual to join our EPR Service team to install and support high technology EPR Spectrometer Systems in customer research labs. This individual will install and service our EPR Spectrometer Systems and train customers for basic operation of the equipment. A BS in electrical engineering, electronics or related fields or equivalent experience is required. Experience diagnosing and repairing electronic, electromechanical and/or mechanical equipment is required. General understanding of analog electronics, digital electronics, high voltage circuitry/circuits, microwave technology, vacuum tech-

nology, cryogenics; strong technical skills on analytical instrumentation required.

Please send resume, cover letter and salary requirements to [bruker.jobseprfse0620@bruker-biospin.com](mailto:bruker.jobseprfse0620@bruker-biospin.com)

### EQUIPMENT

#### Design and construction of EPR electronics

The University of Denver can supply electronic design and construction services for EPR applications. Low-noise pulse amplifiers, low-noise 100 kHz preamplifiers, boxcar integrators, and pulse timing systems are available. We also supply a conversion kit to convert Varian field-control units to voltage-controlled scan operation. A 6-digit 1-ppm frequency counter is available in X-, C-, S-, L-band, or MHz versions. Complete microwave/RF bridges from 150 MHz to L-, S-, or C-band are available from designs previously built and tested at the University of Denver. **Please contact:** Richard W. Quine, e-mail: [rquine@du.edu](mailto:rquine@du.edu), phone: 1-303-871-2419

#### For sale: Varian and ESR equipment

Resonance Instruments has available: (1) Replacement klystrons for Varian EPR bridges and some Bruker bridges (at reduced prices) and other klystrons; (2) Resonance Instrument's Model 8320A is a general purpose Hall-effect based magnetic field controller that provides direct control and precise regulation of the magnetic field between the pole pieces of an electromagnet. Its high resolution permits precise adjustment of the magnet's field either through the front panel keyboard or through an RS232 serial interface with your PC.

**Please contact:** Clarence Arnow, President, e-mail: [8400sales@resonanceinstruments.com](mailto:8400sales@resonanceinstruments.com), phone: 1-847-583-1000, fax: 1-847-583-1021.

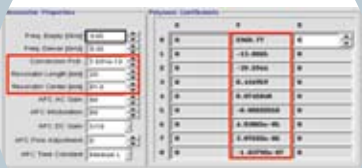
#### Available: Used Varian EPR equipment

(1) Varian E-104 EPR spectrometer with vertical style bridge and e-line fieldial. (2) Varian E-9 EPR spectrometer. Both available with warranty and continued service support. (3) Varian TM cavity with flat cell holders and flat cells. (4) Varian E-257 variable temperature controller with heater sensor and insert holder. (5) Varian E-272B field/frequency lock accessory.

**Please contact:** James Anderson, Research Specialties, 1030 S. Main St., Cedar Grove, WI 53013, USA. phone/fax: 1-920-668-9905, e-mail: [janderson36@wi.rr.com](mailto:janderson36@wi.rr.com)

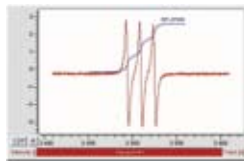
# Single Measurement Quantitative EPR Spin Counting

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Critical resonator properties included

## Double Integration



New tool for high precision double integration

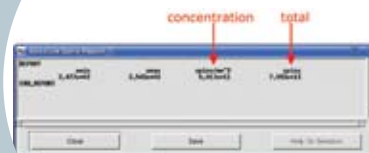
Determine the absolute number of spins in your sample

## User Input



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