epr newsletter

2018
volume 28 number 1-2

The Publication of the International
EPR (ESR) Society

Officers of the International EPR (ESR) Society

PRESIDENT

Thomas Prisner
Institute of Physical and Theoretical Chemistry,
Goethe University
Max-von-Laue-Str. 7, Building N140/Ground Floor
60438 Frankfurt am Main, Germany
phone: +49 (0) 69-798-29406
e-mail: prisner@chemie.uni-frankfurt.de
web: www.prisner.de

VICE PRESIDENTS

Americas

Song-I Han
Dept. of Chemistry & Biochemistry
Bldg 557 Room 1432, UC Santa Barbara
Santa Barbara, CA 93106-9510, USA
phone: 805-893-3504
e-mail: songi@chem.ucsb.edu
web: www.chem.ucsb.edu/hangroup

Asia-Pacific

Hiroshi Hirata
Division of Bioengineering and Bioinformatics, Graduate School of Information Science and Technology, Hokkaido University
North 14, West 9, Kita-ku, Sapporo 060-0814, Japan
phone: +81-11-706-6762
e-mail: hhirata@ist.hokudai.ac.jp
web: www.ist.hokudai.ac.jp/labo/mre/Hirata.html

Europe

Gunnar Jeschke
Dept. of Chemistry and Applied Biosc., ETH Zürich
HCI F 227, Vladimir-Prelog-Weg 1-5/10
8093 Zürich, Switzerland
phone: +41 44-632-57-02
e-mail: gunnar.jeschke@phys.chem.ethz.ch
web: www.epr.ethz.ch

SECRETARY

Aharon Blank
Technion – Israel Institute of Technology,
Haifa 32000, Israel,
phone: +972-4-829-3679, fax: +972-4-829-5948
e-mail: ab359@tx.technion.ac.il

TREASURER

Tatyana I. Smirnova
North Carolina State University, Department of Chemistry,
Campus Box 8204, Raleigh, NC 27695-8204, USA
phone: (919) 513-4375, fax: (919) 513-7353
e-mail: tatyana_smirnova@ncsu.edu

IMMEDIATE PAST PRESIDENT

Hitoshi Ohta
Molecular Photoscience Research Center, Kobe University,
1-1 Rokkodai, Nada, Kobe 657-8501, Japan
phone: +81-78-803-5646, fax: +81-78-803-5770
e-mail: hohta@kobe-u.ac.jp

FOUNDER PRESIDENT

Harold M. Swartz
Dartmouth Medical School,
Department of Radiology & EPR Center,
7785 Vail Room 702, Hanover, NH 03755-3863, USA
phone: 1-603-650-1955, fax: 1-603-650-1717
e-mail: harold.swartz@dartmouth.edu

Fellows of the International EPR (ESR) Society

Anatole Abragam (1914–2011)
John Michael Baker (1930–2017)
Brebis Bleaney (1915–2006)
James R. Bolton
Harvey A. Buckmaster
Anders Ehrenberg
Gareth R. Eaton
Sandra S. Eaton
George Feher
George Fraenkel (1921–2009)
Jack H. Freed
Betty J. Gaffney
Robert Griffin
Edgar Groenen
Erwin Hahn (1921–2016)
Karl Hausser (1919–2001)
Kalman Hideg (1934–2018)
Noboru Hirota
Brian Hoffman
Wayne Hubbell
Clyde A. Hutchison, Jr. (1913–2005)
James S. Hyde
Lowell Kispert
Daniel Kivelson (1929–2003)
Melvin P. Klein (1921–2000)
Harry Kurreck (1932–2015)
Wolfgang Lubitz
August H. Maki (1930–2008)
Harden McConnell (1927–2014)
Bruce R. McGarvey
Keith A. McLauchlan
Klaus Möbius
Yuriy N. Molin
James R. Norris
John R. Pilbrow
Charles P. Poole, Jr. (1927–2015)
Aleksandr M. Prokhorov (1916–2002)
Arnold M. Raitsimring
Kev M. Salikhov
Tengiz I. Sanadze (1930–2011)
Charles P. Scholes
Arthur Schweiger (1946–2006)
Charles P. Slichter (1924–2018)
Sankaran Subramanian
Leslie H. Sutcliffe
Harold M. Swartz
Martyn C. R. Symons (1925–2002)
Takeji Takui
Wolfgang E. Trommer
Yuri D. Tsvetkov
Joan H. van der Waals
George D. Watkins
John A. Weil (1929–2010)
Samuel I. Weissman (1912–2007)
David Whiffen (1922–2002)
Hans C. Wolf

epr newsletter

www.epr-newsletter.ethz.ch

The official publication of the International EPR (ESR) Society is supported by the Society, by corporate and other donors, the Zavoisky Physical-Technical Institute of the Russian Academy of Sciences, Kazan, Russian Federation, and the Swiss Federal Institute of Technology, Zürich, Switzerland.

EDITOR
Laila V. Mosina
Zavoisky Physical-Technical Institute
Russian Academy of Sciences
Kazan, Russian Federation
mosina@kfti.knc.ru

ASSOCIATE EDITORS
Candice S. Klug
Medical College of Wisconsin
Milwaukee, WI, USA
candice@mcw.edu
Hitoshi Ohta
Molecular Photoscience Research Center,
Kobe University, Kobe, Japan
hohta@kobe-u.ac.jp
Sabine Van Doorslaer
University of Antwerp, Antwerp, Belgium
sabine.vandoorslaer@uantwerpen.be

TECHNICAL EDITOR
Sergei M. Akhmin
Zavoisky Physical-Technical Institute
Russian Academy of Sciences
Kazan, Russian Federation
akhmin@kfti.knc.ru

FOUNDING EDITOR
R. Linn Belford
Illinois Research Center, University of Illinois
at Urbana, Urbana, IL, USA
rbelford@uiuc.edu

EDITORIAL OFFICE
Zavoisky Physical-Technical Institute
Russian Academy of Sciences
Sibirsky trakt 10/7, Kazan 420029
Russian Federation
phone: 7-843-2319096
fax: 7-843-2725075

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

The *EPR newsletter* is published quarterly by the International EPR (ESR) Society and is available in electronic and printed form to all members of the Society. The deadlines for submission of news for upcoming issues: Spring March, 15; Summer June, 15; Fall September, 15; Winter December, 15.

ISSN 1094-5571

PRINTING: LaPlume and Sons Printing, Inc.
One Farley Street, Lawrence MA 01843 USA
phone: (978) 683-1009, fax: (978) 683-4594

epr news letter

The Publication of the International EPR (ESR) Society

volume 28 number 1-2 2018

The cover is the fiftieth hard cover of the *EPR newsletter*, which includes all forty-nine previous covers.

IES business

Guest of the issue

Present meets future

New EPR faculty

Software

New books and journals

Tips & techniques

In memoriam

continued on page 2

Eidgenössische Technische Hochschule Zürich
Swiss Federal Institute of Technology Zurich

Conference reports

Readers corner

Editorial

Dear colleagues,

Three numbers, fifty, thirty and fifteen, are key ones in this double issue. To start with 30: 2017 marked thirty years of the *EPR newsletter*, the official publication of the International EPR (ESR) Society (IES). Linn Belford, the founding editor of the *EPR newsletter* from 1987 until 2002, recalled the history of this publication in his Letter of the Editor (12/4, p. 1, 2002). The newsletter preceded the foundation of the Society by about two years and was quickly recognized as being a most appropriate organ for the new Society.

15: In the end of 2002, the Editorial office of the newsletter was moved to Kazan, the birthplace of electron paramagnetic resonance, and our first issue of the *EPR newsletter*, and my first as Editor, appeared fifteen years ago in 2003.

We are pleased to feature in this issue the newly elected IES CEOs for the term January 1st, 2018 until December 31st, 2020 (pp. 3–6). Special greetings to Thomas Prisner, IES President, a former long-term Associate Editor of the *EPR newsletter* and Editor of the Pro and Contra column (see, e.g., 20/4, pp. 23–26).

Our newsletter team have worked hard to include most of our regular columns in this issue: Wolfgang Lubitz (Guest of the Issue) gave the floor to Thomas Prisner on dynamic nuclear polarization at high magnetic fields; Sabine Van Doorslaer (Present Meets Future) interviewed John McCracken and Matthew Krzyaniak; Candice Klug (New EPR Faculty) introduced a cohort of brilliant young researchers: Alexey Silakov, Joseph Zadrozny, Michael Lerch and Sebastian Stoian; Stefan Stoll (Software) gave the floor to Gregor Hagelueken on a software package to interpret PELDOR/DEER distance distributions in terms of macromolecular structure; Keith Earle (Tips and Techniques) invited Kalina Ranguelova and Ralph Weber to overview some applications of EPR in the pharmaceutical field; John Pilbrow (EPR newsletter Anecdotes) shifted the story about EPR in Slovakia and Austria by Georg Gescheidt et al. to 28/3. Evhen Polyhach, our highly responsible webmaster, ensures prompt updating of our website.

50: For this issue the fiftieth color hard cover of the *EPR newsletter* included all forty-nine previous covers (special thanks go to Peter Höfer (Bruker) for the permission to make use of the back cover initially reserved for a Bruker ad). Just looking at the covers exquisitely designed by Sergei Akhmin, our technical editor, one gets the idea about the boundless diversity of the applications of magnetic resonance in different fields of science mainly illustrating the research of laureates of magnetic resonance awards. The cover, originally printed in color only on the front and back, is paid for by Bruker. After some time producing the newsletter our long-term printers, LaPlume and Sons Printing was able to make a minor printing adjustment allowing for full color on the entire cover at no additional cost. Switching to a silk coated stock also improved the printability and look of the newsletter helping to make it what it is today.

To make a soft reminder, 2019 marks the thirtieth anniversary of the IES. It is high time to discuss how to celebrate this event in the newsletter (you may like to have a look at 19/1-2 dedicated to the twentieth anniversary of the IES). Your proposals are welcome!

Laila Mosina

Letter of the President

Dear Colleagues,

I am happy to announce that the International EPR(ESR) Society has elected a new Executive Board for the 2018–2020 term. I, Thomas Prisner, am happy to serve as the new elected President of IES. The three new elected vice presidents for America, Asia and Europe are Song-I Han (University of California, Santa Barbara), Hiroshi Hirata (Hokkaido University) and Gunnar Jeschke (ETH Zürich), respectively. Aharon Blank (Technion Haifa) will continue as Secretary for the society and Christopher Boehme (University of Utah) will take over the job as Treasurer from Tatyana Smirnova (North Carolina State University). Of course, Laila Mosina (Zavoisky Institute Kazan) will continue as editor of the *EPR newsletter* and Hithosi Ohta (Kobe University) will stay in the Executive Board as Immediate Past President. I am very happy to have such competent colleagues in the Board and hope very much that we can help our EPR society grow and become even more visible in the future.

I also want to thank the officers of the last period for their dedication and engagement for IES. Elena Bagryanskaya, Graham Smith and Stephen Hill as Vice Presidents for Asia, Europe and America, respectively. Special thanks of course to Tatyana Smirnova (North Carolina State University), who did an excellent job as Treasurer for the IES. I am of course also very grateful that with Aharon Blank as Secretary, Laila Mosina as Editor of the *EPR newsletter* and Hitoshi Ohta, as former President of our society, three former Executive Board Members stay in the new Board. This will guarantee continuity and help us, the new Board members, a lot to get started!

The International EPR(ESR) Society is the only organization that explicitly represents our method and science on an international level. Of course, some countries have their own local EPR societies. However, some do not, because EPR is still a small research field compared to other techniques such as optical spectroscopy or microscopy, mass spectrometry or NMR. Therefore, I believe IES should and can be the home base for all of us. Over the years, EPR has grown into many diverse and specialized fields of applications, ranging from analytical applications to detailed characterization of paramagnetic molecules or defects in material science and biology, to the investigation of light or chemical driven reactions in catalysis and biology. This broad range of new applications still drives new methodical developments, as for example high field EPR, rapid scan techniques, microcircuits designs, optical single molecule detection or medical imaging. Due to this broad and diverse application range, we tend to go to specialized conferences in our respective application fields. However, I believe that the interaction between all the different branches is very fruitful in advancing further developments in the field of EPR. That is what connects us and encourages us to go to EPR conferences. As important as our internal interaction is a strong interaction and continuous exchange with our NMR colleagues. We share not only the same theory and physical principles but also very much in methodology. Many important techniques couple both research fields, as for example dynamic nuclear polarization (DNP), Electron-Nuclear

▶

Thomas Prisner, IES President

Thomas Prisner received his Diploma in Physics from the University of Heidelberg in 1983, where he performed his Master Thesis at the Max-Planck Institute for Medical Research. He earned his PhD at the University of Dortmund in 1989. After that he spend two years as postdoctoral researcher at M.I.T. in Cambridge, U.S.A. In 1996 he habilitated at the Free University of Berlin. Since 1996 he holds a full professorship at the Institute of Physical and Theoretical Chemistry at the Goethe University in Frankfurt. His research focuses on the one hand on methodological developments in the field of EPR and DNP and, on the other hand, on applications of pulse EPR methods to biomolecules, as membrane proteins and nucleic acids. Much of this work is part of collaborative research centers located at the Goethe University and the Center of Biomolecular Magnetic Resonance Frankfurt, where he is one of the founding members. Currently he serves as editorial board member of *Journal of Magnetic Resonance*, advisory board of *Applied Magnetic Resonance*, managing director of the Rolf-Sammet board of trustees, Chair of the EUROMAR board of trustees and member of the chemistry board of the German Research Society. In the past he served as Chair of the Department of Chemistry and as Chair of the Department of Biochemistry, Chemistry and Pharmacy at the Goethe University, as board member of the German Magnetic Resonance Society and chair of the German EPR subgroup therein. He organized a number of conferences and schools: the EUROMAR meeting in Frankfurt, together with the European Federation of EPR Groups meeting and the German Magnetic Resonance Society Discussion Meeting, the DNP Symposium in Königstein and an EPR summer school in Naurod. He received a number of awards: the Philipp Morris Research Award, the IES Silver Medal in Physics/Material Sciences, the Bruker Lecture Award, the Zavoisky Award and is fellow of ISMAR.

Letter of the President

Double Resonance (ENDOR), Paramagnetic Relaxation Enhancement (PRE), contrast agents in MRI or NV centers as single molecule detectors. Nowadays, also an increasing range of applications profit from combining NMR and EPR, as for example structure determination of large biomolecular complexes. Thus, I very much like magnetic resonance conferences covering all flavors of magnetic resonance ranging from liquid and solid state NMR, to DNP, MRI, ODMR and of course EPR! I know that many of our NMR colleagues agree and very much appreciate our contribution to such conferences. That the board of trustees of EUROMAR elected me as Chair of EUROMAR for the next three years underlines this point. Of course, I would like to see even more EPR on such joint conferences, presented from senior scientists as well as our young researchers! I think this is very important for our research, but even more for the careers of our young scientists. The probability that an NMR scientist is part of a hiring and selection committee for a research position is rather large! EPR will always be only a smaller part of such conferences; but I believe that we can contribute much to the developments in many fields of magnetic resonance, as exemplified above! IES can intensify our visibility on such conferences by poster awards and other contributions, similar to what IES is doing at the EPR conferences worldwide. For me, that is one important point IES can provide for us. The society is not the Executive Board but the members. Therefore, I hope seeing many of you actively supporting the IES. Either by officially joining the society, by paying the dues or by interacting and discussing with us Board members what we can do to make the IES even more attractive for all of us. I would especially like to encourage our young scientists to become active members of the society. We have to ask ourselves what benefits make IES attractive for them.

I hope very much that together with all the other Officers of the IES, I will keep our Society attractive for all of you, hopefully succeed in attracting new members and sponsors and continue to make our society visible and respectable to other scientific communities. It is important to raise the awareness of our research field to scientists outside of our own research field, to scientific granting agencies as well as to hiring committees. I believe that for such issues the IES can be very helpful and important!

Of course, there are many other things the IES society does, for example the prices and fellowships the society awards every year, the poster prices it awards at conferences to PhD students with EPR posters and of course the *EPR newsletter*, beautifully edited by Laila, which keeps us all informed about what is going on. Maybe you have some additional thoughts on what our society could do. If you have any suggestions, wishes, ideas: do not hesitate to contact me via email (Prisner@Chemie.Uni-Frankfurt.de) or talk to me when you see me on conferences! I am very much interested in listening to your thoughts. We should all feel well represented by the IES and see it as our scientific family - his is my hope.

Best regards and hope to see you soon somewhere.

Thomas Prisner

New IES Executives

Song-I Han, IES Vice-President Americas

Song-I Han received her undergraduate degree at the University of Koeln, and her Doctoral Degree in Natural Sciences (Dr.rer.nat) from Aachen University of Technology (RWTH), Germany, in 2001. She pursued her postdoctoral studies at the Max-Planck Institute for Polymer Research, Mainz, Germany sponsored by the Max-Planck Fellowship and the University of California Berkeley sponsored by the Feodor Lynen Fellowship of the Alexander von Humboldt Foundation. Dr. Han joined the faculty at University of California Santa Barbara (UCSB) in 2004, received tenure in 2010, and was promoted to full professor in 2012. She is currently a Professor in the Department of Chemistry and Biochemistry, as well as the Department of Chemical Engineering at UCSB. She is a recipient of the 2004 Camille and Henry Dreyfus New Faculty Award, the 2007 NSF Faculty Early Career Development Award, the 2008 Packard Fellowship for Science and Engineering, the 2010 Dreyfus-Teacher Scholar Award, the 2011 NIH Innovator Award and the 2015 Bessel Prize of the Alexander von Humboldt Foundation. She is currently serving on the Editorial board for the *Journal of Magnetic Resonance*.

Her lab focuses on the development of dynamic nuclear polarization (DNP) instrumentation and methods that amplifies the nuclear magnetic resonance (NMR) signal by several orders of magnitudes. One of her group's contribution is to develop Overhauser DNP at 10 GHz under ambient solution conditions as a tool to measure site-specific surface water diffusivity on a protein surface with tens to hundreds of picosecond correlation times. She also pursues the development of solid-state DNP under magic angle spinning at 200 GHz and under cryogenic temperatures, with concurrent electron paramagnetic resonance (EPR) capabilities and arbitrary waveform generation (AWG) pulse shaping capabilities. The questions of the Han lab are directed broadly towards unraveling the functional role of protein- and surface-coupled water, the mechanism of aggregation

▶

The EPR community has available to it a list server. The address is epr-list@xenon.che.ilstu.edu.
To subscribe to the list, send the words SUBSCRIBE epr-list to majordomo@xenon.che.ilstu.edu.
That sends a message to Reef Morse who will then manually place you on the list. This honors only legitimate requests to join the list. Reef also moderates the list which keeps it spam-free.

and seeding, specifically of the intrinsically disordered protein, tau, in a neurodegenerative disease-relevant state, as well as the fluid-fluid phase separation of protein polyelectrolytes to complex coacervates. In this quest, her lab considers the combined EPR, DNP and NMR a necessary and powerful approach to track complex and dynamic macromolecular systems and processes.

Hiroshi Hirata,
IES Vice-President Asia-Pacific

Hiroshi Hirata graduated from Tohoku Institute of Technology, Japan, in 1988. He received his M.Eng. degree in electrical engineering from Yamagata University, Japan, in 1990, and his Ph.D. degree in applied electronics from Tokyo Institute of Technology, Japan, in 1993.

After finishing the Ph.D. program, he joined the EPR instrument development group at the Department of Electrical Engineering, Yamagata University as a Research Associate, and became an Associate Professor and a Professor. Since 2008, he has joind the Division of Bioengineering and Bioinformatics, Graduate School of Information Science and Technology, Hokkaido University, Japan, as a Professor. From 1999 to 2000, he served as a Research Associate in the EPR Center for the Study of Viable Systems (Director: Prof. Harold M. Swartz), the Department of Radiology, Geisel School of Medicine at Dartmouth, Hanover, New Hampshire, USA. His research interests include RF/microwave engineerting in EPR, EPR spectroscopy/imaging for biomedical applications, and visualiation of tumor tissue oxygenation and metabolism using low-field EPR. He was a recepient of 2003 Young Investigator Award of the Society of Electron Spin Science and Technology (SEST) in Japan.

Gunnar Jeschke,
IES Vice-President Europe

Gunnar Jeschke is Professor for Electron Spin Resonance at ETH Zürich, Switzerland. Born in 1966, he received a diploma in chemistry with Lohmann medal from Technical University Dresden with work in solid-state NMR. After a one-year research stay an RIKEN, Wako-shi, Japan, working on magnetic field effects on photochemical reactions, he studied with the late Professor Arthur Schweiger at ETH Zürich. In 1996, he received his doctoral degree with a thesis entitled "New Concepts in Solid-State Pulse Electron Spin Resonance" and the IES Young Investigator Award. After a brief return to solid-state NMR at University of Bonn, Germany, he joined the Max Planck Institute for Polymer Research in Mainz, Germany in 1998 as a project leader for Electron Spin Resonance. Together with Arthur Schweiger he published the monograph "Principles of Pulse Electron Paramagnetic Resonance" in 2001 and he defended his Habilitation in 2003. He was appointed full professor of Physical Chemistry at University of Konstanz, Germany in 2006 and moved to ETH Zürich in 2008.

He served as Secretary General of Groupement AMPERE from 2006–2018, as Editor of *Molecular Physics* as well as Associate Editor of the *Journal of Magnetic Resonance* since 2011, is an ISMAR Council member since 2013 and was Chair of the Scientific Committee of the ISMAR Conference 2017. In 2003 he was awarded a Dozentenstipendium of the German Chemical Industry Fund, in 2009 the Bruker Prize of the RSC Electron Spin Resonance Group, in 2014 he shared the International Zavoisky Award with Thomas Prisner, and in 2017 he received the IES Silver Medal for Instrumentation.

His current research profile encompasses the development of new experiments in and instruments for pulsed EPR, in particular using wideband shaped-pulse excitation, integrative modelling in structural biology based on nanometer-range distance distribution restraints, and the application of EPR spectroscopy in biology and catalysis.

Aharon Blank,
IES Secretary

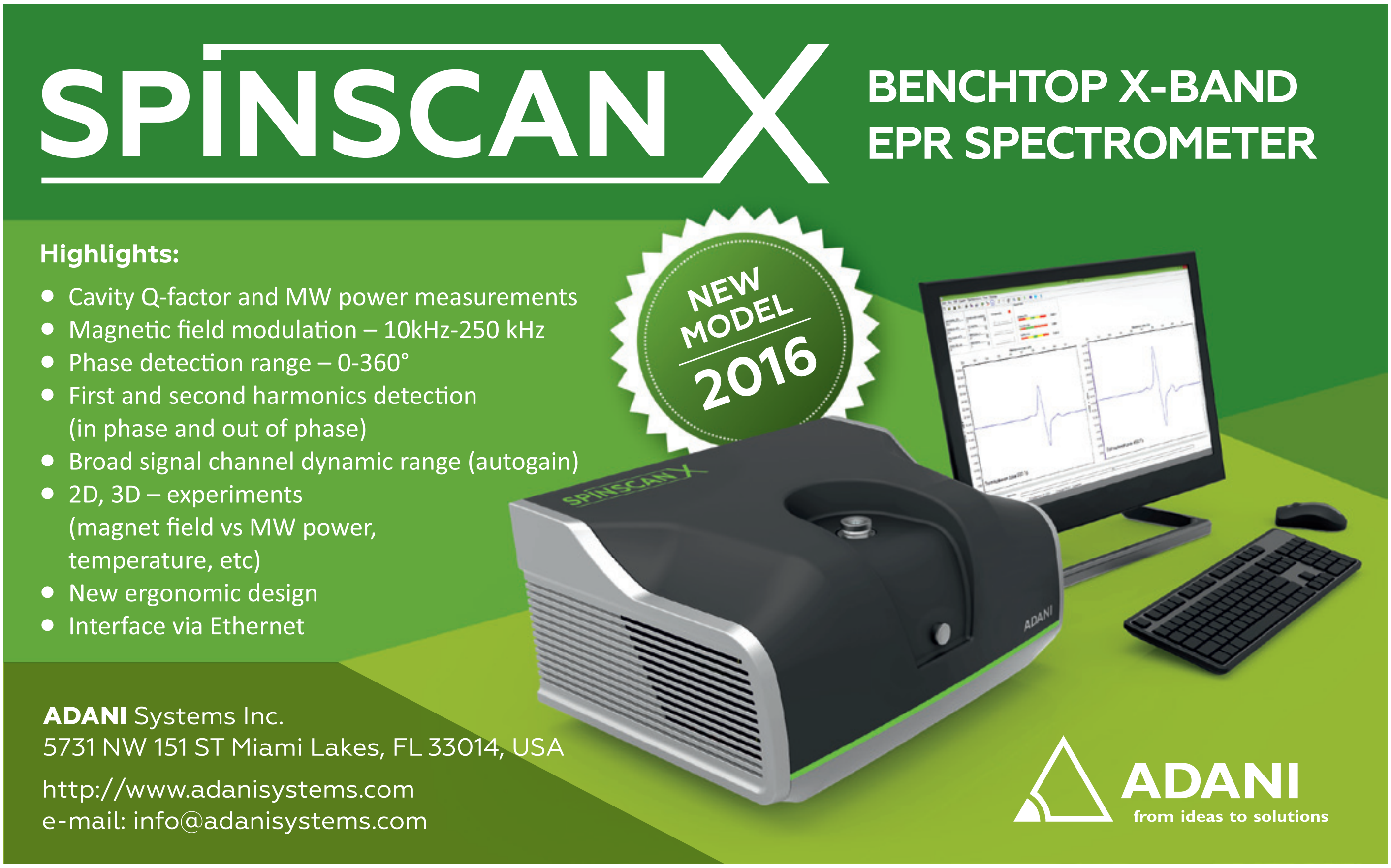
Aharon Blank is an associate Prof. at the Schiluch Faculty of Chemistry, Technion – Israel Institute of Technology. Born in 1972, graduated from the Hebrew University of Jerusalem in 1992 with degrees in Mathematics, Physics and Chemistry; Completed his Master degree at Tel Aviv University in 1997 in electrical engineering – Physical elec-

▶

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 tronics under the supervision of Prof. Raphael Kastner and finished his PhD in 2002 at the Hebrew University of Jerusalem in Physical Chemistry – Electron Spin Resonance (ESR), under the supervision of the late Prof. Haim Levanon. During this time he served 9 years in the IAF as a Scientific Officer and also as a CTO in a medical device company, developing miniature intravascular MRI. Following his PhD he spent 3 years at Cornell University as a Post Doc at the group of Prof. Jack Freed (on a Rothschild post-doctoral fellowship), developing the subject of ESR microscopy, and since 2005 he is a Faculty member at the Technion. Aharon main interests today are development and applications of new methodologies in the field of magnetic resonance. His group works on miniature sensitive ESR resonators; small, self contained NMR and ESR medical tools; ESR probes for micro and nano imaging; and ESR and NMR approaches to quantum technology.

Christoph Boehme, IES Treasure

Christoph Boehme is a Professor at the Department of Physics and Astronomy at the University of Utah. He started his undergraduate studies majoring in physics in Heidelberg, Germany in 1994. He won a Fulbright Graduate Student Exchange Scholarship in 1997 which brought him to Raleigh, North Carolina. In 2000 he moved to Berlin, Germany where he worked for the Hahn-Meitner Institute. He finished his dissertation work as a graduate student at the University of Marburg in 2002 while still working in Berlin where he spent an additional three years after graduation to work as a postdoctoral researcher. He moved to Utah in 2006 to join the U as Assistant Professor. He was promoted into the rank of Associate Professor and awarded tenure in 2010, promoted to the rank of Professor in 2013 and he served as Associate Chair of the Department of Physics and Astronomy from July 2010 until August 2015. His research interests have been focused on the nature of spin-dependent electronic transitions in condensed matter as well as their applications to spintronics and spin-based quantum information concepts. As of April 6th, 2018, Christoph had to resign from his IES treasurer position due to an ad-hoc assignment to an executive role at the University of Utah that he needed to assume on very short notice. Tatyana Smirnova (*EPR newsletter*, 24/4, p. 5) has stepped in for now as acting treasurer until a new elected treasurer will have been found.



Dynamic Nuclear Polarization at High Magnetic Fields

Thomas Prisner

Dynamic nuclear polarization (DNP) is a fast expanding methodology in the field of magnetic resonance (MR). It also serves as exciting example demonstrating how vivid and exciting MR still is, more than 70 years after its discovery and after receiving several Nobel prizes. In fact, DNP belongs to one of the very early methodic discoveries in the field of MR. Shortly after the theoretical proposal by Overhauser in 1953 [1] that NMR signals of metals should be strongly increased by microwave excitation of the EPR transitions, this concept was experimentally proven by Carver and Slichter [2]. A historical review on these early experiments appeared in a special PCCP issue on DNP [3]; highly recommendable to read for everybody ever involved in setting up a new experiment. Soon, this new concept was extended to insolating solids [4] and to liquids [5] in the presence of paramagnetic molecules. Theoretical models explaining the polarization transfer from the unpaired electron spin system to the nuclear spins were developed for all the different mechanisms and cases [6–10]. They quantitatively describe the NMR signal enhancements obtained in DNP experiments under very different sample conditions: metals and diamagnetic solids at very low cryogenic temperatures as well as liquid solutions containing organic radicals measured at room temperature. Despite the very different conditions and mechanisms responsible for the polarization transfer, all theoretical methods predicted a steep loss in DNP efficiency by going to higher resonance RF frequencies for the nuclear spins (and respectively microwave frequencies for the coupled electron spin). For DNP referring to the Overhauser effect (metals and liquids) the spectral density of the electron-nuclear spin cross-relaxation rates mediating the polarization transfer steeply decreases at higher frequencies. For the solid effect, cross effect and thermal mixing the efficiency of the microwave driven forbidden electron-nuclear spin transitions strongly decreases at higher magnetic field values. Therefore, as NMR moved to higher and higher magnetic fields to obtain better chemical shift resolution and higher Boltzmann sensitivity, the DNP method became almost completely forgotten. Only few research groups continued further exploring the potential of these methods [11–13]. The situation changed drastically by two new approaches: solid state DNP at high magnetic fields under MAS conditions explored in the 1990's by Griffin [14] and solid state DNP obtained at very low temperatures and then very fast transferred to the liquid state by dissolution of the sample, invented in the 2000's by Ardenkjaer-Larsen [15].

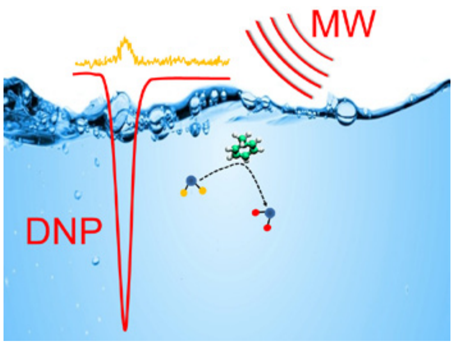
For solid-state DNP under MAS conditions performed at about 100 K a new high power gyrotron microwave source was crucial to obtain high NMR enhancements (>300) for typical NMR conditions. In the second case, a fast dissolution process to liquid was developed and allowed transfer of the sample for spectroscopic or imaging applications with very high ^{13}C polarization gains (resulting from the combined gain obtained from the DNP mechanism and the very low polarization temperature). Both of these new instrumental developments were fast recognized and highly appreciated by the scientific community. Nevertheless it took several years (until commercial instruments became available), before the full potential of DNP at high magnetic fields became obvious. A first DNP symposium, organized by Köckenberger in Nottingham in 2007, brought together many scientists just building up their own DNP setups and researchers from different potential application areas keen to explore the potential of DNP [16]. This created a new boom and enthusiasm to this more than 50-year-old method!

After that, new DNP methodical developments, theoretical descriptions and a vastly increasing range of applications became an integral part at magnetic resonance conferences. The detailed mechanism of the polarization transfer from the rare paramagnetic centers to the much more abundant nuclear spins, either directly or via spin diffusion, in static samples at low temperatures or in rotating solids at 100 K were subject of many theoretical studies [17–21]. They showed that the mechanism is indeed rather complex – somewhat surprising that the DNP experiment works so efficiently anyway!

Most striking is probably the large amount of applications published nowadays that rely on DNP experiments. They are ranging from the observation of fast chemical reactions [22], to investigations of membrane proteins [23–25], large protein aggregates [26–28], surfaces [29, 30], micro-crystals [31] and cell membranes [32], using different types of paramagnetic agents [33–35] as well as several low-gamma nuclear spins [36–38]. Much of this is reviewed in recent articles [39–42]. Liquid-state Overhauser DNP lags somewhat behind these rapid developments, despite the fact that large DNP enhancements at high magnetic fields have been reported for proton [43–47] as well as for carbon nuclear spins [48]. More recently, Overhauser DNP has even been observed in insulating solid samples [49–51] at high magnetic fields.

Thus, it can be stated, that DNP indeed became a success story over the years. Despite the fast growing field of interesting new applications areas, instrumental [52], methodic [53] and synthetic [54] optimizations are still ongoing in many laboratories and companies. In addition, a theoretical understanding of the mechanism is still under work and may lift some of the still existing limitations (as temperature, magnetic field strength and spinning speed). I personally find the field of DNP also very exciting and appealing from a more general point of view: DNP brings together all the different skills and expertises in MR, technical and methodical skills, theoretical spin physics and statistical thermodynamics, EPR and

▶



Liquid state Overhauser DNP enhancement of 40 mM TEMPOL in liquid water. Enhancements of up to –80 have been observed at 9.4 T magnetic field (corresponding to 260 GHz MW frequency). For more details see [55].

NMR, spectroscopy and imaging, medicine, structural biology and material sciences. All these expertises are necessary to optimize the efficiency of DNP at high magnetic fields for various applications. Thus, an intense discussion between all the different branches of magnetic resonance exists on this exciting and challenging topic! I very much enjoy these broad discussions as they for example evolved in the COST Action on *Hyperpolarization Physics and Methodology in NMR and MRI*, which also included all the other hyperpolarization methods (as for example PHIP and optically induced hyperpolarization). DNP brings together almost all of the aspects of MR and is thus very well suited for teaching courses; a holographic view of the beauty of magnetic resonance.

1. A. W. Overhauser, Phys. Rev., 1953, 92, 411–415.
2. T. R. Carver, C. P. Slichter, Phys. Rev., 1953, 92, 212–213.
3. C. P. Slichter, PCCP, 2010, 12, 5741–5751.
4. A. Abragam, W. G. Proctor, Comptes Rendus Hebdomadaries des Seances de L'Academia des Sciences, 1958, 246, 2253–2256.
5. K. H. Hausser, D. Stehlik, Adv. Magn. Reson., 1968, 3, 79.
6. A. V. Kessenikh, A. A.Manenkov, Soviet Phys. Solid State, 1963, 5, 835–837.
7. C. F. Hwang, D. A. Hill, Phys. Rev. Lett., 1967, 19, 1011–1013.
8. M. Goldman, Spin Temperature and Nuclear Magnetic Resonance in Solids, Oxford University Press, London, 1970.
9. A. Abragam, M. Goldman, Rep. Prog. Phys., 1978, 41, 395–467.
10. W. Müller-Warmuth, K. Meise-Gresch, Adv. Magn. Reson., 1983, 11, 1–45.
11. R. A. Wind, M. J. Duijvestijn, C. Vanderlugt, A. Manenschijn, J. Vriend, Prog. Nuc. Mag. Res. Spec., 1985, 17, 33–67.
12. M. Afeworki, R. A. McKay, J. Schaefer, Macromol., 1992, 25, 4084–4091.
13. H. C. Dorn, R. Gitti, K. H. Tsai, T. E. Glass, Chem. Phys. Lett., 1989, 155, 227–232.
14. L. R. Becerra, G. J. Gerfen, R. J. Temkin, D. J. Singel, R. G. Griffin, Phys. Rev. Lett., 1993, 71, 3561–3564.
15. J. H. Ardenkjaer-Larsen, B. Fridlund, A. Gram, G. Hansson, L. Hansson, M. H. Lerche, R. Servin, M. Thaning and K. Golman, PNAS, 2003, 100, 10158–10163.
16. T. F. Prisner, W. Koeckenberger, Appl. Magn. Reson., 2008, 34, 213–218 and papers in this special DNP issue.
17. K-N. Hu, G. T. Debelouchina, A. A. Smith, R. G. Griffin, J. Chem. Phys. 2011, 134, 125105.
18. Y. Hovav, A. Feintuch, S. Vega J. Magn. Reson., 2010, 207, 176–189.
19. K. R. Thurber, R. Tycko, J. Chem. Phys., 2012, 137, 084508.
20. F. Mentink-Vigier, Ü. Akbey, Y. Hovav, S. Vega, H. Oschkinat, A. Feintuch, J. Magn. Reson., 2012, 224, 13–21.
21. A. Karabanov, A. van der Drift, L. J. Edwards, I. Kuprov, W. Köckenberger, PCCP, 2012, 14, 2658–2668.
22. S. Bowen, C. Hilty, PCCP, 2010, 12, 5766–5770.
23. M. Rosay, A-C. Zeri, N. S. Astrof, S. J. Opella, J. Herzfeld, R. G. Griffin, JACS, 2001, 123, 1010–1011.
24. L. Reggie, J. J. Lopez, I. Collinson, C. Glaubitz, M. Lorch, JACS, 2011, 133, 19084–19086.
25. J. Becker-Baldus, C. Bamann, K. Saxena, H. Gustmann, L. J. Brown, R. C. D. Brown, C. Reiter, E. Bamberg, J. Wachtveitl, H. Schwalbe, C. Glaubitz, PNAS, 2015, 112, 9896–9901.
26. M. J. Bayro, G. T. Debelouchina, M. T. Eddy, N. R. Birkett, C. E. MacPhee, M. Rosay, W. E. Mass, C. M. Dobson, R. G. Griffin, JACS, 2011, 133, 13967–13974.
27. I. V. Sergeyev, L. A. Day, A. Goldbourt, A. E. McDermott, JACS, 2011, 133, 20208–20217.
28. A. Potapov, W-M-Yau, R. Ghirlando, K. R. Thurber, R. Tycko, JACS, 2015, 137, 8294–8307.
29. A. Lesage, M. Lewlli, D. Gajan, M. A. Caporini, V. Vitzthum, P. Miéville, J. Alauzun, A. Roussey, C. Thieuleux, A. Mehdi, G. Bodenhausen, C. Copéret, L. Emsey, JACS, 2010, 132, 15459–15461.
30. A. Zagdoun, A. J. Rossini, D. Gajan, A. Bourdolle, O. Quari, M. Rosay, W. E. Maas, P. Tordo, M. Lelli, L. Emsley, A. Lesage, C. Copéret, Chem. Commun., 2012, 48, 654–656.
31. A. J. Rossini, A. Zagdoun, F. Hegner, M. Schwarzwälder, D. Gajan, C. Copéret, A. Lesage, L. Emsley, JACS, 2012, 134, 16899–16908.
32. H. Takahashi, I. Ayala, M. Bardet, G. de Paepe, J-P. Simorre, S. Hediger, JACS, 2013, 135, 5105–5110.
33. C. Sauvöe, M. Rosay, G. Casano, F. Aussenac, R. T. Weber, O. Quari, P. Tordo, Angewand. Chem., 2013, 52, 10858–10861.
34. B. Corzilius, A. A. Smith, A. B. Barnes, C. Luchinat, I. Bertini, R. G. Griffin, JACS, 2011, 133, 5648–5651.
35. M. Kaushik, M. Qi, A. Godt, B. Corzilius, Angew. Chem., 2017, 134, 4359–4363.
36. A. Rossini, A. Zagdoun, M. Lelli, D. Gajan, F. Rascón, M. Rosay, W. E. Maas, C. Copéret, A. Lesage, L. Emsley, Chem. Sci., 2012, 3, 108–115.
37. V. Vitzthum, P. Miéville, D. Carnevale, M. A. Caprini, D. Gajan, C. Copéret, M. Lelli, A. Zagdoun, A. J. Rossini, A. Lesage, L. Emsley, G. Bodenhausen, Chem. Commun., 2012, 48, 1988–1990.
38. F. Blanc, L. Sperrin, D. A. Jefferson, S. Pawsey, M. Rosay, C. P. Grey, JACS, 2013, 135, 2975–2978.
39. Q. Z. Ni, E. Daviso, T. V. Can, E. Markhasin, S. K. Jawla, T. M. Swager, R. J. Temkin, J. Herzfeld, R. G. Griffin, Acc. Chem. Res., 2013, 46, 1933–1941.
40. A. J. Rossini, A. Zagdoun, M. Lelli, A. Lesage, C. Copéret, L. Emsley, Acc. Chem. Res., 2013, 46, 1942–1951.
41. K. R. Keshari, D. M. Wilson, Chem. Soc. Rev., 2014, 43, 1627–1659.
42. A. S. L. Thankamony, J. J. Wittmann, M. Kaushik, B. Corzilius, Prog. Nuc. Mag. Res. Spec. 2017, 102-103, 120–195.
43. P. Höfer, G. Parigi, C. Luchinat, P. Carl, G. Guthausen, M. Reese, T. Carlomagno, C. Griesinger, M. Bennati, JACS, 2008, 130, 3254–3255.
44. M. J. Prandolini, V. P. Denysenkov, M. Gafurov, B. Endeward, T. F. Prisner, JACS, 2009, 131, 6090–6092.
45. E. V. Kryukov, M. E. Newton, K. J. Pike, D. R. Bolton, R. M. Kowalczyk, A. P. Howes, M. E. Smith, R. Dupree, PCCP 2010, 12, 5757–5765.
46. J. A. Villanueva-Garibay, G. Annino, P. J. M. van Bentum, A. P. M. Kentgens, PCCP, 2010, 12, 5846–5849.
47. C. Griesinger, M. Bennati, H. M. Vieth, C. Luchinat, G. Parigi, P. Höfer, F. Engelke, S. J. Glaser, V. Denysenkov, T. F. Prisner, Prog. Nuc. Mag. Res. Spec., 2012, 64, 4–28.
48. G. Liu, M. Levien, N. Karschin, G. Parigi, C. Luchinat, M. Bennati, Nature Chem., 2017, 9, 676–680.
49. O. Haze, B. Corzilius, A. A. Smith, R. G. Griffin, JACS, 2012, 134, 14287–14290.
50. M. Lelli, S. R. Chaudhari, D. Gajan, G. Casano, A. J. Rossini, O. Quari, P. Tordo, A. Lesage, L. Emsley, JACS, 2015, 137, 14558–14561.
51. S. Pylaeva, K. L. Ivanov, M. Baldus, D. Sebastiani, H. Elgabarty, J. Phys. Chem. Lett., 2017, 8, 2137–2142.
52. M. Rosay, M. Blank, F. Engelke, J. Magn. Reson., 2016, 264, 88–98.
53. S. K. Jain, G. Mathies, R. G. Griffin, J. Phys. Chem., 2017, 147, 164201.
54. C. Sauvöe, G. Casano, S. Abel, A. Rockenbauer, D. Akhmetzyanov, H. Karoui, D. Siri, F. Aussenac, W. Maas, R. T. Weber, T. Prisner, M. Rosay, P. Tordo, P. O. Ouari, Chem. Eur. J., 2016, 22, 5598–5606.
55. T. Prisner, V. Denysenkov, D. Sezer, J. Magn. Reson., 2016, 264, 68–77.

PRESENT MEETS FUTURE

Edited by
Sabine Van Doorslaer

Same questions, but always different stories. In each 'Present meets future' feature, I am confronting the dreams and expectations of a young EPR scientist with those of his/her mentor. This time, we meet Matthew Krzyaniak, research associate at Northwestern University (USA), and, John McCracken, professor at Michigan State University (USA), who was his PhD advisor.

How and when did you start your career? Was it a childhood's dream to become a scientist, or was it a consequence of fortuitous choices? What attracted you in EPR?

John: My interest in science dates back to when I was in high school during the early 1970's. I had always been interested in machines and wanted to know how everything worked. I also liked math and puzzles. My high school physics teacher, Mr. Cochran, also taught chemistry. He insisted that we take physics in preparation for chemistry, or that we take a combined physics/chemistry course over a two-year period. These ideas were novel at the time and are still not accepted by most high school instructors in the US. The best part about both of these classes was that Mr. Cochran had developed weekly laboratory experiments to reinforce what was being taught. I really enjoyed both the laboratory and classroom portions of these courses and I was hooked. I became interested in physical chemistry and spectroscopy as an undergraduate at the University of Illinois. As a junior, I took a spectroscopy lab where we did lots of the classical experiments: rotational spectroscopy of OCS, IR spectroscopy of HCl (g), measurement of water proton spin relaxation times using a home-built pulse NMR, etc. Our lab reports were oral and I quickly learned that I really had to understand the experiment to make a good oral presentation. I spent hours in the library reading about the various spectroscopic experiments we were doing so I could make a 20-minute presentation on the chalkboard to my instructor the week after completing an experiment. When I went to graduate school, I looked for a research project that would be spectroscopy-based. I ended up working for Professor Kenneth Sauer at UC Berkeley and studied the light reactions of photosystem 1 using time resolved cw-EPR. The project involved some equipment modification and building that was outside of Ken's expertise. Fortunately, he had a long-standing collaborative working arrangement with Melvin P. Klein, a Berkeley physicist whose office was next to our lab! Mel and Ken were excellent teachers and a joy to work for.

Matthew: A career in science had always been a dream of mine since I was young. And it was a dream that was further nurtured with the help of excellent science teachers in high school. Chemistry and physics were the two subjects that really fascinated me and heading into college at the University of Michigan – Dearborn, I knew that I wanted to do something related to those, but I had no real long term plan. I decided to take the more practical route and major in chemistry, and by practical, I mean I could imagine jobs following graduation as a chemist, a physicist, not so much. However, I did load up on physics electives. At this point, graduate school or pursuing a PhD was never really in my thoughts. That had all changed when in my sophomore year, Dr. Yiwei Deng, an analytical chemistry professor, asked me if I wanted to do some undergraduate research in her lab and I jumped at the opportunity. I was tasked with method development for the separation of the photodegradation products of benzoic acid using capillary zone electrophoresis. Some of my work ended up contributing to a pair of publications, and I presented it at a several local research symposia. This undergraduate research is what really drove me to think about continuing my education in graduate school. Towards the end of my junior year, looking to expand my horizons, I switched over to work with Dr. Sheila Smith, doing inorganic synthesis. It was through her that I was first introduced to EPR spectroscopy, and later to John McCracken, with whom she had done postdoctoral work, and still collaborated with. It was the mentorship as an undergrad which led me to pursue higher education.

I started as a "zero year" student working in John's lab the summer before grad school. He wanted me convert his Fortran ESEEM programs into Matlab and write a gui, ultimately so that people other than him could use them and actually fit data. I had no formal training in programming nor experience with Matlab and Fortran, but I jumped right into it and learned a lot that summer. It was exciting to see how the quantum mechanics that I had learned about in courses could be visualized, through an EPR spectrum, and directly related to structure or the chemistry of a system. I think it was this intersection between chemistry/biochemistry and physics that attracted me to EPR spectroscopy and why I choose John as my PhD advisor, his charisma didn't hurt either.

What accomplishment in your (short or long) scientific career are you very proud of?

Matthew: I'm really proud of most everything I've done so far in my scientific career. I'd hate to discount anything, but it's those Aha moments which are the most memorable. For instance, at Michigan State, I had collected lots of ESEEM and HYSCORE spectra of tyrosine hydroxylase, we were out to understand the ligand binding at the iron active site. I had probably 10-20 spectra all printed off and spread out on the conference table we had in the office. I would stand over it all sipping a cup of coffee just staring at the spectra, trying to figure out how they all fit together. When the aha moment hit me, I could see how the spectra where changing in field, this correlated to the orientation dependence and I knew where to start simulating. It took me a couple months, I needed to rewrite some of the code, I was simultaneously trying to fit 7 or 8 magnetic field values, but I managed to fit all of the spectra in an iterative fashion by hand. We had a pretty good draft of the paper together around the time that I was defending. One question that came up in my defense however, was in regard to the fits, basically where are the error bars and are those fits statistically rigorous. Unfortunately, since I had done all the fitting by hand, I didn't have any real error bars, I knew the limits because I've played with the simulations enough to know what works and what didn't, but it left a bug in the back of

▶

Matthew D. Krzyaniak studied Chemistry at the University of Michigan-Dearborn (getting also a minor in Physics). He performed undergraduate research with Yiwei Deng. He obtained in 2010 his PhD from Michigan State University under the supervision of John McCracken. He then became a post-doctoral research associate at the University of Alabama in the lab of Michael Bowman. Since 2013 he has been a research associate at Northwestern University.

John L. McCracken studied Chemistry at the University of Illinois (Urbana, USA). In 1983 he obtained his PhD thesis at the UC Berkeley. He then moved to Albert Einstein College of Medicine, working first as a postdoctoral fellow till 1985, after which he became associate director of the NIH Pulsed EPR Research Resource till 1990. He was also a lecturer in Biophysics at Albert Einstein College in Medicine from 1988–1990. In 1990 he moved to Michigan State University, first as associate professor and since 2000 as professor in chemistry. He was from 2002–2010 chair of the Department of Chemistry. He has won different prices, both scientific and teaching awards.

John's head. I left for a postdoctoral position with Mike Bowman in Alabama and the draft on his desk. It took him about a year but he went back, refit the spectra using EasySpin and found an actual statistical minimum, within about 5% the results were the same as I had fit by hand. I was rather proud of that, though the paper did turn out to be a lot better with the more robust method of fitting.

John: There are many accomplishments that I'm proud of. The bulk of the work in my PhD Thesis is centered on the orientation dependence of spin-polarized EPR signals that originate from photosystem 1 electron transport. The idea for these measurements stemmed from work that had been done by Bob Blankenship and Chuck Dismukes in the Sauer lab prior to me starting graduate school. I learned how to orient thylakoid membranes from Mark Crowder in Alan Bearden's Lab at Berkeley and was able to show pronounced changes in the spin polarized EPR lineshapes of P700+ with orientation. While my explanation of these lineshape changes was wrong, Dietmar Stehlik, Gerd Kothe and their students picked up the measurements and were able to do them on single crystals a few years later at K-band. At Ken Sauer and Mel Klein's retirement symposium in 1998, Dietmar showed their single crystal data superimposed on the data Ken and I published from oriented membranes and they were strikingly similar. Structure in our spectra that we thought was noise in 1983, was actually real! I had spent a lot of time getting our measurements to be reproducible and seeing how similar our spectra were to those from crystalline reaction centers was gratifying.

As a post-doc in Jack Peisach's lab, I built a pulse EPR spectrometer based on published designs from Bill Mims, Mike Bowman & Jim Norris, and Larry Kevan. I'll never forget the joy of seeing my first electron spin echo from copper-aquo! One of our first papers dealt with quantifying the number of histidyl imidazole ligands coordinated to Cu(II) in a bacterial phenylalanine hydroxylase using ^{14}N-ESEEM combination frequency line intensities. The idea stemmed from an early work of Mims & Peisach. We took their proposal and developed ESEEM simulation software based on the theoretical works of Mims, Tsvetkov, Dikanov, Kevan and Bowman to show that our spectra were best explained by two coordinated histidine ligands at the active site. This was one of the first quantitative uses of ^{14}N-ESEEM to appear in the literature.

In the early days of my independent career at Michigan State University, Kurt Warncke, then a post-doc in my lab, was able to use ^{14}N- and ^{2}H-ESEEM to show that a radical intermediate that appeared upon anaerobic addition of substrate amine to amine oxidase, was due to a semi-quinone cofactor at the active site that had been modified by the addition of an imine nitrogen derived from the substrate. In collaboration with the late Jerry Babcock, Kurt also developed a ^{2}H-ESEEM measurement that we used to distinguish the redox-active tyrosine residues associated with the Oxygen Evolving Complex in Photosystem 2 based on their phenol side-chain conformational dispersion.

Can you describe one of the most enjoyable moments in your (short or long) scientific career?

John: At one of the Rocky Mountain EPR Conferences that I attended in the mid-1990's, I was presenting some nice ^{2}H-ESEEM work that Kurt Warncke had done to characterize a semiquinone intermediate that we trapped in Amine Oxidase. Mel Klein was in the audience and I happened to glance at him at some point during my presentation. He was grinning from ear-to-ear. Two of his former students, Dave Britt and Ann McDermott, were also at the meeting and both had presented some novel spectroscopic studies on Photosystem 2. I realized at that moment how proud he was of all of us. It was wonderful.

Matthew: This is a hard one, there have been a lot of enjoyable moments at conferences over the years, but since this is an article about John and I, I'll have to go with the first Rocky Mountain EPR conference I had attended. We had been given the afternoon off, this was in Snowmass, Colorado so there was quite a few things around to explore. John and I had decided to grab a burger and beer at the bar in Snowmass before heading out. Just about when we were finishing up, a couple of people that were attending the conference wandered by and asked if they could join us. We got a pitcher of beer or two, John launches off into stories and he keeps us all laughing. That group picks up and has to run, leaving us with half a pitcher of beer, and it would be a crime not to finish it so we stick around. Shortly after, another group comes by, beer, stories and laughter, then they pick up and leave. This repeated probably another time, before a lot of the conference goers who had actually gone off to explore start trickling back and we continue late into the evening. What had started as a burger and a beer ended up being a marathon of drinking and chatting, science, stories and laughing. And I'm proud to say, I did make it to the early morning talks the next day.

Choosing a scientific career is not always an easy road to go. What were the problems that you met (or are still having) in pursuing your scientific career and your EPR dream?

John: I have loved every minute of my scientific career. I have worked for and with the best people. I don't know what I did to get so lucky. I have a rewarding job at Michigan State University and get up every morning eager to go to work. My wife and I had 4 children and she did a great job raising them. I wish I had spent more time with them while they were growing up, but most parents will say that!

Matthew: I love what I'm doing. Right now, I'm a research associate in Mike Wasielewski's lab at Northwestern University, I'm the team leader/manager of the EPR and Spintronics subgroup in his lab. There is a ton of interesting science happening, both in the lab and through various collaborations, which leaves me really busy every day. And I think that's probably my biggest problem, with everything going on it's hard to find the extra time to devote to some of my own independent research interests and projects. Also, I still haven't completely figured out the recipe to balance work and life with my recently married to wife.

What are your passions outside of science and how does this combine with your scientific career?

Matthew: In graduate school I was quite the home brewer, making all sorts of beer, cider and mead. However, when I went to Alabama for the post-doc I didn't bring my brewing equipment with me. This was largely due to the fact that in Alabama, home-brewing wasn't exactly legal at that time. I haven't gotten back into it since moving up to Chicago. But down in Alabama I mostly replaced the brewing with learning to cooking really well. Beyond the cooking and eating, I read a lot, mostly science fiction and I enjoy tinkering with computers and hardware.

John: I like reading history books. I spend a lot of time outdoors in the summer, working in the gardens around my house and riding my bicycle out on country roads near where I live. I like to work on cars and still do most of the maintenance on the vehicles I own. At one point when all of my children were home and a few were in college, I had 5 cars and none of them were from the current century!

Matt, what expectations and plans do you have for your further career? Do you want to continue in academia?

Matthew: I do plan on continuing my career as an EPR spectroscopist and remaining in academia. As of right now I plan on staying at Northwestern for the foreseeable future. Though I do need to find the time to pursue some independent projects.

John, are their specific expectations that you cherished as a young researcher that have come true? Or did your career take turns that you never anticipated at the start, but that have been fulfilling nevertheless? Is there an old scientific dream that you still want realize?

John: As a post-doc in the mid-1980's, I was struck by the ease and reproducibility of the ESEEM experiment. I thought as we worked through problems in chemistry and biology, more scientists would become interested in the method, and the field would grow. Specifically, the method would become accessible to scientists outside of physics and physical chemistry. I still have this goal, but we are far from realizing it.

How do you see the future of EPR and your role in this?

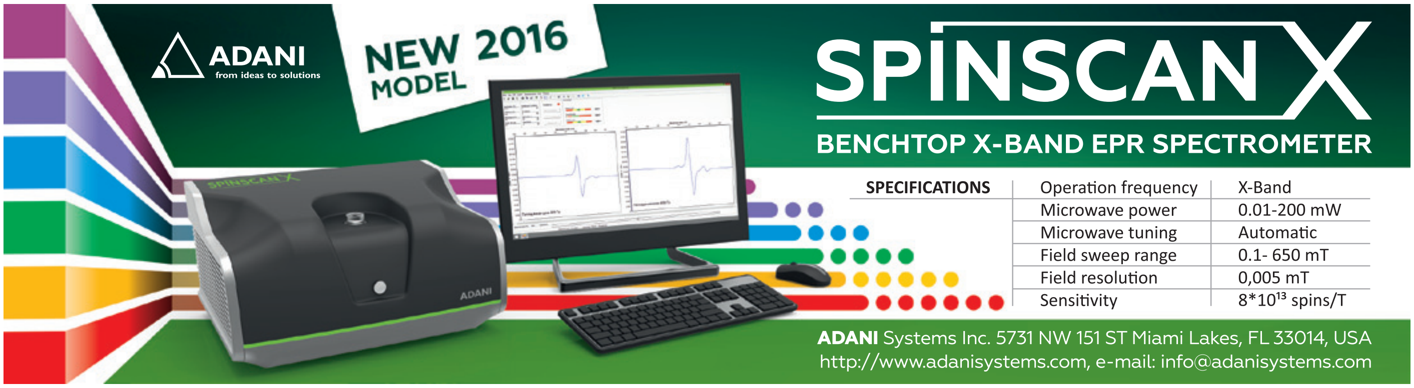
Matthew: I feel we're living in quite an exciting time for EPR spectroscopy. There are a number of lower cost benchtop spectrometers available making EPR spectroscopy more available to a wider audience. Digital electronics have hit the point where it is now cost efficient to have an arbitrary waveform based spectrometer, opening up a huge range of potential experiments and lowering the bar for home built instruments. Software packages such as EasySpin or Deer Analysis make it so that it doesn't take an EPR specialist to simulate or interpret results, again opening the science up to a broader audience. I hope I can remain at the leading edge of EPR spectroscopy, demonstrating the utility of different experiments and developing new methodologies to simplify analysis and understanding. Ultimately working toward the goal of making EPR spectroscopy as turnkey as its cousin NMR spectroscopy.

John: I'm worried about our future. We need to be as supportive as we can of the next generation of EPR spectroscopists because our numbers are decreasing.

Are there matters that you think the EPR community should pay more attention to?

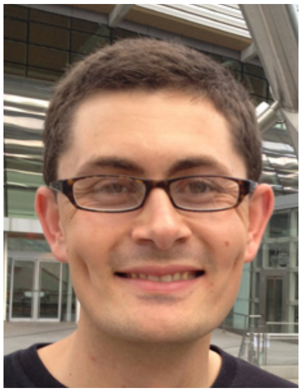
John: I would encourage everyone to try to make their papers as accessible to the scientific community as possible. I try to talk to as many visiting scientists at my institution as possible. For the most part, our visitors are synthetic chemists or analytical chemists and they are finding our literature difficult to read. It takes time to carefully explain data analysis strategies, and/or interpretations to readers who aren't trained in EPR spectroscopy. It is essential that we do this as a community or we will never be able to expand our user base

Matthew: I think the EPR community should always be on the lookout for and accepting of potential collaborations. The best way to reach a broad audience is through demonstration and results. We should never be afraid to just try something out, you might be surprised with the result.



Alexey Silakov became an assistant professor of chemistry at the Pennsylvania State University in 2017. Alexey received his M.S. degree in physics at Kazan State University in 2003, followed by his PhD in physical chemistry from Heinrich-Heine-Universitat Dusseldorf at the Max Planck Institute for Bioinorganic Chemistry in 2007. He completed postdoctoral fellowships at the MPI for Chemical Energy Conversion with Wolfgang Lubitz and at Penn State Department of Chemistry working with Michael T. Green, Carsten Krebs, J. Martin Bollinger Jr. and Squire J. Booker. Alexey received the society's John Weil Young Investigator Award in 2011. His current research focuses on the interplay of biology, chemistry and physics with an emphasis on developing novel EPR spectroscopic methods for the fields of structural biology and (metallo)enzymology.

Joseph Zadrozny became an Assistant Professor of Chemistry at Colorado State University in August 2017. Joe received his undergraduate degree in chemistry in 2007 from Virginia Tech. His PhD was obtained in 2013 from the University of California–Berkeley, wherein he used synthetic inorganic chemistry to design transition metals with lanthanide-like magnetic moments. His postdoctoral work, at Northwestern University with Professor Danna E. Freedman, focused on the design of molecules and materials that may serve as the fundamental units of logic in an electronic spin-based quantum computing system. In 2016, Joe won the Outstanding Researcher Award from the International Institute of Nanotechnology, the SciFinder Future Leader Award, and the European Institute of Molecular Magnetism's Award for PhD Dissertation in Molecular Magnetism. Joe's current research focuses on employing synthetic inorganic chemistry to control the properties of electronic and nuclear magnetic moments. With this ability, his lab's research will take fundamentally new approaches to chemical detection through electronic and nuclear magnetic resonance imaging. Separately, his group will investigate the impact of metal-based magnetic moments in photochemistry toward new spin-based paradigms in reaction discovery.



Michael Lerch became an Assistant Professor of Biophysics at the Medical College of Wisconsin in September 2017. Mike earned his undergraduate degree in Chemistry in 2009 from the University of San Francisco and his PhD degree in Physical Chemistry from the University of California, Los Angeles in 2015. His graduate and postdoctoral work in Wayne Hubbell's laboratory at the Jules Stein Eye Institute focused on developing and applying pressure-resolved electron paramagnetic resonance (EPR) techniques for studying the functional role of protein conformational flexibility, and on providing structural insights into the mechanisms of membrane protein function using double electron-electron resonance (DEER) spectroscopy. Mike won the JEOL Prize for best presentation by a student or first-year postdoctoral fellow at the 49th annual international Royal Society of Chemistry EPR Meeting in 2016. Mike's current research focuses on defining the molecular mechanisms of G-protein-coupled receptor function, particularly how the central process of ligand-mediated activity is modulated by factors such as post-translational modifications, membrane composition, and accessory proteins.

Sebastian Stoian has accepted a position as assistant professor of chemistry at the University of Idaho starting in August 2017. Sebastian received his undergraduate degree in Chemistry from the University of Bucharest (1999) and his PhD in Chemistry from Carnegie Mellon University (2006). He performed his doctoral work under the supervision of Eckard Münck. Using Nuclear Gamma Resonance (NGR or Mössbauer) and EPR spectroscopies, Sebastian studied iron complexes with uncommon oxidation states relevant to small-molecule activation and uncovered an unprecedented mechanism by which some synthetic complexes activate dinitrogen, the most difficult step of nitrogen fixation. In collaboration with Emile Bominaar he also performed extensive computational studies to establish the electronic structure of these iron sites. Sebastian then completed postdoctoral training in Prof. Daniel Nocera's group at MIT, where he investigated several chemical species involved in the catalytic oxidation of water. Most recently, Sebastian was awarded the Jack E. Crow postdoctoral fellowship at the National High Magnetic Field Laboratory in Tallahassee, FL. As a Crow fellow, Sebastian joined the EMR group led by Steve Hill, where he expanded the existing NGR facility. In collaboration with Andrew Ozarowski and others at the MagLab, he utilized high-field EPR to determine the electronic structure and magnetism of metal-containing compounds. As a faculty member, Sebastian seeks to develop a research program at the interface of physical and inorganic chemistry focusing on the design, development, and spectroscopic characterization of earth-abundant metal catalysts. The aim of these studies is to provide spectroscopic benchmarks and mechanistic insights on energy-relevant systems, paving the way to more active inorganic catalysts and, ultimately, to sustainable energy platforms.

JEOL RESONANCE

New

A new design of microwave unit.

Much better sensitivity using 'High Sensitivity Mode'

the next generation in electron spin resonance

JES-X3 Series,

JEOL RESONANCE

Manufacturer : JEOL RESONANCE Inc.
1-2 Musashino 3-chome Akishima Tokyo 196-8558 Japan Sales Division ☎ +82-42-526-5226 sales@j-resonance.com

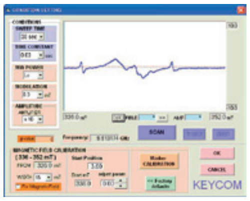
JEOL
Serving Advanced Technology

JEOL Ltd. http://www.jeol.com/
1-2 Musashino 3-Chome Akishima Tokyo 196-8558 Japan
Sales Division Telephone:+81-42-528-3381 Facsimile:+81-42-528-3386

JEOL
ISO 9001 Certificated
ISO 14001 Certificated

KEYCOM
Characteristic Technologies

http//www.keycom.co.jp/
info@keycom.co.jp



Potable ESR instrument : ESR-X10SB

Specification:
Sensitivity: S/N ≥ 10 at 4 mW, 1 μM TEMPOL water solution
Better at 80 mW
Frequency: 9.6 GHz (applicable to customize)
Sweep magnetic field: 15 mT (applicable to customize)
Size: 28(W)×26(D)×35(H), 27 kg

Applications:
KEYCOM desktop electric spin resonance ESR-X10SB is characterized by its simple system with only one box. It is light and compact. Truly portable!
Sweep magnetic field and frequency can be custom designed according to your specific purpose.

Magnetic Test and Measurement Equipment

- Fluxgate Nanoteslameters for measurement of environmental fields with 1 nT (10 μG) resolution.
- Hall effect Teslameters for magnet field measurement and control with resolution to 0.1 μT (1 mG)
- NMR Teslameters with field measurement from as low as 1.4 μT (14 mG) up to 23.4 T.
- Digital Voltage Integrators for flux change measurements.
- Precision Current Transducers and Electromagnet Power Supplies.
- Laboratory Electromagnet & Helmholtz Coil Systems for spectroscopy and imaging.

GMW

955 Industrial Road, San Carlos, CA 94070
Tel: (650) 802-8292 Fax: (650) 802-8298
E-mail: sales@gmw.com Web: www.gmw.com

mtsslSuite – a software package to interpret PELDOR/DEER distance distributions in terms of macromolecular structure

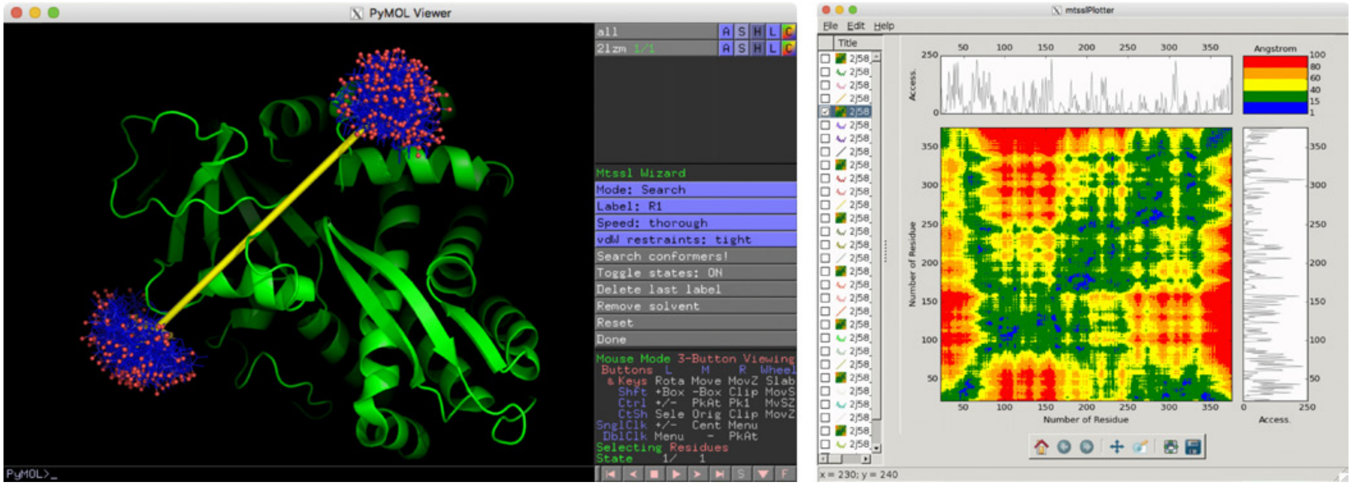
Gregor Hagelueken

Institute for Physical and Theoretical
Chemistry, University of Bonn,
Wegelerstr. 12, 53115 Bonn, Germany

Only a few decades ago, our knowledge about the inner workings of cells was limited by the resolution of optical microscopes and indirect evidence gained from biochemical experiments. This changed dramatically with the elucidation of the DNA structure by Watson & Crick and the first protein structures solved by Kendrew and Perutz, which kindled the fast development of structural biology. Since then, X-ray crystallography, cryo-electron microscopy and NMR spectroscopy have provided fascinating insights into the molecular basics of life. Still, each of these methods has its limitations, for instance the size limits of NMR (structures > ~70 kDa are difficult) and cryo-EM (structures < ~100 kDa are difficult) or the limited ability of crystallography to resolve conformational flexibility. This is why structural biologists often need complementary information from other sources to prove or disprove functional hypotheses about certain aspects of a macromolecular structure. In recent years, pulse EPR spectroscopy and especially the PELDOR/DEER pulse experiment has become a very popular method to investigate questions about conformational flexibility of macromolecules, particularly in the field of membrane proteins.

In 2008, I was a research fellow in the protein crystallography laboratory of Jim Naismith, who was then a professor at the University of St Andrews (UK). We were interested in the structure and function of a particular membrane protein called Wza, an export channel for capsular polysaccharides in *Escherichia coli* bacteria. A closed-state crystal structure of Wza had been determined in Jim's lab [1], and to investigate how Wza opens up to export the polysaccharide, we cooperated with the EPR groups of Olav Schiemann and John Ingledew, who used PELDOR/DEER to determine distances and to study conformational changes in macromolecules. I thus spin labelled the Wza protein with MTSSL, and Olav taught me how a PELDOR experiment is set up and how the data are processed and evaluated. As a crystallographer, I was aware of the problem that the structure of the spin label would have to be carefully considered when interpreting the experimental distance distribution. Olav told me that a brand-new (at the time) MATLAB-based software package called "MMM" by Yevhen Polyhach and Gunnar Jeschke could be used to estimate the conformation of the spin label on the protein surface [2]. However, it is safe to say that MATLAB licenses are a rarity in crystallography labs and instead, I wrote a Python script that superimposes a model of the MTSSL spin label onto the Wza structure, quickly generates an ensemble of conformers that do not clash with the protein and then measures the distribution of distances between the spin centers of two such ensembles. We were pleased, how well this rather simple procedure reproduced the experimental distance distributions and thereby helped us to better understand the Wza protein. Since the Python script appeared quite useful, I decided to convert it into a "wizard" i.e. a custom function of the PyMOL molecular graphics software (www.pymol.org), which is very widely used in the structural biology community. I called the program (not very imaginatively) **mtsslWizard** (Figure 1, left). As a newbie in the EPR world at the time, I had unknowingly created an implementation of the accessible-volume approach pioneered by Ken Sale, Peter Fajer and colleagues [3]. Olav and I had many discussions, whether or not the mtsslWizard should consider preferred rotamers of MTSSL. Eventually,

▶



Mtssl Wizard
Mode: Search
Label: R1
Speed: thorough
vdW restraints: tight

Figure 1: Left: The mtsslWizard is a PyMOL plugin that allows to attach models of spin labels such as MTSSL (blue lines) to any macromolecular structure (green ribbon). The program can measure distances between the ensembles (yellow). Right: The mtsslSuite contains a number of other programs, for example mtsslPlotter, which can be used to visualize calculations of the mtsslWizard. In this case a distance map is shown, which allows to quickly identify optimal labelling sites.

we decided against it, because doing so did (in our hands) not improve the prediction results and it currently seems impossible (at least on feasible time scales) to accurately predict the conformation of the spin label at a particular labelling site. Since its release in 2011, testing by various groups has shown that the mtsslWizard predicts inter-spin distance distributions equally well as e.g. MMM, PRONOX or ROSETTA-EPR [4, 5]. Nowadays, I routinely use PELDOR/DEER in my own research at the Institute for Physical & Theoretical Chemistry of the University of Bonn, which prompted the development of several new features for the mtsslWizard. One example is the calculation of difference distance maps. These are very helpful to find optimal spin labelling positions, if a conformational change is to be analyzed by PELDOR/DEER. Also, quite a few users have contacted me with various feature requests, most often regarding user-definable spin labels.

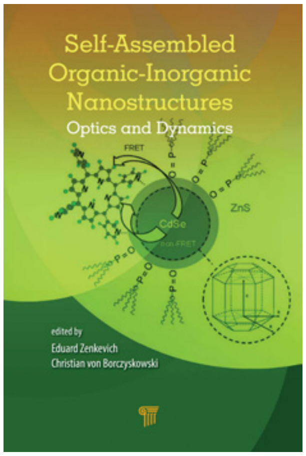
More recently, new functionalities were added, which could not be accommodated in the deliberately simple mtsslWizard graphical user interface (GUI) and were thus designed as separate programs with dedicated GUIs: The **mtsslPlotter** plots distance distributions generated by mtsslWizard and experimental distance distributions from DeerAnalysis [6]. The program can also draw the aforementioned distance matrices and difference distance matrices, together with an accessibility estimation for each possible labelling site (Figure 1, right). The idea for the **mtsslTrilaterate** program came from Olav Schiemann and was developed by Dinar Abdullin. It is helpful in cases were a spin center such as an elusive metal ion needs to be localized inside a macromolecule [7]. The **mtsslDock** software determines the geometry of macromolecular complexes or multidomain proteins based on distance constraints between the complex partners [8]. All programs of the **mtsslSuite**, including detailed manuals, are freely available at www-hagelueken.thch.uni-bonn.de.

The mtsslSuite is a cross-platform application and is (as of now) tightly integrated into the PyMOL molecular graphics system. While this has obvious advantages, it also means that the installation and maintenance of the software package can be challenging (depending on the hacking skills of a particular user). To improve this situation, an mtsslSuite web server is currently under construction, which will always run the newest version and features of the mentioned software packages and circumvent the installation problems. Finally, I would like to thank all users of the mtsslSuite for giving the programs a try and for their constructive feedback.

---

1. C. Dong, K. Beis, J. Nesper, A.L. Brunkan-Lamontagne, B.R. Clarke, C. Whitfield, et al.: Wza the translocon for *E. coli* capsular polysaccharides defines a new class of membrane protein. Nature 444 (2006) 226–229.
2. G. Jeschke: MMM: A toolbox for integrative structure modeling. Protein Sci. 181 (2017) 223.
3. K. Sale, L. Song, Y.-S. Liu, E. Perozo, P.G. Fajer: Explicit treatment of spin labels in modeling of distance constraints from dipolar EPR and DEER. J. Am. Chem. Soc. 127 (2005) 9334–9335.
4. G. Jeschke: Conformational dynamics and distribution of nitroxide spin labels. Prog. Nucl. Magn. Reson. Spectrosc. 72 (2013) 42–60.
5. N.S. Alexander, R.A. Stein, H.A. Koteiche, K.W. Kaufmann, H.S. Mchaourab, J. Meiler: Rosetta-EPR: Rotamer Library for Spin Label Structure and Dynamics. PLoS One 8 (2013) e72851.
6. G. Jeschke, V. Chechik, P. Ionita, A. Godt: DeerAnalysis2006 – a comprehensive software package for analyzing pulsed ELDOR data. Appl. Magn. Reson. 30 (2006) 473–498.
7. D. Abdullin, N. Florin, G. Hagelueken, O. Schiemann: EPR-based approach for the localization of paramagnetic metal ions in biomolecules. Angew. Chem. Int. Edit. 54 (2015) 1827–1831.
8. G. Hagelueken, D. Abdullin, R. Ward, O. Schiemann: mtsslSuite: In silico spin labelling, trilateration and distance-constrained rigid body docking in PyMOL. Molecular Physics 111 (2013) 2757–2766.

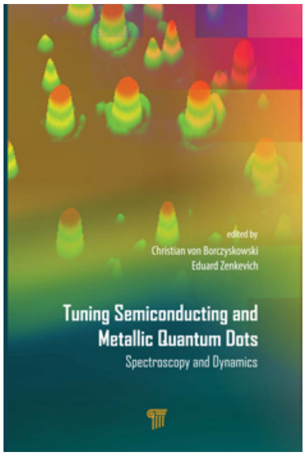
new Books & Journals



Self-Assembled Organic-Inorganic Nanostructures – Optics and Dynamics

Edited by: Eduard Zenkevich (National Technical University of Belarus, Minsk) and Christian von Borczyskowski (Chemnitz University of Technology, Germany)

Hardback 394 pages
Print ISBN: 9789814745819
eBook ISBN: 9789814745833
DOI: 10.4032/978981474583



Tuning Semiconducting and Metallic Quantum Dots – Spectroscopy and Dynamics

Edited by: Christian von Borczyskowski (Chemnitz University of Technology, Germany) and Eduard Zenkevich (National Technical University of Belarus, Minsk)

Hardback 387 pages
Print ISBN: 9789814745246
eBook ISBN: 9789814745253
DOI: 10.4032/9789814745253

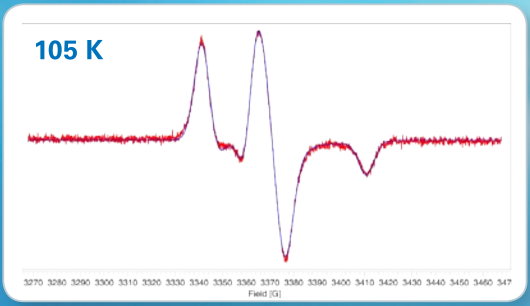
Key Features

- Introductory chapters discuss optical features and detection of single quantum dots, dynamics of quantum dot-dye nanoaggregates via self-assembly, quantum dot applications, and lithographic preparation of optical nanostructures
- The chapters are a combination of scholarly presentation and comprehensive review
- Case studies from the authors' research, including unpublished results, broaden the understanding
- The combination of introductory parts and actual research topics makes the book attractive both for undergraduates and for researchers in the field of optical nanoscience, materials science, and nanotechnology

The Correct Result

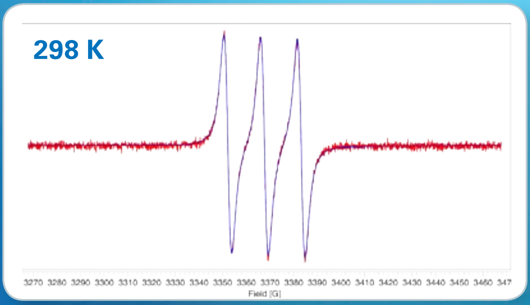
Regardless of Temperature

Anisotropic SpinFit for solids

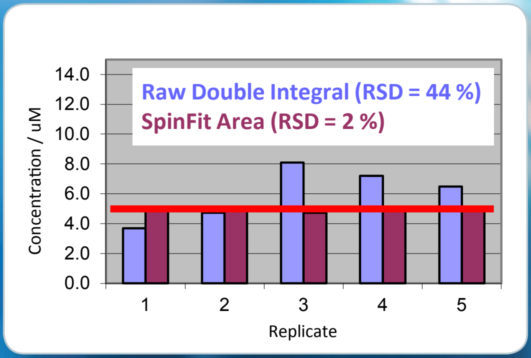


Tempo / Toluene @ 105 K
SpinCount = 1.36 mM

Isotropic SpinFit for liquids



Tempo / Toluene @ 298 K
SpinCount = 1.34 mM



Improved results through spectral fitting

Spectrum fitting improves the determination of the spin concentration in a sample. With the SpinFit package for simulation and fitting, both liquid and solid spectra can be rapidly fitted and the spin concentration can be precisely determined with SpinCount.

SpinFit

- Powder fitting (Anisotropic)
- Liquid fitting (Isotropic)
- Multiple species
- Multiple hyperfines

SpinCount

- Reference free
- High precision & accuracy
- Sample temperature taken into account
- All parameters accounted for

Contact us for more details: **www.bruker.com/epr**


EMX nano
BRUKER

EPR and its pharmaceutical applications

Kalina Ranguelova and Ralph Weber
Bruker Biospin Corp., Billerica, USA

Electron Paramagnetic Resonance (EPR) spectroscopy has the unique capability to detect and characterize free radicals and transition metals and it has been successfully applied to measure both in vitro and in vivo free radical intermediates from drugs and oxidative processes and to study the free radical/antioxidant properties of pharmaceuticals. The aim of this article is to provide a short overview on some applications of EPR in the pharmaceutical field and to demonstrate how this technique can be useful in several steps of drug development and delivery, as well as in quality control studies.

I. Detecting and evaluating degradation in pharmaceuticals

Heat, light, oxygen, moisture, sterilization processes, impurities, and excipient interactions are some of the factors that can compromise drug product stability. Moreover, all these factors may cause degradation of the active pharmaceutical ingredients (APIs), inert materials (excipients), or the final medicinal products (formulations) resulting in loss of product potency or toxic by-product generation. Degradation processes quite often involve free radicals and transition metals that are responsible for most of the damage that occurs in drug products. The International Conference of Harmonization (ICH) guidelines state that the intrinsic photostability characteristics of all new APIs and products should be evaluated to demonstrate that light exposure does not result in unacceptable change. These guidelines have been implemented in Europe, the United States, and Japan for over 20 years. EPR spectroscopy can be used to determine the root cause of degradation, measure the extent of degradation, and predict long-term stability characteristics of the APIs, excipients and formulations (Figure 1).

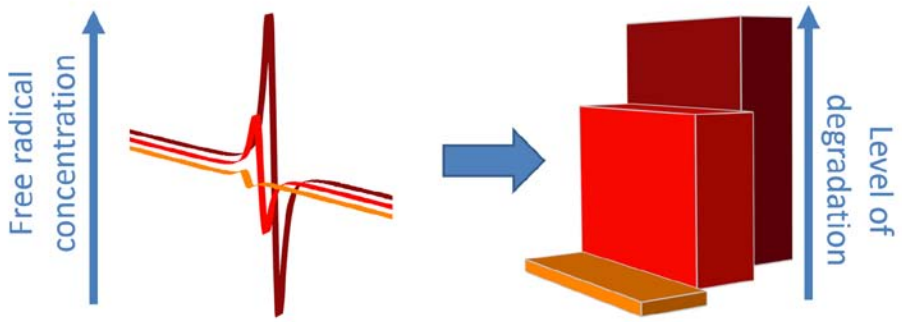


Figure 1. Degradation correlates with the EPR signal.

II. Optimizing stability and shelf-life

Forced degradation (stress testing) is routinely used in pharmaceutical development to predict the stability of drug products that affects purity, effectiveness, and safety. In stress testing the drug product is exposed to heat, light or chemical agents with the goals of understanding degradation pathways, determining the intrinsic stability and shelf-life, developing stable formulations, and evaluating antioxidant efficiency. Forced degradation often involves a free radical pathway therefore identifying the radical intermediates is extremely important. In addition, antioxidants' efficiency in drug formulations is characterized by the ability to scavenge the free radicals and eliminate stability issues.

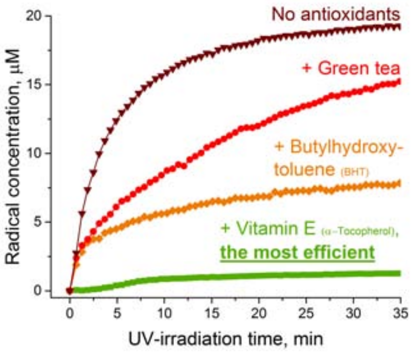


Figure 2. Evaluation of antioxidants' effect on a skin care product during UV-irradiation shows vitamin E to be the most efficient antioxidant. The data were collected with a Bruker EMXnano EPR spectrometer equipped with a light guide for in situ photochemical reactions. The concentration was determined with the SpinCount module.

EPR can successfully predict long-term stability of drug products by detecting and monitoring short-lived free radicals produced during stress testing via chemical, thermal, or photochemical reactions. EPR can also determine radical scavenging effectiveness and efficiency of antioxidants (Figure 2).

III. Reaction monitoring

Reaction monitoring is critical for process understanding, optimization and scaling up, leading to cost savings and ensuring the quality of the final product. It often provides insights into the chemical reaction mechanisms. Kinetic information can be extracted from EPR time course data to build kinetic models that will be used to predict conditions, enabling effective process optimization, risk assessment and control. The quantitative and non-intrusive nature of EPR makes the technique extremely powerful for the identification and characterization of radical reaction intermediates, providing insights into the reaction mechanisms and kinetics of chemical reactions. Free radical chemistry may offer many advantages over traditional synthetic approaches such as greener chemistry and higher yields. Transition metal-containing catalysts are also widely used in pharmaceutical synthetic routes. EPR can be used to identify reaction intermediates to obtain mechanistic information and to answer key chemical questions about reaction yield and reaction kinetics (Figure 3).

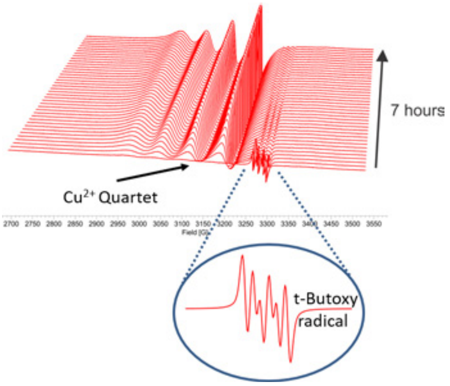


Figure 3. EPR monitoring of Cu(I) catalyst's oxidation to Cu(II) and formation of t-Butoxy radical during indole (a key intermediate for a new Hepatitis C drug) synthesis. Data courtesy of Merck. The EPR experiments were performed by a 2D Field vs Time scan on a Bruker EMXplus spectrometer equipped with an ER4119HS high-sensitivity resonator.

IV. Sterilization processes

APIs, excipients, final drug formulations, laboratory equipment, and medical devices, may need to go through a sterilization process. The most commonly used sterilization methods are: gamma or electron beam irradiation, dry heat, and pressured vapor sterilization. However, all these sterilization processes can generate free radicals that are responsible for the degradation of the sterilized materials and cause alteration of their physicochemical properties. Free radicals decrease drug potency by partial decomposition during sterilization and finally they may be a toxicological hazard. EPR complements the many methods used to analyze sterilization effects. Specifically, it investigates the role of free radicals in the degradation of pharmaceutical products. The ability of the technique to characterize free radicals and identify their source in post-sterilized drug products makes it suitable not only for R&D laboratories but also provides easy 'go/no go' decisions based on radical quantification for quality control and assurance (Figure 4).

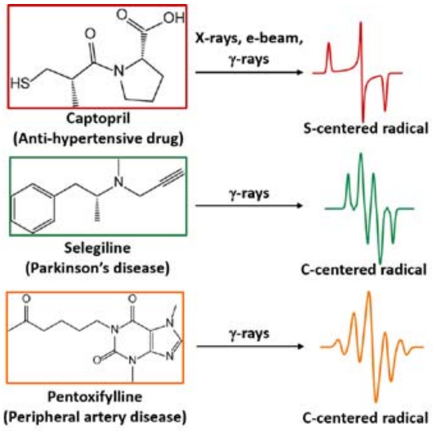


Figure 4. Sulphur- and carbon-centered radicals detected in γ-irradiated drugs.

V. Impurity profiling

All drugs contain impurities that can arise from the drug formulation's APIs or excipients. They are also introduced into the drug product during the formulation processes, packaging, and storage. Impurities have many unwanted effects, such as decreasing the therapeutic effect, lowering the product shelf-life, and inducing toxicity. Identification, quantification, and control of impurities in the API and the drug product are critical in drug development. Organic impurities are often free radicals from by-products, intermediates, or degradation products. Inorganic impurities include transition metals, reagents, and ligands. Detecting transition metals is important due to their potential to catalyze the degradation of APIs or drug formulations. With EPR one can identify and quantify paramagnetic impurities down to parts per billion levels (Figure 5).

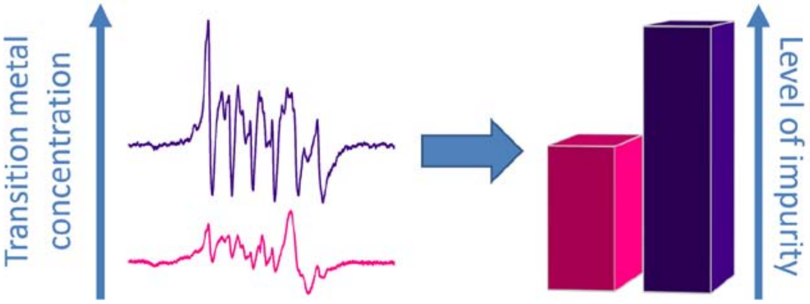


Figure 5. Mn(II) present at trace levels in the excipient calcium hydrogen phosphate, that is commonly used as a filler in tablets, can be easily detected and quantified by EPR. The EPR spectra were recorded on a Bruker EMXnano with a sweep width of 2000 G.

VI. Analysis of reactive oxygen and nitrogen species (ROS and RNS)

Development of new biological drugs (biologics) in the pharmaceutical industry involves studying of reactive oxygen and nitrogen species (ROS and RNS) due to their potential for oxidative stress and damage in cells. The major reason to measure these species in biological systems is to determine whether they play a role in physiological or pathophysiological processes. Detection and characterization of these highly reactive free radicals in biological materials is done using EPR. Most of the biologically relevant radicals are very short lived and, therefore, impossible to detect in biological samples. For this reason, compounds (spin traps and spin probes) have been used that form stable adducts with radicals. With EPR a variety of ROS and RNS can be identified and time evolution data provide concentration of each reactive oxygen or nitrogen species (Figure 6).

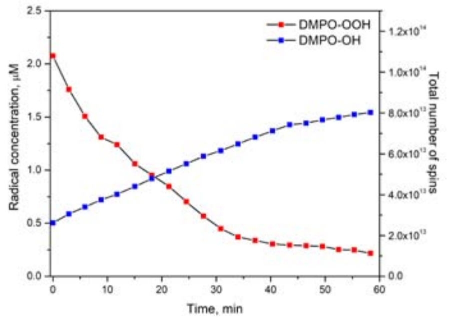


Figure 6. Detecting of superoxide and hydroxyl radical using DMPO as spin trap in xanthine/xanthine oxidase system. The EPR spectra were collected on a Bruker EMXplus spectrometer and SpinFit/SpinCount moduels were used for quantitative analysis.

* * *

EPR is a powerful tool within pharmaceutical analysis and has wide range of applications. It is a non-destructive and very sensitive technique that requires a small sample size and it can be applied to solids, liquids, suspensions, solutions, whole tablets, etc. It can be performed over a wide temperature range to gain further information about any radical species or transition metal present in the drug system. Samples can be photolyzed or thermally force oxidized in situ in the spectrometer. Because of EPR's inherent high sensitivity the EPR measurements are very quick and only use a small quantity of the API or drug formulation, so the technique could be used during the early drug development phase. It should be noted, that only a small number of examples were discussed. There are many more presented in the literature.

---

1. Williams H. et. al.: The power of electron paramagnetic resonance spectroscopy in pharmaceutical analysis. *Spectroscopy Europe* (**2006**) 18(1)
2. Williams H. et. al.: Predicting the photostability characteristics of active pharmaceutical ingredients using electron paramagnetic resonance spectroscopy. *Drug Dev. Ind. Pharm.* (**2012**) 38(2) 200
3. Mangion I. et. al.: Using electron paramagnetic resonance spectroscopy to facilitate problem solving in pharmaceutical research and development. *J. Org. Chem.* (**2016**) 81 6937
4. Peng F. et. al.: A mild Cu(I)-catalyzed oxidative aromatization of indolines to indoles. *J. Org. Chem.* (**2016**) 81 10009
5. Engalytcheff A. et al.: Attempts at correlation of the radiolytic species of irradiated solid-state captopril studied by multi-frequency EPR and HPLC. *Radiat. Res.* (**2004**) 162 616
6. Köseoğlu R. et al.: Electron paramagnetic resonance of some γ-irradiated drugs. *Appl. Radiat. Isot.* (**2003**) 58 63
7. Lam X.E. et al.: Site-specific tryptophan oxidation induced by autocatalytic reaction of polysorbate 20 in protein formulation. *Pharm. Res.* (**2011**) 28 2543

Charles P. Poole Jr. (1927–2015)

Charley Poole passed away on November 1, 2015 at the age of 88 years. Most people know him mainly because of his book, which may be considered a bible in experimental ESR, entitled "Electron Spin Resonance: A Comprehensive Treatise on Experimental Techniques" (Wiley Interscience, 1967), apart from his other distinguished achievements as a researcher. He obtained his B.S. in Pre-Medicine, M.S. in Physics in 1952 from Fordham University (Bronx, NY), and Ph.D. in Solid State Physics from the University of Maryland in 1958. His career started with professional experience as a physicist at Westinghouse (1952-53), designing microwave components for radar and subsequently his involvement with NMR at the Gulf Oil Research Center in Pittsburgh, PA (1958-64). Thereafter he moved into academia as a professor at University of South Carolina in 1964. During his lifetime, Dr. Poole published over 150 research articles and numerous books on a wide variety of condensed matter physics topics, including superconductivity and Clifford Algebras. He was an extremely diligent researcher, gifted with an enormous memory. His other scholarly achievements include, among others, a Fellow of APS, Editor of Magnetic Resonance Review, a member of Editorial Board of Bulletin of Magnetic Resonance, a recipient of 1971 Russel Award of the University of South Carolina for Creative Research in Science and Engineering and 1980 Jesse W. Beams Medal of APS, Southeastern Section. He served as Treasurer of ISMAR for several years beginning 1984. He was named a Fellow of International EPR Society in 1999.

I came to know of Charley's famous book when I started my research in ESR in mid-1970's and needed to learn the experimental techniques. It was a very thorough and a complete treatise on the subject at that time. Then I personally met him at international conferences. He was a down-to-earth modest and humble person, and very pleasant to be with. He seemed not only very knowledgeable on the subject of EPR, but on lots of other things, e.g. the Russian language having translated some scientific books from Russian to English, classical mechanics, wherein he participated in the revised version of the famous Goldstein's book on classical mechanics.

I invited him a couple of times to Concordia University to give seminars, and jogged with him as that was our favorite activity. Wherever he visited, he continued on with his jogging, and attending Sunday Catholic masses if possible, as he was an ordained deacon. I have had the privilege of jogging with him on Flamingo Beach in Rio de Janeiro when we met at the ISMAR conference in 1986, in 1989 we jogged together in Poznan (Poland) during the Congress Ampere conference. Then in 1995, I jogged with him in Kazan, Russia, where we attended the 50th anniversary of discovery of EPR by Zavoisky, organized by Kev Salikhov.

When his 70th birthday was celebrated at University of South Carolina, I was honored to be invited to attend it and to give a lecture. There were some important guests invited to that event, e.g. Slichter and Redfield.

He invited me to write review articles on transition metal ions and on rigorous evaluation of spin-Hamiltonian parameters using least-squares fitting in ESR for Magnetic Resonance Review, of which he was the editor. He also served as external examiner to two of my Ph.D. students in early 1990s, which resulted in a couple of more of his visits to Montreal.

He was really my mentor in ESR. I was extremely honored when he invited me to write chapters on transition-metal ions ESR in his ESR Handbook, volume 2. With the advent of high-frequency EPR, he wanted to write a book on multi-frequency EPR with me as co-editor. Unfortunately, when his wife passed away, he was too depressed and withdrew himself from the task. But he wanted me to carry on with the book as the solo editor, assuring me of his continued mentorship on the project. This he did, and I finished the book with his continued advice, appearing in print in 2011 entitled "Multifrequency Electron Paramagnetic Resonance: Theory and Applications". He also encouraged me to edit a follow-up book "Multifrequency Electron Paramagnetic resonance: Data and Techniques", which appeared in 2014.

He wrote several books on superconductivity ("Handbook of Superconductivity", "Superconductivity", "Copper Oxide Superconductors", "The New Superconductors"), as well as on nano-materials ("Introduction to Nanotechnology" and "Physics and Chemistry of Nanosolids"). In addition, he wrote "Handbook of Electron Spin Resonance (volumes 1 and 2)", "The Theory of Magnetic Resonance", "Relaxation in Magnetic Resonance", "Magnetic Resonance of Phase Transitions", "The Physics Handbook" and "Physics Qualifying Examination", among others.

Over the remaining years I continued this close relationship with him and kept in touch by telephone. After he suffered a stroke in 2014 he moved to an assisted living center, but still continued to write. A few months before he passed away my telephone messages were not returned; I did not know any of his next-of-kins to get news about him. Finally, I called the Department of Physics, University of South Carolina to get the sad news that he passed away at the end of 2015. The EPR community has lost a great researcher.

Sushil Misra


From left to right: Charles Poole, Sushil Misra and Horacio A. Farach.


From left to right: Charlie Slichter, Sushil Misra and Charles Poole.

* * *

My first contact with Charlie came after I had written a review of his pioneering book concerning the design and instrumentation of EPR spectrometers in 1967. This book was the result of an enormous amount of work to locate and summarize every published paper that had EPR instrumentation details that were innovative. My review was very positive but did include a few criticisms. It was not long after this review was published that Charlie telephoned me and asked if I would be interested in writing annual reviews that summarized all new spin Hamiltonian data for paramagnetic ions. After reading the summary that had already been published in Magnetic Resonance Reviews for the years up to 1970, I agreed to do the summary for 1971. Charlie liked what I had done and asked me to continue. The consequence was a sequence of reviews for 1971–1979. Charlie managed to encourage the summarization of an enormous amount of EPR data and information while also writing some fine books on the subject with several collaborators. His vision and his willingness to work extremely hard provided an enormous service to the EPR community for which we should all be grateful.

Harvey A. Buckmaster

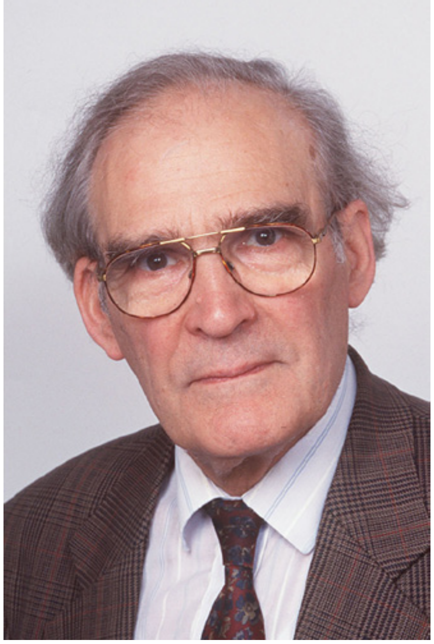
* * *

While I haven't had the opportunities to know Charlie personally over the years, I have certainly interacted with him many times at EPR meetings and occasional grant review panels. It's not really correct to say that I haven't known him personally as he was one of the most personable, humble, modest scientists that I have known who makes everyone feel comfortable and was always candid about his opinions. It's fair to say that his contributions and importance as a teacher in the EPR field equates with the three or four great leaders in the field.

Perhaps his most timeless contribution was the book *Electron Spin Resonance: A Comprehensive Treatise on Experimental Techniques* (1967, Wiley Interscience Publishers), which is literally the 'bible' of EPR in experimental techniques. It was meticulous, totally complete, and very readable and understandable (perhaps an accurate definition of a bible?). One could go to this book consistently for information and details about the experimental techniques in EPR and associated applications. He diligently revised/updated this book in the 1980s. (I once heard a scientist present some incremental work at an EPR session and gloat over the fact that he found an error in an exponent in one equation (of thousands) in Charlie's book. The audience was not impressed, knowing full well that this alleged error was either trivial or a typographical oversight.) He also edited two volumes of ESR Handbook, covering current topics in ESR in 1990s. He also wanted to write a book on multifrequency EPR, covering the latest developments, in collaboration with Sushil Misra, which he was unable to continue due to poor health, resulting in Sushil (his lifelong colleague and student) taking on the editorial responsibility, following the topics that Charlie wanted to include, and writing many chapters himself.

But the real joy that I'll always cherish was sitting with Charlie and Sushil over a drink or a meal and discussing the history of our field, the science and life in general. We have truly lost a giant and a delightful human being who I'll never forget.

Larry Berliner



John Michael Baker (1930–2017)

Professor John Michael Baker, known as Michael, who died on August 10th aged 86, was an inspirational and patient teacher and a modest but world recognised researcher, who spent a lifetime in Oxford nurturing students, solving research problems and helping colleagues.

Michael, was born on December 21 1930 in Shropshire, into a family who were dedicated to education. Michael's father, a schoolmaster died when he was only 14, leaving his mother, with the challenge of bringing up him and his seven year old brother. In the summer of 1945 Michael, who was educated at Wellington Grammar School, got distinctions in his mathematics, chemistry, and physics School Certificate exams and his mother, encouraged him to continue into the Sixth Form (rather than working to support the family) with a view to going on to Cambridge. Michael continued to excel but was awarded a State Scholarship tenable at Oxford University, rather than the "other place", and was advised by a former Wellington pupil already studying in Oxford to apply to St. John's. Here Michael met a brilliant new Physics tutor, Brebis Bleaney, who had perhaps the greatest influence on his life. It is clear that Michael enjoyed the undergraduate life, he rowed (St. John's Captain of Boats 1950-51), played tennis and squash, and acted. He of course excelled in Physics and was awarded first class in Physics Finals, qualifying him for a funded studentship for three years to do research on paramagnetic resonance in Bleaney's research group.

During the second world war Bleaney had been involved in the development of radar (microwave) technology in the Clarendon Laboratory, and as is well documented in 1945 had pioneered the development of electron paramagnetic resonance (EPR), exploiting the new microwave technologies for fundamental research. Unknown to Bleaney, an experiment had, in fact, been carried out in 1944 by E. K. Zavoisky at the University of Kazan in the Soviet Union. Much later the University of Kazan recognised Bleaney's contribution with an honorary professorship and a medal. As we know the field of EPR spread rapidly worldwide and remains an important technique not only in physics but also in chemistry, life-sciences and quantum technology. Michael's doctoral research concentrated on EPR of hydrated salts of rare earths, then a relatively unknown group of the periodic table. Michael recalled Bleaney sitting on his desk, writing their first joint paper, as he was still measuring the data, which showed that Vanadium 50 has a nuclear spin of 6, the largest nuclear spin then discovered. During his graduate research (DPhil awarded 1954), Michael moved from St. John's College to St. Antony's College, Oxford where he was awarded a Senior Scholarship (1953-55). Michael survived a laboratory accident in which the glass Dewar vessel around the microwave cavity and filled with liquid hydrogen exploded embedding fragments of glass into his thigh. Michael described this as the era before the invention of Health and Safety. Fortunately, Bleaney was in the lab and took his student to the Accident and Emergency Department of the Radcliffe Infirmary. The operation to remove the glass fragments was interrupted half way through because the starting surgeon had to go and play rugby, with a substitute taking over the operation; this was most definitely Oxford. During the same Hilary term in 1953 Michael met Gay (Gabrielle) Prefect, who had come the Clarendon Laboratory as secretary/librarian. They were married in July 1954.

Michael, with Gay, visited Harvard University as a Fulbright Scholar, 1955-56, where he worked with the Nobel Prize winner (for molecular beam spectroscopy) Norman Ramsey. On return from Harvard in 1956, Michael gained an ICI fellowship at the Clarendon Laboratory, loosely attached to St. John's. At this stage he may have been considering pursuing a career in the USA, but instead became a University lecturer in the Physics Department and the first Tutorial Fellow in Physics at Merton College on 1st January, 1957. Along with the Clarendon Laboratory, Merton remained the centre of his professional life for the rest of his career. Initially he had rooms in St. Albans Quad, but for most of his career he had a room on the second floor of Staircase 4 in Fellows' Quad, overlooking both the quadrangle and Merton Field. Officially it was J. R. R. Tolkein's former room, though Tolkein had informally swapped rooms with Professor of Political Economy John Jewkes, next door.

When Michael arrived, Merton had only one physics undergraduate. Michael quickly built up numbers, initially with six students per year, later increasing to about eight a year. As a tutor, Michael aimed to emphasise the excitement of physics, to inculcate physical understanding as well as mathematical rigour, and to point out the links between the different parts of the subject. He was very proud of his pupils' success, and was remembered in particular by his students for always being prepared to make time to discuss a problem. He enjoyed following his pupils' achievement in their subsequent careers, and catching up with them at reunions. In turn, many of his pupils maintained either professional or personal contact with him after their time at Merton. He became an Emeritus Fellow of Merton on his retirement in 1998, and continued to participate fully in college life.

Michael often recalled his "big-miss" in 1954/55, when he was slow to take up Bleaney's suggestion, that they should try exciting both EPR and nuclear magnetic resonance in the same sample. Because of the pressure of other work, Michael delayed following up Bleaney's insight until after his visit to Harvard (1955/56), during which time George Feher of the Bell Laboratories got to what he called Electron Nuclear Double Resonance (ENDOR) first. From 1957, Michael now leading his own research group was however, with his graduate students Tito Williams and John Hurrell, the pioneer in the UK of ENDOR. Indeed another one of Michael's students E. R. Davies, invented "Davies ENDOR" while working in his group. Michael continued to specialise his research in applying EPR and ENDOR to a range of problems, but at the suggestion of Bleaney's successor as Dr. Lee's Professor (1978-88) Sir William (Bill) Mitchell, Michael with his graduate students began an extensive programme of research to use these techniques to study the characteristics and properties of "point defects" in natural and lab-grown diamonds. These studies allowed the identification of defects and impurities which can for instance alter the colour of diamond (greatly changing the value of gems) and today the properties of such point defects are being exploited in nanoscale magnetic sensors and even in prototype quantum computers. Thirty years on this research programme continues, and in no small part led to the foundation of the UK's multi-university EPSRC and industry funded Centre for Doctoral Training in Diamond Science and Technology. Michael an internationally renowned researcher who travelled the world with his work did not shirk his teaching and administrative duties in Oxford, he lectured, demonstrated in undergraduate laboratories and examined. He was Chairman of the Sub-Faculty of Physics and Member of the Physical Sciences Board, 1972-75, a Member of the Departmental Committee of the Clarendon, 1978-80; and Head of Condensed Matter Physics at the Clarendon, 1993-97.

Michael was elected a Fellowship of the International EPR/ESR Society in 2011; he was delighted by this accolade. Michael was author or co-author of some 150 refereed publications, many of which were foundational in the early days of EPR and results of which found their way into "Electron Paramagnetic Resonance of Transition Ions" by Abragam and Bleaney. Despite ill health in recent years he continued to be actively involved in research on defects in diamond up to his death. In his will Michael endowed lectures at the Clarendon Laboratory to be entitled "the Brebis Bleaney Memorial Lecture".

Michael is survived by his three children, Christopher, Timothy, and Claire, and hundreds of students (including over 30 Ph.D. students) who hold him in great affection and are eternally grateful for his patience in teaching them the things they never thought they would understand. I will always remember Michael's great determination, seen both in the laboratory and cycling up the Woodstock Road into driving rain into his 80's, but mostly his razor sharp mind and his ability to always make time to help others.

Mark Newton
University of Warwick,
Department of Physics

* * *

I have previously explained (*EPR newsletter*, 13/1-2, pp.16, 17) that the Baker group had to pass through our lab [041] to reach their lab [042]. Michael and his group were of course always at the 041 Coffee Club that took place in our lab. Over the three years at Oxford, I got to know Michael pretty well.

When I arrived in Oxford in September 1961, Michael's student, FIB [Tito] Williams was finishing his thesis which reported the first ENDOR done in England, on Divalent Eu^{2+} ions in Calcuim Fluoride [see also Baker and Williams: PRS **267A** 283 (1962)].

In August 1969 Michael became the fifth sabbatical visitor from the Clarendon Laboratory to spend an extended period at the still relatively new Monash University in Melbourne's Southeast. He was preceded by Martin [later Sir Martin] Wood, Founder of Oxford Instruments. Monash University, established in 1958, enrolled its first graduate students one week before its first undergraduates in 1961! Michael arrived with his family just in time to attend the International Symposium on Electron and Nuclear Magnetic Resonance held at Monash [11–15 August, 1969]. He remained with us for about eight months during which time he presented a series of graduate lectures on pair-interactions of paramagnetic rare earth ions in crystals. This was of particular interest to me and my group as we'd been involved for some time studying transition metal ion dimers in chemical complexes with a group in our Chemistry Department. Michael's lectures, presented in the precise and careful manner in which he pursued his science, were written up and later published in Reports in Progress in Physics [**34**, 109 (1971)].

While I had hoped to catch up with Michael during a short visit to Oxford in 1971, this did not happen as he was away. However in 1986, firstly at the AMPERE Congress in Rome and shortly afterwards in Oxford, we managed a good deal of time to talk and catch up. Then in 1994 we caught up again at London Airport on our way to Frankfurt to connect with the Aeroflot Charter Flight to the AMPERE Congress in Kazan and enjoyed a good many long conversations during the following week. In June 1995, I attended the Brebis Bleaney 80th Birthday Conference in Oxford, excellently organised and managed by Michael.

As recently as January of this year, Michael and I engaged in e-mail correspondence concerning our recollections of Erwin Hahn's 1961-62 sabbatical in the Clarendon Lab at Oxford, particularly as he often came to the 041 Coffee Club referred to earlier and which I have written about in two or three previous *EPR Newsletters*. Michael gave no hint of health problems at that time so it came as a big shock to learn in August from his son, Timothy, that he had passed away on August 10th.

Michael belonged to that select group of Brebis Bleaney's doctoral students in those heady days at the beginning of transition metal and rare earth ion EPR for which Oxford was famous. When I started doing EPR in New Zealand in 1960, I used to scan short reviews of recent physics publications published by the UK Institute of Physics. Known to me then simply as J. M. Baker, he was a frequent contributor reporting on the latest EPR in physics! His early pioneering work on the EPR of rare earth ions in crystals provided important insights into ^{4}f electron configurations, something for which EPR spectroscopy turned out to be essential. Michael's later career focussed on paramagnetic defects in diamond, much of which involved his co-worker Mark Newton, whose tribute to Michael is also published in this issue. Some readers will recall Michael's fine Plenary Lecture at Ampere in Kazan [1994] on defects in diamond.

Towards the end of his career, Oxford University conferred on him the title of Professor, something that was richly deserved.

Ever unassuming, Michael was a dedicated and gifted teacher [Tutor in the Oxford system] and a family man. Those of us who crossed his path professionally came to know him as a good and loyal friend.

John Pilbrow
Emeritus Professor, Monash University

Kálmán Hideg (1934–2018)

Professor Kálmán Hideg passed away on January 19, 2018 at the age of 83.

Kálmán was born in Belsőböcs, a small village in North-East Hungary, on July 9, 1934. His parents Maria and Kálmán were smallholders. He lost his father early, in his teenage years, and his widowed mother took over the small farm with Kálmán's elder brother Lajos. Although he left his native village, Kálmán has always been proud of his industrious "peasant ancestors". He attended secondary schools in Sárospatak and Mezőkövesd, at two prestigious protestant boarding schools, and then moved to Lajos Kossuth University in Debrecen (Hungary) where he obtained an MSc degree in chemistry in 1957. He met his future wife, Olga during his university years, and they got married in 1958 at Reformed Great Church of Debrecen. They were married for almost 60 years, until his death and worked together until Olga's retirement in the 90's.

After graduation, Kálmán was briefly employed as an assistant chemistry lecturer at the Agricultural School in Debrecen. Two years later, in 1959 he and his wife moved to South-West Hungary, to Pécs to work at the Medical School of Pécs University, where he started his independent career until his retirement. Their daughter, Éva was born in Pécs, and she is a professor of Plant Biology at the Faculty of Sciences of Pécs University since 2011.

Early in his carrier, Kálmán worked on heterocyclic chemistry and medicinal chemistry problems and received his PhD degree in 1962. He spent a year at St. Andrews University (UK) in Prof. Douglas Lloyd's group as visiting scientist, working on non-templated N-containing macrocycle synthesis. Continuing this work he obtained a DSc degree in 1979 from the Hungarian Academy of Sciences on his thesis "Access of heterocycles by reactions of dielectrophilic ketones with dinucleophiles." In the 70's Prof. József Tigyi, chair of Biophysical Institute and Medical School, proposed a new topic: "research on stable nitroxide free radicals". Kálmán and his co-workers, including his wife Olga, were successful with this new project, and the laboratory's growing international reputation was marked by the organization of the first Nitroxide Symposium in 1979 at the Medical School of Pécs University. This was the first occasion when scientists studying EPR spectroscopy and stable nitroxide free radicals met from the both sides of "iron curtain".

Later, Kálmán was a visiting scientist in Prof. John Keana laboratory in Eugene, Oregon, USA in 1980. In 1985, he was elected as a distinguished professor of his alma mater, Lajos Kossuth University at Debrecen. Soon after this, he was promoted to full professor at the Pécs University in 1986.

The 80s was an important period in Kálmán's scientific carrier. He published the reversible, SH-specific methanethiosulfonate (MTS) spin label synthesis and its applications with Prof. Larry Berliner, and he created the theory of in vivo scavenging of reactive oxygen species with pre-nitroxides. He was one of pioneers of multitarget-directed compounds, synthesized by combining nitroxides as low molecular weight antioxidants with original drug molecules. This work continued in the 90s in collaboration with Prof. Wayne Hubbell (UCLA), Prof. Peter Fajer (Florida State University), Prof. Derek Marsh (Max Planck Institute), Dr. Murali Krishna Cherukuri (National Cancer Institute) and Prof. Periannan Kuppusamy (Dartmouth College). In 1997, Kálmán became the founding director of the Institute of Organic and Medicinal Chemistry at Pécs University and he held the position until his retirement in 2004. After 2000, he started new collaborations with Prof. John Voss (UC Davis), Prof. Heinz-Juergen Steinhoff (Universität Osnabrück) and Prof. Alex Kokorin (N. Semenov Institute of Chemical Physics, Russian Academy of Sciences). Kálmán remained an active member of the Institute as professor emeritus until the last months of his life.

An organic chemist at heart, he was a passionate and innovative researcher dedicated to synthesis of macrocycles, nitrones, spin traps, cardioprotective and anticancer drugs, and spin labels.

During his long carrier, Kálmán published over 330 research papers, patents and book chapters with over 5000 independent citations. He supervised many MSc students and 8 PhD dissertations, acted as adviser of two habilitations. He was the head of Doctoral School in Pharmacology between 2001–2004. His achievements were recognized by several Hungarian and International decorations, such as the "Eötvös prize" from Hungarian Academy of Sciences and "Silver Medal for Chemistry" from International EPR Society in 2006.

Professor Kálmán Hideg was the recipient of the "Fellow of the International EPR Society" award in 2017.

His legacy lives on.

Periannan Kuppusamy

Messages from Kálmán's collaborators:

"Kálmán's accomplishments were not driven by ambition for fame or money, but based on his high regard for consequential work, independent of the scientific area. As a result, his creativity and productivity in spin label chemistry is unsurpassed. Kálmán appreciated that life is brief and demanding and was dedicated to making his life matter. Let us not forget his singular contributions to the field of EPR." – Prof. John Voss, UC Davis, CA

"Kalman was the embodiment of a 19th century University Professor. Scholarly, erudite, dignified and exacting." – Prof. Piotr Fajer, Florida State University, FL

"Kalman had single-minded devotion to medicinal chemistry that I have to mention an anectode. I was driving him to Johns Hopkins from Bethesda. On I-95 highway, we were caught in a terrible traffic jam. Kálmán continued discussion on structures some of which I could not visualize. To help me, he blew his breath on the window and drew the structures on the moist window glass. To keep a hard copy of these structures, I pulled out at the next exit, went to a restaurant and had him draw them on napkins. Such is the intensity of Kálmán." – Dr. Murali Krishna Cherukuri, NCI, Bethesda

"We will miss Kálmán – as a passionate and enthusiastic scientist, as a longtime and tireless colleague and as a friend. Scientific work was a vocation for Kálmán, his passion for nitroxide chemistry was contagious. His insights, inventions and impulses will certainly accompany us in the future." – Prof. Heinz-Juergen Steinhoff, Universität Osnabrück, Germany

"Kalman was probably the most prolific, insightful organic chemist in the field, especially how his contributions catalyzed the Biochemistry that immediately followed, including applications from his own lab. Kalman was an unselfish inspiration to the spin labeling community" – Prof. Larry Berliner, University of Denver

Reminiscences of Kálmán Hideg

My association and friendship with Kálmán goes back to the late 1970s. I 'may' have been his first American collaborator and frequent host, but I can't be as certain as it was almost a half century ago! Kálmán contacted me about a visit to Ohio State, probably from my earlier spin label work on proteins, but I am also guessing from our edited publication of *Spin Labeling: Theory and Applications* (1976). It's important to note that the entire field, particularly the organic synthetic chemistry, was driven significantly by chemists from the former Soviet bloc (aka the East European Iron Curtain) all of whom I met over that time period, many facilitated by Kálmán organizing the First International Conference on Nitroxides in 1979 in Pećs, Hungary. The conference was first class in every aspect. A large delegation of Soviet Union participants arrived on a special charter flight, perhaps the first time so many of them in the spin label/nitroxide field were allowed to all travel to an international conference.

But I had already been in contact with Kálmán earlier as mentioned. Every time he visited Ohio he would bring me new spin label precursors and present a seminar where rapidly sailed through literally hundreds of compounds that his team had developed since the prior visit! He was the prime example of the clever, productive organic chemist. I was informed that Kálmán and his group has synthesized more than 8000 new compounds, most of them are nitroxides or nitroxide precursors.

By 1980, we were naively trying to make a reversible spin label thiol reagent but were amateur organic chemists at the best. Our synthetic schemes were not sophisticated enough to allow an easy route to the desired product. After less than a year, Kálmán arrived for another annual visit and presented a talk with over 100 new compounds displayed at his usual pace of 15–20 per slide. All of a sudden, I saw the precursor to my desired label (eventually coined MTSL or MTSSL)! I asked Kálmán to go back to that structure, showed him the desired final structure and arrogantly said, "Kálmán, if you can produce this label, I'll make you famous!"

The compound was ready in just a few months, but the other problem was whether we could ship it from a communist country to the USA (or another non-communist country) without lots of 'red tape', delays or potentially never receiving it. At the time, spring, 1981, I was working in Israel for a short term visiting professorship. As it turns out, I returned to the USA for a thrombosis meeting in Toronto at the same time that Kálmán attended a free radical meeting in, I believe Guelph, that Ed Janzen organized. Clandestinely, we met in the passenger terminal at a prearranged time, he handed me the sample from his pocket and I took it back to Haifa with me to work with a past postdoctoral student (now working in Ness Ziona) on the active site cysteinyl enzyme, papain. In less than two days, we had impressive proof of concept results, which we repeated in Columbus and submitted shortly thereafter to *Biochemistry*. While I might mention that there's a plethora of papers utilizing MTSL in *Biochemistry*, the journal at that time rejected our paper on the pretense of not being relevant. It was, however, quickly accepted by *Analytical Biochemistry*. Soon after publication my past Stanford friend and lab mate, Wayne Hubbell, telephoned me about his interest in MTSL with a very clever idea to genetically incorporate cysteines selectively in proteins and subsequently spin label with high specificity. This quickly became known as site-directed spin labeling or SDSL. It was truly a renaissance in the field and resulted in many collaborations between Kálmán and Wayne.

Kálmán bemoaned the fact to me that, despite being the senior author on the paper, all of the community called it the Berliner label. I tried to convince him otherwise but, unfortunately, Kálmán's strong pride was hurt and he ceased future visits to Ohio for over a decade, much to my disappointment. Sometime in late 90s, I was invited by Kuppusamy to Baltimore which coincided with Kálmán's regular collaboration visit to Kuppusamy's group at Johns Hopkins. We had discussions and dinner together where the atmosphere was like nothing had transpired in the interim. Kálmán presented me with photos from the Pećs meeting. It was a wonderful reunion! I would see him occasionally at scientific meetings, but I believe that his health problems prevented him from traveling as frequently as in the past.

Kálmán's influence in the progress of these fields was quite significant. I will miss him dearly.

Larry Berliner

Charles P. Slichter (1924–2018)*

The great magnetic resonance physicist, Charles Pence Slichter, passed away peacefully on Feb. 19, 2018, at the age of 94, in Boulder, Colorado. Charlie (as he liked to be called) was one of the early pioneers in the development of magnetic resonance methods, making numerous contributions to NMR, EPR, and NQR. He was President of ISMAR from 1987 to 1989, received the ISMAR prize in 1986, and became an ISMAR Fellow in 2008. Charlie was universally admired, not only for his scientific accomplishments, but also for his personal warmth, generosity, and positive outlook on life and science.

Charlie was born on Jan. 23, 1924 in Ithaca, NY. He received his Ph.D. in 1949 from Harvard University under the direction of Prof. Edward Purcell, then joined the faculty of the University of Illinois in Champaign-Urbana, where he remained for his entire career. He was promoted to Full Professor in 1955 (at the age of 31!). He formally retired in 1997, but continued other physics-related activities (e.g., attending colloquia, participating in group meetings of colleagues, reviewing papers, etc.) until his death.

Charlie's service contributions, both national and international, were considerable. In addition to his service to ISMAR, he was a member of President Lyndon Johnson's Science Advisory Committee (PSAC) from 1965 to 1969, a member of the Harvard Corporation from 1970 to 1995, and a member of the National Science Board from 1975 to 1984.

In addition to his honors from ISMAR, Charlie received many other honors during his illustrious career, including being an Alfred P. Sloan Fellow (1955–1961), a member of the National Academy of Sciences (1967), a Fellow of the American Academy of Arts and Sciences (1969), a member of the American Philosophical Society (1971), and a Fellow of the American Physical Society (APS) (1955). He received the APS Langmuir Prize in Chemical Physics in 1969, the U.S. Department of Energy's Division of Materials Science Award for Sustained Outstanding Research in Solid State Physics in 1984 and again in 1992, the Department of Energy's Award for Outstanding Scientific Accomplishment in 1993, the Comstock Prize of the National Academy of Sciences in 1993, and the APS Oliver E. Buckley Prize in Condensed Matter Physics in 1996. Charlie received the National Medal of Science from the U.S. President in 2007. He also received honorary doctoral degrees in Science from the Universities of Waterloo (1993) and Leipzig (2010) and an honorary doctorate in Law from Harvard (1996).

Charlie's research spanned many subfields of physics and chemistry and was characterized by elegant theoretical analyses and many, many beautiful experiments. His research not only answered fundamental questions in science but also resulted in the development of important techniques in magnetic resonance, many of which are widely used today. One of his most important early contributions to solid state physics was his measurements of the temperature dependence of spin-lattice relaxation in aluminum, which provided the first experimental support for the electron pairing concept of the Bardeen-Cooper-Schrieffer (BCS) theory of superconductivity. In this landmark study, first reported only four months after the first paper by BCS in 1957, Charlie and his student Chuck Hebel observed an unanticipated peak in $1/T_1$ immediately below the superconducting transition temperature, arising from coherence factors unique to BCS. Slichter and Hebel then worked out the theory to explain this peak (now widely known as the "Hebel-Slichter peak") using the BCS wavefunction. In order to do this experiment, Slichter and Hebel first had to develop techniques for measuring NMR relaxation experiment in zero magnetic field. Their approach led to such methods as field cycling and adiabatic demagnetization in the rotating frame (ADRF).

Charlie and his students made important contributions to the development of double resonance techniques, including nuclear-nuclear double resonance and electron-nuclear double resonance (ENDOR). In a famous experiment, Charlie and his student Tom Carver provided the first experimental verification of Albert Overhauser's prediction that saturation of the conduction electron resonance in a metal would lead to a 1000-fold increase in the polarization of the nuclear spins (the original "Overhauser effect"). When Charlie reported these results in a meeting of the APS, he was lambasted by a group of skeptical physicists, including several Nobel Prize winners who thought (incorrectly) that Overhauser's ideas violated the second law of thermodynamics. (Erwin Hahn facetiously described this as a Nobel Prize grilling.) The Carver-Slichter experiment was the first demonstration of dynamic nuclear polarization (DNP), a phenomenon that continues to grow in importance to the present day.

Charlie's contributions to chemistry were also significant. He was a co-discoverer (with Herb Gutowsky and Dave McCall) of indirect spin-spin (J) coupling in molecules. He also developed a theory of chemical shifts in which he explained the origin of the large ^{19}F paramagnetic chemical shifts. In addition, Charlie provided the first detailed theory of the effect of rate processes on NMR spectra.

In 2017, the American Chemical Society recognized the discovery of J couplings at the University of Illinois with a Chemical Breakthrough Award. Charlie's own description of this discovery can be found at http://physics.illinois.edu/news/article/23848.

Other important achievements include the first absolute measurement of the electron spin susceptibility in metals, elucidation of the Kondo effect in solids by observation of nuclear quadrupolar satellites near magnetic atoms in dilute alloys, and observations of the motions of charge density waves in solids under applied electric fields. Charlie pioneered the use of rotating frame relaxation to extend by orders of magnitude the range over which translational diffusion and molecular motions could be observed in solids, developed spin temperature techniques for solving otherwise intractable problems such as calculating spin-lattice relaxation in zero applied field and in the ultraslow motion regime, and introduced phase-coherent detection of pulsed NMR

---

* Reproduced from http://www.weizmann.ac.il/ISMAR/charles-p-slichter-1924-2018 with permission from ISMAR. Special thanks go to David Ailion. Photo courtesy of Jürgen Haase.

(widely used in modern NMR equipment), thereby allowing the measurement of NMR signals that are much weaker than the noise.

Charlie's personal account of the history of superconductivity, including a recording in his own voice, is available at http://history.aip.org/history/exhibits/mod/superconductivity/01.html. Additional information about Charlie's life and times can be found at http://jfi.uchicago.edu/~leop/AboutPapers/Slichter-Bardeen.pdf and http://engineering.dev.engr.illinois.edu/news/article/24443.

And then, of course, there is Charlie's influential textbook, "Principles of Magnetic Resonance". The first edition of this book resulted from lectures that Charlie gave in the Spring of 1961 when he was a Morris Loeb Lecturer at Harvard. Subsequent editions greatly expanded the scope of the book to include more recent developments in multiple-pulse techniques, two-dimensional spectroscopy, magnetic resonance imaging, and multiple quantum NMR. One of the most significant characteristics of this book is Charlie's method of describing different ways of looking at the same phenomenon as leading to an apparent contradiction, which he then proceeds to explain completely and clearly, thereby resolving the apparent contradiction. This fantastic book is still widely read by students of magnetic resonance and used as a basic reference by many of us, more than 50 years after its initial publication.

In addition to his outstanding research record, Charlie was one of the great physics teachers of our time. He supervised approximately 64 Ph.D. students and over 50 postdocs (including Sir Peter Mansfield, who received the Nobel Prize as a co-inventor of MRI). Many of these students and postdocs have gone on to leading positions in education and industry and have thus spawned even more great contributions.

Charlie had an interesting low-key approach to getting his students to work longer hours. He would show up in the lab at 8:00 AM for coffee and then disappear for the rest of the day. If we wanted to talk to him, we had to be in the lab by 8:00 AM. Charlie would always start conversations by saying "How did it go last night?". Yes, we were expected to work long hours at night and still show up early in the morning.

Charlie had an amusing way of achieving privacy when necessary, even though he hated to say no to a student with questions. He acquired an additional nameplate with the name "Roger Willoughby" that he placed on the door to his office, covering his name. Whenever we saw that, we knew that he was not to be interrupted, yet solicitors and others would not know where to find him.

As many of his friends are aware, Charlie had a somewhat round head (or "moon-shaped" as he liked to say). We had in the lab a large and completely round 50 liter vessel on wheels for transporting liquid nitrogen between rooms. In order for us not to lose this vessel to another lab, we painted the letters "C.P.S." on it. Charlie seemed to identify with this N_2 vessel, since, whenever it was wheeled into his presence, he would break out into a broad grin.

Charlie's interests in his former students and postdocs did not end with their leaving his labs. A "Slichter tradition" at each March Meeting of the APS for about 30 years was that all attending former Slichter students and postdocs and their students, etc. were invited to dinner, usually at a Chinese restaurant on a Tuesday night. Charlie's sensitivities to the needs of students were illustrated by the requirement that all current students would be free and the rest of us would cheerfully chip in to cover their costs.

Charlie's love for interactions with students is typified by a statement that he made at the 80th symposium held in his honor at the University of Illinois: "...If I were not in a research setting, I would have to find students to work with."

Charles P. Slichter was greatly loved and will be sorely missed.

We thank Prof. David C. Ailion for preparing this obituary. David received his Ph.D. in physics from the University of Illinois for experiments in Charlie Slichter's lab in the mid-1960s, which demonstrated the use of rotating-frame NMR relaxation as a probe of ultra-slow atomic translational motions in solids. David also served as Treasurer of ISMAR from 2011 through 2017.

* * *

Early last year, Charlie and I engaged in a very pleasant email correspondence which led to us both preparing tributes to the late Erwin Hahn [EPR Newsletter **27**, 1-2, 2017]. Charlie had known Erwin since 1949, having come from Harvard where he'd gained his PhD in NMR under Edward Purcell. Charlie's lengthy article contained much anecdotal material that most would not have known. So the pages of the *EPR Newsletter* continue to secure connection with the history of our subject.

I cannot claim to have been a special friend of Charlie's as our paths crossed only a few times over almost 50 years! However I shall recount the nature of these encounters briefly.

It was in 1962 that I encountered Charlie's *Principles of Magnetic Resonance*, based on lectures he'd given at Harvard the year before. And then from October of 1962, Professor Larry Slifkin from the University of North Carolina joined the Hayes Group in the Clarendon Lab in Oxford and it was arranged he would work with me. Larry would teach me about the silver halides and I would teach him the rudiments of EPR.

Slifkin and I had a very fruitful 10 months or so studying Fe^{3+} in both AgCl and AgBr. Larry, who'd been a Postdoc in Physics at the University of Illinois a decade earlier and had

▶

From left to right: John Pilbrow, Charlie Slichter and Karl Hausser.

got to know Charlie, discovered that Charlie had a student also working on the EPR of the same system but we had no details.

My attempts to observe Chlorine ENDOR in AgCl:Fe^{3+} was not successful. So I made my first foray into EPR simulations and was able to obtain credible results for the Cl hfs and also reasonable estimates for the Cl quadrupole parameters [Hayes, Pilbrow & Slifkin: J. Phys. Chem. Solids **25**, 1417, 1964]. Slichter's student, J. G. Garth succeeded in achieving the chlorine ENDOR, carried out at 4.2 K which, of course, provided more accurate Cl hfs and quadrupole tensors [Garth: Phys. Rev. **140**, A656]. Then, the following year, Charlie and his Japanese Postdoc, M. Satoh, reported observation of silver ENDOR 4.2 K, [Satoh & Slichter: Phys. Rev. **144**, 259, 1966], confirming the correctness of the model proposed by Slifkin and me. My mistake had been to limit ENDOR experiments to 20 K, as liquid H_2 was plentiful at Oxford whereas though liquid Helium was available it was rationed!

I moved to Australia in 1965 to the Department of Physics at Monash University and in 1966 Martin Spaeth came for about 10 months after gaining his doctorate at the Technische Hochschule in Stuttgart. On our way home one evening we called in at our local Community Library and to our great astonishment discovered a copy of Charlie's book, something of an anomaly there! Martin was actually looking for books on Australian Indigenous Art as I recall.

Early in 1967 I was asked by the late Dr Clive Coogan from the neighbouring CSIRO Chemical Physics Division if I would be Secretary for The International Symposium on Electron & Nuclear Magnetic Resonace to be held at Monash University during August 1969. The Organising Committee had five international members including Robert Blinc, Herb Gutowsky and Charlie. Clive was on good terms with both Herb and Charlie as he had spent a couple of years in the mid-50's working with Gutowsky at Urbana, during which time he also got to know Charlie. But I didn't meet Charlie and the others until they arrived for the Symposium. I bumped into Charlie just before the official Opening Ceremony and had the first of many most enjoyable chats; he was very easy to talk to. I recall that Charlie examined a PhD Thesis for a student of my colleague Gordon Troup back in 1967.

I met Charlie again during my sabbatical in Urbana between June 1971 and January 1972 where I worked with Linn Belford in the Chemistry Department. I had many chats with Charlie when I attended seminars in the Physics Dept. Early in Jan 1972 I was invited to give a talk to Charlie's Group about the work Linn and I had been doing on **g** and **A** matrices for Cu^{2+} in monoclinic sites to which he responded to most generously.

It was not until a visit to Urbana in September 1986 where I presented a seminar at Urbana in the Chemistry Dept that we met up again. Charlie had advised us he planned to attend, and although several physicists came such as Harvey Stapleton and some of his group, there was no Charlie! It turned out he'd been at Harvard the day before, where he was a Member of the Harvard Corporation [on which he served from 1970 to 1995], and had not looked at his diary. However I went to see him late in the afternoon when we had a very enjoyable chat. My topic that day included a detailed discussion of the differences between field sweep [as is usual in EPR] and frequency sweep. I had particularly specialised in $S = 1/2$ systems. But for $S > 1/2$, it was Robert Coffman [J. Phys. Chem. **79**, 1129] who had generalised a result for the detected signal derived by Charlie in his book to include magnetic field [B] explicitly along with frequency [ν]. Charlie wanted to know more about what I had discussed and he was initially a bit puzzled. Then I remember saying, "But it's in your book, Sir" and then he realised that he had established the germ of the argument in his book!

It was until 1998 at the Ampere Congress in Berlin that our paths again crossed. As IES Secretary, I deputised for Jim Norris in presenting IES Fellowships to both Charlie and Karl Hauser. Karl, incidentally, who had attended the conference at Monash University in 1969 was by this time becoming quite frail. When Charlie saw that Karl was remaining seated for the presentation, he also remained seated so as not to cause embarrassment. The mark of a true gentleman!

The last time we met face-to-face was in March 1999 during the American Physical Society Centenary Conference in Atlanta [Attendance ~ 12,000]. I along with some 40 other National Presidents of Physics Societies from around the world was a guest of the APS. Again we managed a nice conversation in course of which he reminded me of the presentation in Berlin the year before and that he had worried whether he was right to have remained seated. I assured him it had been a most appropriate gesture.

Whenever we met, Charlie always greeted me as if I were a long lost friend! And I am sure others would be able to testify to the same experience. He was always courteous and pleased to see one. I always knew that I was in the presence of someone of sharp intellect but who was always generous in acknowledging the work of others.

Charlie Slichter's passing has taken from us one of the pioneers of magnetic resonance whose contributions spanned a wide range of problems in NMR and, of course, EPR. His NMR contributions involved superconductivity, the Overhauser Effect, with Gutowsky and McCall the discovery of *J*-coupling, measurement of the Pauli susceptibility of conduction electrons and much more.

I am glad that I had several opportunities to talk with and learn from a great scientist and a very fine person.

We all express condolences to Charles's family at this time.

John Pilbrow
Melbourne, Australia, 23 April 2018

---

notices of Meetings

EMBO workshop on 'Challenges for magnetic resonance in life sciences'
Grosseto, Italy, May 27–31, 2018
Contact: embo@cerm.unifi.it
Web: http://meetings.embo.org/event/18-nmr

International EPR School
Marseille and Carry-Le-Rouet, France, June 3–7, 2018
Contact: guigliar@imm.cnrs.fr
Web: www.a-rpe.fr

EUROMAR 2018
Nantes, France, July 1–5, 2018
Contact: info@euromar2018.org
Web: www.euromar2018.org/EventPortal/Information/EUROMAR18/HOME.aspx

Rocky Mountain Conference 2018
Snowbird, Utah, July 22–26, 2018
Contact: info@rockychem.com
Web: www.rockychem.com

APES-IES 2018
Brisbane, Australia, September 24–28, 2018
Contact: apes_ies2018@uq.edu
Web: www.apes-ies2018.org

Annual International Conference "Modern Development of Magnetic Resonance-2017" (MDMR2017)

September 25–29, 2017, Kazan, Russia

From left to right: Alexey A. Kalachev, Director of the Kazan Physical-Technical Institute, Il'shat. R. Gafurov, Rector of the Kazan Federal University, Takeji Takui, Zavoisky Awardee 2017, Myakzyum Kh. Salakhov, President of the Academy of Sciences of the Republic of Tatarstan, Kev M. Salikhov, Chairman of the Zavoisky Award Selection Committee, and Endel' N. Fattakhov, Deputy Prime-Minister of the Republic of Tatarstan.

In September 2017, Kazan welcomed 120 participants from Belgium, China, France, Germany, Israel, Japan, the Netherlands, Russia, Sweden, and the United States in order to discuss achievements and new tendencies in applications of magnetic resonance within the Annual International Conference "Modern Development of Magnetic Resonance 2017" dated to the Zavoisky Award 2017 ceremony. Professor Takeji Takui (Osaka City University, Japan) got the Zavoisky Award 2017 for his outstanding contributions to the development of organic high-spin and open-shell molecules and their EPR-based quantum spin technology.

The conference was organized by the Zavoisky Physical-Technical Institute of the Russian Academy of Sciences and the Kazan Federal University under the auspices of the Groupement AMPERE. It included eight plenary lectures, fifty-two invited and oral talks, and sixty posters within the following sections: Theory of magnetic resonance; Low-dimensional systems and nano-systems; Electron spin based methods for electronic and spatial structure determination in physics, chemistry and biology; Molecular magnets and liquid crystals; Spin-based information processing; Strongly correlated electron systems; Chemical and biological systems; Medical physics; Magnetic resonance imaging; Other applications of magnetic resonance; Modern methods of magnetic resonance; Magnetic resonance instrumentation; Related phenomena. A special session was devoted to the 110th anniversary of Evgeny K. Zavoisky, who discovered the phenomenon of electron paramagnetic resonance in 1944.

The Zavoisky lecture of Takeji Takui "Topological High-Spin Organic Chemistry and Molecular Spin-Qubit Quantum Technology Underlain by Electron Magnetic Resonance" was devoted to multispin molecules, which are of great interest from the point of view of the creation of monomolecular magnets and spin based quantum computing. Impressive results on the development of terahertz EPR spectroscopy were highlighted by Hitoshi Ohta in his plenary lecture "Multi-Extreme THz ESR: Developments and Future Biological Applications". The plenary lecture of Aleksandr I. Smirnov "Spinon Magnetic Resonance in a Quasi 1D $S = 1/2$ Antiferromagnet with a Weak Exchange Interaction" was concerned with model objects of great fundamental importance for physics of macroscopic quantum phenomena. Plenary lectures by Martina Huber "EPR Methods to Determine Properties of Intrinsically Disordered Proteins" and Robert Bittl "Insight into Protein Function by EPR" demonstrated the applications of EPR spectroscopy in biology.

The lecture "Soft Spins and Higgs Mode in Ruthenates" was given by Giniyat Khaliullin, and Michael K. Bowman et al. presented the lecture "The Different Faces of Free Radical Spin Dynamics in Frozen Solution". The plenary lecture of Valery F. Tarasov was devoted to studying dimer self-organization of rare-earth impurity ions in synthetic forsterite, while Elena G. Bagryanskaya in her lecture "Pulse Dipole EPR-Based Distance Measurements at Ambient Temperatures" discussed recent developments and perspectives in the field of EPR spectroscopy of spin labels.

The conference demonstrated the increasing interest in magnetic resonance studies in diverse fields of science. The financial support of the Government of the Republic of Tatarstan, the Russian Foundation for Basic Research, and Bruker BioSpin is gratefully appreciated.

Kev M. Salikhov,
Co-Chairman of the MDMR 2017
Violeta K. Voronkova,
Scientific Secretary of the MDMR 2017

---

notices of Meetings

The 2018 ACERT Hands-On Tutorial Workshop on Denoising ESR Signals Via Wavelets

Cornell University, Ithaca, NY, USA, July 30–31, 2018
Contact: acert@cornell.edu
Web: www.acert.cornell.edu/index_files/acert_news.php

School for young scientists "Magnetic Resonance and Magnetic Phenomena in Chemical and Biological Physics"

Roschino, St. Petersburg (Leningrad) region, Russia, September 15–20, 2018
Contact: mrschool2018@gmail.com
Web: http://mrschool2018.from-siberia.ru

"Modern Development of Magnetic Resonance 2018" (MDMR2018)

Kazan, Russian Federation, September 24–28, 2018
Contact: mdmr.kazan@yandex.ru
Web: http://kfti.knc.ru/mdmr/2018

Pre and post tests. What to ask, why, and the results

Chapter 3

by Reef Morse

How do you know that the students you are teaching learned anything you taught them? What knowledge do they bring to the table/classroom that is pertinent to the subject you are teaching? How varied is their background?

These are questions that all of us as educators face in every teaching/learning situation. One of the ways to obtain this information is to find out what they knew before they took your class, and what they know after taking your class. In a traditional class environment, this is usually sorted out by the exams we give: unprepared students do poorly; prepared students do well. We test based on what we teach since the object of the class is to pass rather specific knowledge about the subject to the student.

The pre-test I give the SMART Center students has three types of questions. The first type are questions that I am sure to cover in the class and that I'm pretty sure a student will know nothing about. An example of such a question is "Compare and contrast T_1 and T_2". The second type of question checks for prior knowledge; for example "What steps are taken in designing and performing a scientific experiment?". This usually dredges up some list of 3, or 5, or 8 steps that the student was taught in some science class. The last type is questions that I'm not likely to cover explicitly but deal with subject content appropriate to the topic, for example "What is the effect of oxygen concentration on an EPR spectrum?"

An example of scoring is shown in Table 1. The course that session did not cover material in Question 5. An interesting observation is that, even if the students did not know the answer, they were much more willing to try to answer the question after they had the course compared to before they had the course. The high success rate on the T_1/T_2 question was the result of a hands-on double pendulum experiment where they could easily see the transfer of energy from one pendulum to another (T_2) and decay of the motion of both pendula (T_1). Pedagogically, this provided a smooth transition into the discussion of line widths and microwave saturation and served to provide an intimate experience of relaxation mechanisms. Although I have modified, sometimes drastically, almost every other part of the course, I have not changed this experiment much.

I do use a comparison of pre- and post-tests to see if there were any conceptual problems, material I covered well or poorly, areas that the students seemed to understand during the class but did not have clarity to convey concepts well, and what I can do better. This has led to an evolving course structure not based on changes in the scientific concepts, but on the ability of the students to understand and work with the ideas.

Over the 10 years I have been teaching this course, I have noticed changes in attention span, willingness to work with ideas and instruments they are not comfortable with, and willingness to question and think about their results. There are exceptions, but, in general, the population is changing in their thinking habits. I have the test results to prove it.

Table 1. Scoring pre and post test questions. Students need to get the question correct to get full credit (1 point) for each question but partial credit is given.

Questions	Pre-Test Attempts	Correct	Post-Test Attempts	Correct
1. Define spectroscopy	13	1	13	7
2. What does EPR stand for?	9	0	13	11
3. List at least two common items that would give an EPR signal.	3	0.5	13	13
4. Compare and contrast T1 and T2.	3	0.5	13	13
5. Briefly, how would the following physical sample parameters affect an EPR spectrum:				
A. Sample viscosity	2	0	12	3
B. Probe concentration	3	0	12	1
C. Polarity (water vs oil, for example)	1	0	10	1
D. Oxygen concentration	1	0	11	2
6. Briefly, how would the following instrumental settings affect and EPR spectrum:				
A. Modulation amplitude	3	0	13	8
B. Gain	1	1	13	6
C. Field Center	2	0	13	10
D. Sweep width	2	0	13	8.5
7. What steps are taken in designing and performing a scientific experiment? Explain the purpose and desired outcome of each step. Number each step for use in question 8.	13	13	13	13
8. What biases do you have that might affect each of the steps above? Use the step numbers from question 7.	13	13	9	7
9. Explain how you would organize and present your research to: your family, your school classmantes, other students at the SMART Center, other scientists at the SMART Center, a group of scientists at a scientific conference, a newspaper reporter	13	13	13	13
10. Draw the chemical structure of a stable free radical.	4	0.5	11	7.5

POSITIONS

Research Specialist Senior Position at West Virginia University

The Department of Biochemistry is searching for a Research Specialist Senior, with a strong background in RF/MW engineering experience. This position is available immediately and will be in the In Vivo EPR Multifunctional Magnetic Resonance center, Department of Biochemistry, Health Sciences Center, West Virginia University in Morgantown, WV. The selected candidate will assist Dr. Tseytlin in designing and manufacturing electron paramagnetic resonance (EPR) spectrometers and imagers. The position will be for one year in length, with a possibility of extension. The duties and responsibilities for this position are: designing, manufacturing, assembling, and experimental testing of EPR spectrometers and imagers. Applicants must hold a minimum of Master's Degree (or foreign equivalents) in Electrical Engineering, Physics or a related field and two years of experience, or a combination of education and experience. Qualifications must be met by time of appointment. All interested, qualified candidates should apply to jobs.wvu.edu with a cover letter of interest and current CV.

West Virginia University is an Equal Opportunity/Affirmative Action Employer and the recipient of an NSF ADVANCE award for gender equity. The University values diversity among its faculty, staff and students, and invites applications from all qualified individuals, including minorities, females, individuals with disabilities and veterans.

Postdoc in optical nuclear magnetic resonance, joint Los Alamos/UNM, USA

The quantum nanophotonics and biosensing lab at U. of New Mexico (PI: Victor Acosta) and the atomic magnetometer/NMR/MRI labs at Los Alamos National Lab (PI: Igor Savukov) seek a motivated postdoctoral candidate for a joint UNM/LANL project. The successful candidate will work on a highly multidisciplinary project that combines new techniques in optical nuclear polarization using NV centers in diamond nanostructures with low-field and optically-detected NMR/MRI. The goal is to develop a microfluidic platform operating at room temperature and low magnetic field which can deliver biochemical analytes with a nuclear polarization exceeding that possible using large superconducting magnets and/or cryogenic temperatures.

While working on the project, the postdoc will:

- Apply quantum mechanics, nanophotonics, and biochemistry to emerging new fields.
- Learn semiconductor nanofabrication techniques in world-class cleanrooms (CHTM, CINT).
- Work with partners in academia, national labs (LANL, Sandia), and industry (ODMR Tech).

The position will initially be based in Albuquerque, NM at the UNM lab. There the work will be focused on developing the NV hyperpolarization apparatus. Then the postdoc will transition to conducting NMR/MRI experiments at LANL to characterize and optimize its performance.

A Ph.D. in Physics, Chemistry, Optical/Electrical/Chemical/Biomedical Engineering, or a related field is required. Experience in spin physics, biophysics tool development, and/or quantum optics is desired. Experience in magnetometry, semiconductor nanofabrication, and/or solid-state color centers is also helpful. Compensation is commensurate with experience and is expected to increase when work transitions to LANL. Candidates with very strong publication records will have an opportunity to apply to highly competitive LANL fellowship programs. More information is available on our webpage. Interested candidates, please send Cover letter (describing research interests and career goals) and CV (with complete publication record) to Victor Acosta (vmacosta@unm.edu) and Igor Savukov (isavukov@lanl.gov). UNM and LANL are equal opportunity employers.

Postdoctoral Fellow

The Magnetic Resonance Spectroscopy group at Rensselaer Polytechnic Institute (RPI) is conducting cuttingedge research in the fields of solar energy transduction in natural and artificial systems and the development of novel materials and solar technologies. By performing fundamental and applied research, we work on sustainable solutions for major challenges facing energy and the environment. RPI is committed to the training of future scientists and engineers and is one of the oldest science and engineering universities in the United States. We are looking for a Postdoctoral Fellow for advanced multi-frequency pulsed electron paramagnetic resonance (EPR) spectroscopy of natural and artificial systems.

Your tasks

– Operation and further development of pulsed EPR spectroscopy experiments (including ENDOR, HYSCORE and transient EPR spectroscopy)

– Performance of experiments on redox proteins, metal oxides and thin films

– Analysis and numerical simulation of experimental data

– Presentation of scientific results at national and international conferences

– Publication of scientific results in international journals

– Scientific collaboration with graduate and undergraduate researchers at RPI

Your profile

You are a flexible team member able to work independently on different projects. You have completed your Ph.D. in chemistry or physics and have experience with pulsed EPR spectroscopy. A good command of various software tools enables you to analyze pulsed EPR spectra readily. You have active interest in experimental work with good practical skills and enjoy working in an interdisciplinary team. Your broad knowledge in physical chemistry and spin physics is a valuable asset to understanding the results of complex experiments. Good communication skills in English are required.

For further information please contact:

Prof. K. V. Lakshmi
Department of Chemistry and Chemical Biology and The Baruch '60 Center for Biochemical Solar Energy Research Rensselaer Polytechnic Institute
Troy, NY 12180
e-mail: lakshk@rpi.edu
lakshmi@baruch60center.org
phone: (518) 698 7976

Please send your application materials to Prof. K. V. Lakshmi through e-mail correspondence.

Cryogenic EPR Postdoctoral Position

A postdoctoral position is available immediately for a collaborative project between Professor Cory's lab at The Institute for Quantum Computing (IQC) and a local startup. The project uses superconducting resonators and Optimal Control Theory (OCT) to increase the sensitivity of pulsed electron spin resonance at cryogenic temperatures. The goal of the project is to also demonstrate applications to molecularly thin samples. This program is expected to reveal new and interesting results of EPR of biochemical processes.

The successful applicant should have an advanced degree (Ph.D.) in chemistry, biochemistry or physics with significant experience in EPR of biomolecules.

The appointment will be for two years with the possibility of renewal. The salary is competitive and commensurate with experience, ranging from $55,000 to $70,000. Women and minorities are encouraged to apply.

Apply to grum.teklemariam@highqlp.com.

Post-doctoral research associate

We are looking for post-doctoral associates interested in measurement/instrumentation development of EPR spectroscopy applied to energy and biologically-derived nanomaterials to join us at the Center for Nanoscale Science & Technology (http://cnst.nist.gov) at NIST, a highly interdisciplinary and very well resourced user facility. My laboratory contains a Bruker E580 EPR spectrometer (X-band) as well as a NIST-designed spectrometer (Q-band), both of which are equipped with arbitrary waveform generators for pulse-shaping. UV and visible CW lasers have been interfaced with the instruments to enable photoexcitation of samples. Current work is in the areas of spectroelectrochemical EPR spectroscopy development for solid catalyst materials and nanoscale structural measurements of biomaterials, including DNA-based nanomaterials. The ideal candidate would lead a project that makes use of the CNST nanofabrication capabilities as a means to provide meaningful EPR-based measurements. Typical successful applicants have a strong research background and academic record. Letters of reference are required.

Contact: Dr. Veronika Szalai
phone: 301-975-3792;
e-mail: veronika.szalai@nist.gov

Available: Used EPR Spectroscopist

Old, but well maintained EPR Spectroscopist, with wide application experience, seeks opportunity to support active research group. EPR- based publications over 5 decades, most recently 2011, mass spectrometry publications to 2014. Experience in the study of transient organic and organometallic free-radicals; transition metal complexes; spin-trapping; spin probe; radical ions; matrix isolation and γ-radiolysis techniques; photocatalysis; polymer degradation and stabilisation etc. Has Bruker, Varian and JEOL operating experience, plus track record in NMR and mass spectrometry techniques. Would prefer to work in biochem/biological area, but would consider anything interesting. Opportunity arises due to restructure of Research Department in Australian steel company after 26 years faithful service. Excellent grant writing skills, 100% success rate. Speaks English, German and some French.

Please Contact: Phil Barker pba02985@bigond.net.au or pbarker@uow.edu.au to receive user logbook and publications list.

Postdoctoral Associateships in Magnetics at NIST

We offer postdoctoral opportunities in magnetics at the National Institute of Standards and Technology in Boulder, Colorado, USA. Annual salary is $65,600 plus benefits. Appointments are for two years. Application deadlines are 1 February and 1 August annually (but inquire earlier).

The application process is competitive. Typical successful applicants have a strong research background and academic record. Letters of reference and an original research proposal are required.

U.S. citizenship and a background investigation are required (no exceptions). www.nist.gov/pml/electromagnetics/magnetics

EPR Specialist Position at Johns Hopkins

Postdoctoral or specialist (staff) position is available immediately to study membrane proteins at the Johns Hopkins University School of Medicine in Baltimore, Maryland, USA. We study conserved membrane enzymes with implications for human health (see Nature Chem Biol 8:759, eLife 1:e00173, and Nature Rev Micro 7:411), and are generously funded by the National Institutes of Health (NIH) and the Howard Hughes Medical Institute (HHMI). The project uses site-directed spin labeling (SDSL) with nitroxide probes to study the dynamics, distance measurements, and saturation kinetics with CW-EPR methods. The applicant must have at least 3 years of prior experience in SDSL, EPR, spectrum simulations, and distance measurements as evidenced by publications. Experience with membrane proteins is preferred but not essential. Position will come with generous salary and benefits, depending on experience and record of achievement. Interested applicants please send detailed CV and contact information for 3 references to rosanna@jhmi.edu.

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

Please send resume, cover letter and salary requirements to bruker.jobseprfse0620@bruker-biospin.com

EQUIPMENT

Wanted: Badly needed certain parts of, or even a complete Bruker X-Band microwave unit from the mid-seventies, the one which came with the Bruker B-ER 420 system. Particularly, the klystron heating and protection board, B-E-Z 10. Please contact Prof. Dr. Wolfgang E. Trommer, Department of Chemistry, TU Kaiserslautern, P.O.Box 3049, D-67653 Kaiserslautern, Germany. E-mail: trommer@chemie.uni-kl.de.

EPR parts, electronics and hardware

Pulse generators, amplifiers, frequency counters, etc. We also offer X-band cavities, waveguide, klystrons, cells, etc. for Varian instruments.

Please contact techepr03@gmail.com for availability and pricing.

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, phone: 1-303-871-2419

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

SPIN PHYSICS

Cryofree® research tools for quantum technologies and physical sciences

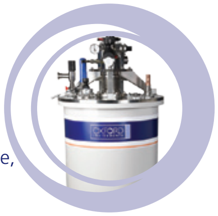
Low temperatures and high magnetic fields

OptistatDry cryogenic systems for optical and spectroscopy applications

Triton ultra low temperature, integrated dilution refrigerator/ magnet systems

Io compact continuous cooling solution for milliKelvin devices

Our tools support a wide range of research in low temperature and condensed matter physics, addressing the needs of spin physics and chemistry through specialised EPR and solid state NMR designs, ranging from bulk sample measurements to spin qubit development.



TeslatronPT low temperature, magnet systems

Custom-engineered superconducting magnet systems

OXFORD INSTRUMENTS

The Business of Science®

nanoscience@oxinst.com

www.oxford-instruments.com/nanoscience

EPR makes a Quantum Leap

Bruker's Benchtop microESR™ spectrometer for industrial, academic labs and educational institutions.

BRUKER

- Spin-trapping (PBN, TEMPOL, DMPO)
- Shelf life of food products (vegetable oil, beer, wine)
- Crude oil analysis: asphaltene & vanadium content
- Biodiesel oxidative stability
- Catalyst coking
- Stability of emulsions & solids
- Lipid oxidation
- Lubricants analysis: oxidation of engine oil, hydraulic oil & turbine oil
- Soot

www.bruker.com

Creating an Exciting World of EPR...

BRUKER

www.bruker.com/epr


epr news letter
The Publication of the International EPR (ESR) Society