epr newsletter

2005
volume 15 number 2

The Publication of the International EPR (ESR) Society

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

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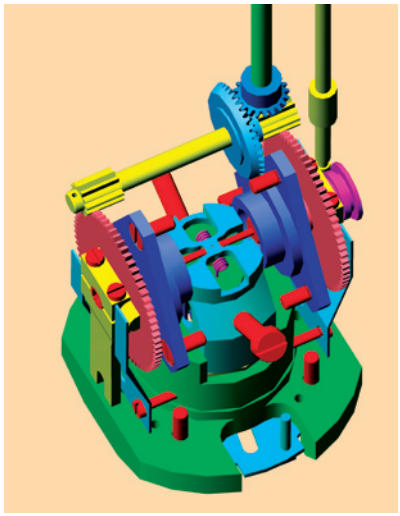

The cover picture shows an open drawing of the cavity, the tuning mechanism and the ENDOR coils of the 275 GHz EPR/ENDOR spectrometer of the group of Edgar Groenen, the recipient of the 2003 Silver Medal for Physics/Materials Science, an acknowledgement of his distinguished career in EPR theory and applications to the field of (bio)molecular physics. In the center the coupling hole to the horizontal TE_{011} mode cavity (diameter, 1.4 mm) and the vertical slits to enable ENDOR operation are visible. Two 2.5 turn RF coils are located perpendicular to the cavity's main axis. One gear train does the symmetrical tuning of the cavity with two plungers and a second gear enables rotation of the sample-tube that is positioned in the centerline of the two tuning plungers. The diameter of the base plate is 58 mm.

The inset shows a wide sweep ENDOR spectrum from a 150 µm crystal of $ZnGeP_2$:Mn^{2+} with resonances of ^{55}Mn and ^{31}P (from Blok H. et al.: J. Magn. Reson. **173**, 49–53 (2005)).

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volume 15 number 2 2005

Eidgenössische Technische Hochschule Zürich
Swiss Federal Institute of Technology Zurich

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

photo of the issue

see page 18

TAKE OUR QUIZ!

As an EPR spectroscopist, does this photo remind you of something related to EPR? Send an e-mail message to the editor with your answer. Deadline September 15, 2005. The most striking answers will be published in one of the next issues of the *EPR newsletter*. The readers of the *EPR newsletter* may vote by e-mail message to the editor to decide who is the prize winner. The prize is the lovely Bruker BioSpin calendar "Science meets Arts".

Editorial

Dear colleagues,

On June 6, 2005 Anatole Abragam and Brebis Bleaney met at a very nice party in St. John's College in Oxford celebrating Brebis Bleaney's 90th birthday. A warm article written by Michael Baker and a photo taken by Nina, the wife of Anatole Abragam, give us the wonderful opportunity of a virtual participation in this jollification (see p. 7). Our belated heartfelt congratulations, Brebis!

Surprise! You will find TWO articles in the *EPR newsletter* Anecdotes column (see p. 9). Anatole Abragam (born 1914) and George Feher (born 1924) take us to the world of their personal remembrances, which give us the feeling of eye-witnessing the bygone times.

I am pretty sure that all of us wrote verses even if it was only once (at least I did it and I know several persons who still do it). However, one needs an ever-vibrating poetic string in the heart to have poetry as a life-long passion. The Another Passion column tells us about poetry in the life of the late Boris Kozyrev, one of the founders of magnetic radiospectroscopy in the former Soviet Union, whose 100th birthday was recently celebrated in Kazan (see p. 4).

The latest news: the Russell Varian Prize 2005 goes out to Nicolaas Bloembergen (born 1920) for his seminal paper about relaxation published in 1947. In 2004, this ▶

★ IES BUSINESS ★ IES BUSINESS ★ IES BUSINESS ★ IES BUSINESS ★

International EPR (ESR) Society Awards 2006

Call for Nominations

Nominations are invited for: Silver Medal (Chemistry) and Silver Medal (Biology/Medicine) and Fellowship of the Society (see extract from by-laws below or visit ieprs.org for full constitution and by-laws).

All nominations must be accompanied by a 100–150 word citation in support of the nomination. No nomination can be considered without a citation. Additional supporting material may be included.

Nominations are to be sent to the President by email in word or pdf format to:
tsvetkov@kinetics.nsc.ru
(before 1 October 2005)
or
lubitz@mpi_muelheim.mpg.de
(after 1 October 2005)

The closing date for nominations for Awards in 2006 is 15th November 2005.

By-laws

A *Silver Medal* shall be awarded for significant contributions to EPR (ESR) Spectroscopy in the area of Instrumentation.

A *Fellowship of the Society* may be conferred on individuals who have made influential and distinguished contributions to the practice of EPR (ESR) Spectroscopy and its welfare over a long period.

prize went out to Erwin Hahn (born 1921) (15/1, p. 3). My attentive reader, you certainly catch my hint: our patriarchs still are news in the life of our magnetic resonance community, nicely and regularly reflected in the columns of the *EPR newsletter*. The life of our patriarchs was, and still is, exciting, they did great research and they met great scientists of the beginning of the 20th century [e.g., see Erwin Hahn's story about his meeting with Albert Einstein (15/1, p. 14)]. Our younger generation has its part of joy: they do their research (time will tell how great it is) and they have the good fortune to meet our great contemporaries.

One of our younger colleagues, Heinz-Jürgen Steinhoff, introduces us to the pros and cons of site-directed spin labeling (see p. 15). Shirley Fairhurst's article about the 38th Annual International Meeting "Advanced Techniques & Applications of EPR" in Bath (see p. 19) provides detailed information about this event and makes us look forward, with great anticipation, to her report about the IES General Meeting during the 28th International Symposium in Denver, July 31 – August 4, 2005.

Meet you in Denver!

Laila Mosina

Awards

The Zavoisky Award 2005

Harold M. Swartz
Dartmouth Medical School,
Hanover, USA

The Russell Varian Prize 2005

Nicolaas Bloembergen
University of Arizona,
Tucson, USA

The Bruker Prize 2006

Yurii D. Tsvetkov
Institute of Chemical Kinetics and Combustion, Novosibirsk,
Russian Federation

for his contribution to the development of pulsed EPR techniques, especially for distance determinations via PELDOR, pulsed electron double resonance, and its applications in free radicals research

Detailed information on this awards will be given in a future issue of the *EPR newsletter*

The Bruker Prize 2005 to Klaus-Peter Dinse

From left to right: Dieter Schmalbein (EPR Division, Bruker BioSpin GmbH), Klaus-Peter Dinse (Chemistry Department, Darmstadt University of Technology), and Shirley Fairhurst (ESR Group Chair).

For details, see this newsletter, p. 19.

The Jeol Young Investigator Prize to Malika Bouterfas

From left to right: Evi Vinck (University of Antwerp), Katerina Pirker (ARC Seibersdorf Research GmbH), John Gibson (Jeol UK) and Malika Bouterfas (École Polytechnique Fédérale de Lausanne).

For details, see this newsletter, p. 19.

Another Passion

The Poetic String in the Heart of EPR Spectroscopist

Boris M. Kozyrev

Recently the Zavoisky Physical-Technical Institute (Kazan, Russian Federation) celebrated the 100th anniversary of Boris M. Kozyrev, one of the founders of magnetic radiospectroscopy in the former Soviet Union, and corresponding member of the Academy of Sciences of the USSR. Many of us are familiar with the book "Electron Paramagnetic Resonance in Compounds of Transition Elements" written by Semen A. Altshuler and Boris M. Kozyrev (2nd ed., Moscow: Nauka 1972) and translated into several languages, including English, German and Polish. The first edition of this book, "Electron Paramgnetic Resonance", was published in 1961 (Moscow: Fizmatgiz). It is interesting to cite here the words of Zavoisky characterizing the first steps of Kozyrev in science at the Chair of Physics of the Kazan State University headed by E. K. Zavoisky: "In 1940 Boris M. Kozyrev studied intensively one of the most interesting and difficult problems of modern magnetochemistry, namely, paramagnetic relaxation. The main subject of his investigations were relaxation phenomena in paramagnets in a strong magnetic field parallel to the oscillating field… It is very important that he managed to measure paramagnetic absorption at room temperature for the first time… Boris M. Kozyrev is a mature, self-dependent researcher, a perfect experimenter, and a well-educated and broad-minded person…"

B. M. Kozyrev worked together with E. K. Zavoisky at the Branch of the Academy of Sciences of the USSR established in Kazan in 1945. In 1947, when E. K. Zavoisky went to Moscow, Boris Kozyrev headed the EPR research carried out at the Kazan Physical-Technical Institute of the Kazan Branch of the Academy of Sciences. In one of his papers, he formulated the problems to solve at that time as follows: to establish the basic regularities of EPR and to expand the range of substances studied. One of the main achievements of Boris Kozyrev was the application of EPR to studying the structure of the solutions of paramagnetic salts.

The readers of the EPR newsletter remember already the name of Boris M. Kozyrev in the articles by Natalia Zavoiskaya (13/1-2, p. 13 and 14/4, p. 6). She warmly mentioned him, who was a friend and colleague of Evgeny K. Zavoisky and a great connoisseur of art. However, his other passion was poetry. As a literary critic, he studied works of Fedor Tyutchev[1] and Innokenty Annensky[2] on a highly professional level. He wrote poetry himself and translated verses of English and French poets into Russian. The examples below give you the possibility to listen to the poetic voice of Boris Kozyrev.

[1] Fedor Tyutchev (1803–1873), Russian poet, see, e.g., eprnl.org/15-2/tyutchev1 and eprnl.org/15-2/tyutchev2

[2] Innokenty Annensky (1856–1909), Russian poet, see, e.g., eprnl.org/15-2/annensky

From "The letters about Tyutchev"

translated by M. Kozyreva

Published in: Historical-philological studies. A collection of articles dedicated to the memory of academician N. I. Konrad, p. 123. Moscow: Nauka 1974.

More than in one third of Tyutchev's verses we meet images connected with his beloved element – the water; nearly in one tenth these images appear to be central, forming the main theme of the poems. It seems there is no known form of moisture in nature that was not noticed by Tyutchev. Besides traditional "terms" as "water, waters, moisture", we meet in his verses such varieties as ocean, sea, deep, lake, river, flood, spring, brook, fountain, downpour, cloudburst, drops of water, drops of tears, etc. The only form one never comes across in his poems – the images of dirty, stagnant water of a marsh. What a variety of the movements of water Tyutchev uses! Waves, billows, ripple; absolutely alive though mythological sea horse with his pale green mane and merry neigh; breakers, tide, foam, sprays; the alive cloud of a fountain, the snow-storm of humid dust under the wheels of a steamer, the golden threads of the spring rain, human tears, uncountable, endless as an autumn rain… It is just impossible to name all the examples of the direct and metaphorical usage of the water images in Tyutchev's poetry…

…the water for him is in the most one of the four elements. It is the purest element, the closest to the divine principle… It is the origin of every living thing. The poet hears in his heart "the mysterious whisper of a brook", the glance of his beloved is like a spring of life in "the deeps of the soul"; the whole poem "O willow tree, why are you leaning over the waters you crown…" appears to be a polysemantic symbol in which the running water is an expression of something supreme, free and victorious as life itself. Even the stars are moist as in the poem "the Summer Evening", they "rise the canopy of heaven / with their moist heads". In the same poem the evening calmness of the nature is formed by the following mythological image: "And the sweet trembling, like a spurt, runs through the veins of nature, as if her hot feet were touched by the spring waters."

All the primordial subindividual life (superior for Tyutchev) is defined as a "life-giving ocean". Even when our poet happens to depict the good element of water in its destroying violence, he blames the fire, not the water, for it: "Is it Hades or some infernal force / which made the fire under the boiling cauldron / and made havoc / and turned upside down the deep."

Not only the natural life, but all the spiritual existence, all that is precious and real in it is close to moisture in Tyutchev's verses.

A poem by Boris Kozyrev

translated by M. Kozyreva

«Духи вечера»

Я помню, в детстве,
После яркого солнца,
После пятнашек, смеха и долгой беготни
с палочкой в руке за взлетающими
кольцами серсо,
Когда сердце стучало сильно и часто,
В сумерках меня вдруг охватывала
неодолимая усталость.
Я застывал на каменном крыльце старого
дома
С глазами, заблудившимися в тишине.
Шло время.
И я начинал видеть – о, я ясно видел! –
На сером небе
Взмахи огромных серых крыльев,
(Ангельских ли, демонских ли, я не знал,
чьих)
Реющих, веющих, колеблющихся
В смутных, таинственных сочетаниях,
То взмывающих ввысь,
То падающих
И словно качающихся у самого моего
лица,
Но все-таки далеких,
Далеких Небесных.

Это случалось в летние вечера,
В деревне,
Чаще всего на старом крыльце нашего
дома.

Духи вечера не стеснялись показывать
ребенку
Свои странные игры.

И я их не позабыл.

(лето 1962)

"The Spirits of Evenings"

I remember
Being a child,
After the bright sun,
After touch-last, laughter and long running
with a stick playing hoopla,
When my heart was beating strong and fast,
In the twilight I suddenly felt an invincible
weariness.
And I stood still on the stone porch of our
old house
With my eyes got lost in the silence.
The time passed.
And I started to notice – oh, how clearly I
saw it! –
On the grey skies
The strokes of gigantic grey wings,
(were they of angels or demons – I didn't
know)
Hovering, trembling, swinging
In strange, mysterious combinations
Going up,
Then falling down
And – it seemed – fluttering close to my face,
But at the same time so remote,
Remote,
Divine.

It used to happen in summer evenings
In the village,
On the stone porch of our old house.

The spirits of the evenings were not shy to
show
Their games to a child.

I have not forgotten them.

(Summer 1962)

Translation by Boris Kozyrev

A. Ch. Swinburne
"Shadow, Silence and the Sea"

All night long, in the world of sleep,
Skies and waters were soft and deep;
Shadow closed them, and silence made
Soundless music of dream and shade:
All above us, the brief night long,
Silence, kindled with sense of light,
All around us, the brief night long,
Silence, laden with sense of song.
Stars and mountains without we knew,
Watched and waited, the soft night through;
Thrilled the touch of the sea's breath near:
All unheard, but alive like sound,
Throbbed the sense of the sea's life round:
Soft as darkness and keen as light.

Ф. Ч. Суинберн
«Тень, молчание и море»

На ночь стихли в пучине сна
Высь небес и вод глубина;
Тень их скрыла, и тишь легла,
С дремой пела беззвучно мгла.
И над нами всю ночь шатром –
Тень, что словно сквозит огнем,
И вокруг нас – во всей ночи –
Тишь, что словно напев звучит.
Звезд и гор хоровод, таясь,
Сторожил этот тихий час;
И незримый, был ласки полн
Близкий трепет вздыхавших волн;
И неслышный, но словно звук
Жизни моря дрожал вокруг,
Здесь и там и вокруг звуча,
Мягче тьмы и острей луча.

Translation by Boris Kozyrev

W. Blake
From "Auguries of Innocence"

To see a World in a Grain of Sand
And a Heaven in the Wild Flower,
Hold Infinity in the palm of your hand
And Eternity in an hour.

У. Блейк
Из «Пророчеств Невинности»

Чтобы в песчинке Мир увидеть
И небо в Цветке долины,
Сумей Бесконечность в ладонь собрать
И Вечность – в час единый.

90th Birthday of Brebis Bleaney

Brebis Bleaney was 90 on 6th June 2005. He is regarded, together with Zavoisky, as one of the founding fathers of EPR, who independently invented this spectroscopic technique in the post-war years in Oxford. For Bleaney, based in Oxford, EPR was a natural extension of his wartime work on microwave generation and circuitry followed by microwave spectroscopy of gases and his earlier thesis work on the properties of matter at very low temperatures. It was also encouraged by the ambience of the emerging field of low temperature physics of magnetic salts in Oxford under Simon and Kurti. The first paper in 1948 was on EPR in chrome alum and was co-authored with R. P. Penrose, who later discovered hyperfine structure in EPR, which was to become an important factor in much of Bleaney's work. The second paper in 1949 on copper Tutton salts was co-authored with both Penrose and Betty Plumpton, who was subsequently to become Bleaney's wife.

Two features probably allowed the extremely rapid exploitation of EPR of transition metal ions in paramagnetic salts by Bleaney's group: (a) the use of dilution by a diamagnetic isomorphous salt, so enormously reducing the line width and obtaining high resolution, and (b) the use of low temperatures to lengthen the spin-lattice relaxation time, so overcoming lifetime broadening. There followed a prolific output of papers with a succession of research students (of which I was eventually privileged to be one). The progress was also enormously stimulated by collaboration with a succession of theoretical physicists in Oxford: Maurice Pryce, Anatole Abragam, Ken Stevens, Roger Elliott and Ray Orbach. Concurrently with the development of EPR, Bleaney continued to collaborate with the low temperature group at Oxford on nuclear cooling and nuclear orientation.

Bleaney first came to Oxford as an undergraduate at St. John's College in 1934, and except for sabbatical leave and short-term visiting appointments, he has been in Oxford ever since. He completed his D.Phil. thesis in 1939 and became a Lecturer and Tutorial Fellow at St. John's College in 1947 where he remained until 1957.

The take-off of his very productive period of research had been delayed by the war, and this period coincided with his ten year tenure of the Tutorial Fellowship at St. John's College. Considering its brevity, I feel extremely privileged to have been his pupil during this period for three years as an undergraduate and then four years as his graduate student. If personal anecdotes are permitted, I first heard of Brebis in the summer of 1947, when I was awarded a State Scholarship and was directed by the post-war Labour Government to go to Oxford, as part of an attempt to distribute talent around British universities. This meant having to choose a college, and my reaction was to ask advice from the only person to have gone to Oxford from my backwoods school. He told me of a bright and lively new tutor at St. John's College (Brebis), so that was the seed of our association. From 1948, I went to Brebis once per week for an hour of one-to-one tutorial conversation. When we had exhausted my problems with the week's work, he would tell me about the results of his recent EPR measurements; for example, the hyperfine structure of copper and manganese, or the fine structure of chromium and iron. Of course, I understood these ideas only partially, but Brebis conveyed to me the excitement of research, which probably influenced my choice to undertake graduate research with him. He was an extremely stimulating, hands-on supervisor, in the laboratory everyday, tweaking the equipment for better performance, sometimes analysing data as it came off the production line; so he led his troops from the front. I remember that for our first publication together, on the nuclear spin of vanadium 50, he was drafting the letter before I had finished collecting the data! It was also very stimulating that the group met twice a day, mid-morning and mid-afternoon for coffee or tea, because these were times to exchange ideas and experiences, and to meet a succession of important visitors to Brebis.

Reflections about this period, now over half a century ago, emphasise how things have changed. The first tasks which Brebis gave me to do when I became a graduate student were to build an oscilloscope, a klystron 2 kV power supply and a magnet power supply. All of the equipment was home made, except for the klystron and the magnet; fortunately Brebis had retained a wizard in the workshop to make the microwave components and a talented Research Assistant who designed and made some of the more sophisticated electronics. The signal was either detected by a galvanometer and plotted out point by point, or displayed on an oscilloscope using 50 Hz modulation. Measurements at very low temperatures were made using liquid hydrogen and glass dewars. In one respect Brebis's hands-on approach could be hazardous. One collected liquid hydrogen for the day's experiments in an open necked glass transfer dewar of a few litres capacity, in the top of which a plug of cotton wool prevented condensation and room temperature radiation, but allowed the gently boiling hydrogen to escape. Brebis used very occasionally to smoke a cigarette, and I remember occasions when he sat at my desk pouring over some new data, with ash falling from his lit cigarette down onto the "smoking" dewar vessel parked out of the way on the floor beside the desk!

Brebis's deep appreciation of hyperfine structure from many points of view (it was he who proposed the method of nuclear alignment by turning an anisotropic crystal through 90° to exploit the anisotropy of the hyperfine interaction), together with his knowledge of electron resonance and relaxation, led him to talk to me during 1954/5 about the possibility of exciting the nuclear transitions in a paramagnetic ion, which would be much narrower than the electronic transitions. If I had listened to him, rather than chasing other hares, we might

have beaten George Feher to the invention of ENDOR (his first publication was in 1956).

After the untimely death in 1956 of Sir Francis Simon, who had just become the Dr. Lee's Professor of Experimental Philosophy at Oxford, and the head of the Clarendon Laboratory, Brebis was elected to succeed him in 1957. This post involved a great deal of administration, and so took Brebis away from the spectrometer. He of course kept in close contact with the research of his acolytes, giving them enormous support and stimulation. He resigned his professorship in 1977 to resume personal research, first as a Warren Fellow of the Royal Society (77–80) and then as a Leverhulme Emeritus Fellow (80–82), still working in the Clarendon Laboratory, and starting a new venture with a new research group in enhanced NMR. Even after this, formally becoming "Emeritus" at the age of 67, Brebis continued to come into the lab daily until quite recently.

Betty and Brebis Bleaney and Anatole Abragam at the party in St. John's College in Oxford celebrating Brebis Bleaney's 90th birthday. Taken by Nina Abragam

Brebis has been associated with numerous articles published in the scientific journals. Even in the period while he was Dr. Lee's Professor, when his administrative duties did not allow him the time to run his own research laboratory; he produced many mainly single-author publications ranging over many new ideas as he found time to think about them. After this period his publications became focused on enhanced NMR together with the members of his new research group; for a year or two his publication rate was so prolific that it was more than several of his junior colleagues in condensed matter physics put together. Brebis has also co-authored two major books. The first, entitled *Electricity and Magnetism*, came out of his teaching at St. John's College: it was written with his wife and forms a basic textbook for both electromagnetism and solid state physics. The second written with Anatole Abragam, a long-standing collaborator also recently entered on his tenth decade, is entitled *EPR of transition ions*: it arises out of their work together as experimentalist and theorist on magnetic resonance of paramagnetic solids.

During his illustrious career Brebis has received too many prizes and honours to list here: they are listed in *Who's Who* and *International Who's Who*. Perhaps the most significant to readers of the Newsletter are his election to a Fellowship of the Royal Society at an early age, in 1950, in recognition of the pioneering work on EPR, and the award of the Zavoisky prize in 1992 (he was also made a Fellow of the International EPR Society in 1995).

Apart from physics, Brebis's main interests are in his family (an earlier edition of *Who's Who* listed his interests as playing with son and daughter), listening to music (he also played the violin in chamber music with friends and with his wife at the piano) and playing tennis. He still loves going to concerts, in spite of deteriorating hearing, and he played a good set of tennis (and rode his bicycle to the lab and to college) until very recently.

Brebis's penetrating and agile scientific mind meant that at seminars, lectures and conferences, he was often the first on his feet at the end of a talk with a searching question, always asked with humility and kindness, never aimed at making an impression. These human traits emphasise the qualities of the good tutor and supervisor that he was, always approachable, always having time, always interested in the welfare of his pupils and students, but always with a light touch: not abrasive, authoritarian or demanding. He was always keen to introduce his students to, and draw then into discussion with, the great and famous. So his personality engendered both great respect and great affection for him. Hence, it was not surprising that a meeting to celebrate his 80th year attracted many old students and collaborators from around the world. Now, even most of Brebis's scientific children, like me, have retired; but he must be pleased that those remaining and his scientific grandchildren continue to mine gold in the immensely productive field he staked out in the late '40s.

Michael Baker

How I Discovered EPR

Anatole Abragam

I realize that the title is misleading. I mean "discovered", the way people would say "I discovered sex at thirty three". (This is actually how old I was when I discovered it (I mean EPR)).

After the defeat of Germany in 1945 I had found a job at the newly founded French Atomic Energy Commission, doing calculations on the first French Atomic reactor with the code name Zoe. We formed a team of four theoreticians: Jules Horowitz, Claude Bloch, Michel Trocheris and myself. The last two are still alive at the time of writing (this author has just turned ninety). After our work was completed and Zoe diverged satisfactorily, all four of us were eager to take part in the new developing (and fashionable) Quantum Theory of Fields. I got from an organization called "The British Council" a Fellowship to spend one or two years in Birmingham, working under Professor Rudolf Peierls on the aforesaid subject. Two things worried me though. The first was that the domain of "Quantum Field Theory" was terribly overcrowded. Practically all the young French theorists I knew wanted to work on it. But there was something else. During a previous stay I took a good look at the good city of Birmingham and what I saw did not enchant me. The provocative ugliness of its architecture, the tasteless dreariness of its streets, the guttural idiom spoken by the natives, which had little in common with what I had heard on the BBC, the unspeakable food (at least for a Frenchman, even one whom four years of German occupation had not cosseted much) all this led me to wonder whether the game was worth the candle. On an impulse I went to the offices of the British Council and told them that I was not very keen anymore on going to Birmingham and could I go somewhere else. "Where would you like to go?" I have not quite decided yet. Where could I go? They opened their files for me. "There is not much left. Oh yes, there is a new Professor of Theoretical Physics in Oxford, Maurice Pryce, who has room for another student. We don't quite know what he is doing but it is probably far removed from your interests in Field Theory. He collaborates with some experimentalists, in the Clarendon Laboratory who work in the field of Magnetic Resonance, if you know what it is." I did not know and I did not care. I had heard the magic word Oxford. I had been to Oxford once before on my way to Birmingham and the idea that one could stay and work in Oxford for a long time was for me like working in Florence and good enough for me. "Oh yes, I have been interested in Magnetic Resonance for some time. Could you put my name down for Oxford." "We shall put you down as a candidate but you must get Professor's Pryce agreement as soon as possible." Pryce accepted me and I spent two happy years in Oxford. Among the few things I did there was the invention of what is known as the "Abragam and Pryce spin Hamiltonian" and the quantitative explanation of the hyperfine structure of the salts of the iron group through the introduction of what is known in atomic spectroscopy as "configuration interaction".

In Oxford thesis examinations there are two examiners, one of them an Oxford Professor (it was Bleaney) and an external one who comes usually from Cambridge. How was I to know that the external examiner would be Professor Hartree? He wanted to know why I had written in my thesis: "Hartree wave-functions are notoriously unreliable for the calculation of hyperfine structures". I explained hastily that Hartree wave functions had been discovered by a variational method to give the best possible values for the energy levels and one could hardly expect them to perform as well for finer details like hyperfine structures. This seemed to satisfy him. Bleaney asked me a treacherous question: what were the colours of the various salts whose energy levels and hyperfine structures I calculated so glibly? I had not the slightest idea and told him so. That did not prevent them from giving me my D.Phil. (doctorate of Philosophy). For the reader who might wish more details on my work in EPR I can warmly recommend a book by Bleaney and myself (900 pages in the Oxford Press edition and about as much in the French and in the Russian translation). What I might say about Oxford could (and actually did) fill half a book, my autobiography in French, Russian or English.

Nina and Anatole Abragam in Oxford

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Reminiscences of Encounters with Some of the Pioneers of Magnetic Resonance

George Feher

UC San Diego, La Jolla, CA

In a recent issue of the *EPR newsletter*, the editor, Laila Mosina presented a very interesting collection of articles about the pioneers of magnetic resonance. It triggered memories of encounters that I had with several of them that maybe of interest to readers of the newsletter.

Let me start with **Wolfgang Pauli** whom I met on a couple of occasions. In a strict sense, Pauli cannot be considered a "Magnetic Resonator" but his involvement and deep understanding of the consequences of the electron spin (the thing we are flipping) is appreciated by all, albeit not enough according to him, as the narrative will show.

In the spring of 1957 Pauli visited Bell Labs at Murray Hill, NJ where I had developed the ENDOR technique a few months earlier. The management of Bell Labs brought Pauli to my lab and asked me to explain this new technique to him. It was an extremely uncomfortable feeling to explain anything to the great Pauli. His usual demeanor did not help. He was sitting silently in my lab with closed eyes, seemingly asleep, nodding his head in a periodic motion. I was warned that I should watch closely the frequency of his head motion; when the frequencies changed I had probably said something wrong or disturbing. Sure enough, when I mentioned the word hyperfine interaction not only did his nods accelerate, he opened his eyes and said; "Hyperfine interactions? They are still around?" upon which he reverted to the sleeping position and left after half an hour without saying another word.

Pauli's remark haunted me for years until I had dinner with Sam Goudsmit to whom I relayed the encounter. Goudsmit thought he understood Pauli's reaction and told me the following story: Pauli did not speak to him for 30 years and avoided him at conferences. Finally Goudsmit could no longer take it, cornered Pauli and asked what he had against him. Pauli accused him of having stolen from him the idea of the hyperfine interaction and the existence of the electron spin. (Goudsmit and Uhlenbeck are credited with postulating the electron spin). Pauli claimed he had given a seminar in Berlin in the 1920's that Goudsmit had attended in which he – Pauli, had postulated the electron spin and the hyperfine interaction. Goudsmit had no recollection of this seminar or of anybody else postulating an electron spin. At any rate this story seems to provide a plausible explanation of Pauli's reaction when he heard me say the words "hyperfine interaction".

Pauli's visit at Bell Labs ended on an interesting note. At the end of the day everybody connected with his visit gathered in the conference room to say goodbye to Pauli who, as usual, was nodding his head with closed eyes. The director of the Labs, who no doubt was expecting a pat on the shoulder, asked: "Professor Pauli what do you think of our research?" The frequency of Pauli's nods increased and after what seemed an interminably long time, he uttered his verdict: "Harmless, harmless." It was quite a sight to see the faces of the management.

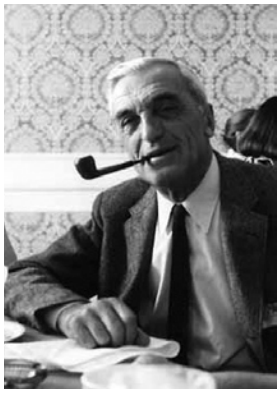
The second time I met Pauli was in the Fall of 1957 at the International Conference on Nuclear Structure held at the Weizmann Institute in Rehovot, Israel. At that time we had used ENDOR to determine the spin and magnetic moment of P^{32} and the hyperfine structure anomaly of Sb^{121}/Sb^{123}, both appropriate topics for the conference. At one of the luncheons I was seated at the table with Pauli and C. S. Wu (who was the leader of one of the groups that had recently shown that parity is not conserved). They showed a particular interest on the subject of the kibbutz movement, and so I told them that I would be happy to show them Merchavia, the kibbutz I belonged to in 1941–42.

To understand the rest of the story I have to digress and describe briefly the kibbutz. A kibbutz was a communal, agricultural settlement (many now have industry and other capitalistic undertones) made up of a group of idealists and ideologues who shared everything, from worldly goods (of which there were few) to the soul and emotional life. It was a community devoid of privacy and individuality. It was not easy to take. For some of us, ideology clashed with the realities of daily life and so in the Fall of 1942 I reached the difficult decision to leave the kibbutz. I was, of course, aware that at the time I was considered a traitor to the cause. However, I thought that now in 1957, 15 years later I will not only be forgiven but some may even feel proud that one of their ex-members is participating in this important international conference. The conference on nuclear structure enjoyed great publicity in Israel because of the rumors that Israel had developed an atomic bomb. Anybody I met who heard of my participation in the conference immediately asked: "Nu, do we have it?" Well, I was gravely mistaken in my evaluation of the situation at the kibbutz.

Pauli and Wu took me up on my offer and one afternoon we drove to kibbutz Merchavia. To my great embarrassment most of the people gave me a cold shoulder, some of them even turned around and refused to speak to me. They clearly had not forgiven me for leaving in 1942. Pauli asked me if I had murdered somebody in the kibbutz. It was typical of him to address a problem head-on with little regard for the feelings of others. Madame Wu was more sympathetic. In any event, it was a most painful experience for me.

I will conclude this section on Pauli with an episode showing that even the greatest are not infallible. In the fall of 1956, T. D. Lee sent a preprint of his article with C. N. Yang in which he suggested that parity is not conserved in weak interactions. Pauli replied that he was convinced that they were wrong and that parity is conserved and he was willing to bet his reputation on it. While the letter was en route, the non conservation of parity was proven experimentally. Everybody was anxiously waiting to see how Pauli would ▶

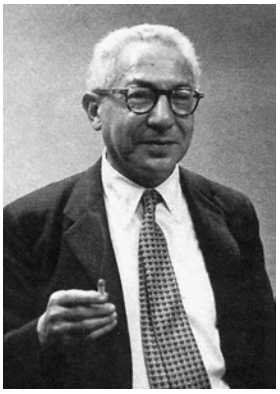
react to it. A letter finally arrived with Pauli acknowledging that he had been wrong but adding that we must admit how clever he was in betting his reputation of which he had plenty to spare and not betting money, of which he had little.



In 1958 while working at Bell Labs I received a phone call from **Felix Bloch** asking me whether I would be interested in visiting Stanford University to interview for a faculty position. I had been greatly impressed by Bloch's beautiful and farsighted 1946 paper on his discovery and description of NMR and immediately accepted the invitation. At Stanford I was struck by everybody's seriousness and formality (as compared to Bell Labs). A couple of feeble attempts on my part to lighten the atmosphere fell completely flat. For instance the chairman, Leonard Schiff (from whose textbook I had learnt quantum mechanics) suggested in the interview that I should teach Physics 143. I didn't have the foggiest idea what Physics 143 was. But instead of asking politely about its contents, I jokingly said that I would rather teach Physics 137. "But we don't have a course by that number," replied Schiff. Later, when Bloch mentioned that the position carried a salary of the order of $10,000/year, I replied that salaries and electronic *g*-values should be quoted to more significant places. He was not amused by the remark. Looking back at the visit now, almost 50 years later, I don't really understand why I behaved in this "smart-alecky" way. Perhaps the heaviness bordering on pomposity irritated me (this may be an explanation but hardly an excuse).

I mentioned my visit and misgivings to my friend and colleague Erwin Hahn at Berkeley, who had been Bloch's post-doc in the early days of NMR. He agreed that Bloch is a formal person but much less so now than he was before Hahn came to him as a post-doc, implying that he took credit for making Bloch less formal. Knowing Hahn's informality and humor I can believe it. He gave me as an example the following story: Bloch, as you know, was mainly a theorist. One day when he encountered difficulties in his theoretical work, he went to Hahn and asked him to give him something to do in the lab to distract his mind. So Hahn gave him a piece of waveguide to file. After a while Bloch came to Hahn and said that a pump nearby was making strange noises. Hahn took in the scene and immediately understood: All the filings had fallen into the pump. So Hahn told Bloch to listen to what the pump says. "What does it say?" asked Bloch. Hahn replied, "It says Bloch, bloch, bloch…" Last week I asked Hahn if I remembered this episode correctly. He replied yes I did and added that he used to amuse guests at Bloch's house parties by playing tunes bonking his head. This is precisely what you show in your photograph of the Vol 14 #3 issue of the Newsletter. Hahn also taught Bloch to master this art form.

Going back to the interview at Stanford. In spite of what I perceived to have been a poor performance at the interview, I was offered a tenured position. It was a tempting offer but after much thought I did not accept it. One of the main reasons was that I still had plans to return to Israel and to contribute to the budding scientific developments there. When I told Bloch about my decision, he was initially incredulous that I preferred Bell Labs over Stanford, but after I explained to him my personal reasons, he understood and told me the following story: In the 1940's his conscience started to bother him. As a Jew he should contribute to the building of a nation in Palestine instead of sitting comfortably in the USA. These feelings became stronger with time until he could no longer sleep at night. At this point his wife intervened: "Felix, we can't go on like this. Go and see your friend Albert in Princeton; he is supposed to be a wise man, discuss your problems with him." So Bloch went to see Einstein and told him of his problem. "But Felix, I have solved this problem a long time ago. I am first a scientist and second a Jew", said the great man. Bloch heaved a sigh of relief: "Oh, thank you Albert" and from then on the problem never bothered him again. Well, unfortunately Einstein's solution didn't work for me and I never really made peace with not living in Israel. Two weeks after our conversation Bloch called me up and asked whether I had changed my mind. When I said no, I hadn't, he asked: "In spite of the story that I told you?" I told him laughingly, "Yes but you heard it first hand from the great man and I heard it only second hand." I should add that Bloch never held my decision against me and we stayed on friendly terms till his death in 1983.



A year later, in 1959, I met **Isidor I. Rabi** at Columbia University. The physics department was thinking of starting a program in Solid-State Physics (now called Condensed Matter Physics). Furthermore, Charlie Townes, the co-inventor of the maser and laser was leaving and somebody had to take over his students. Having worked both on Masers and in Solid-State Physics, I was approached to apply for the position. I. I. Rabi, the strong man at Columbia, was vehemently opposed to a program in Solid-State Physics. So, when I gave a colloquium, at Columbia, he interrupted and heckled me throughout, presumably in the hope of seeing me fall apart, as I heard a few of my predecessors did. But I wasn't rattled or concerned. There wasn't much at stake; I had a good job with the Bell Telephone Co. It also helped that throughout the colloquium I kept before my eyes a scene from my graduate student days in Berkeley. There, a famous and difficult person, E. O. Lawrence, the inventor of the cyclotron, once entered a lab and saw a young man with his feet on his desk munching a sandwich. To Lawrence, such behavior in the Sacred Halls of Science was intolerable. He quietly closed the door to give the man time to shape up but when he opened the door again, the man hadn't changed his position or activity. Lawrence got furious, red in the face and started to shout at him. At this point, we, graduate students, emerged to witness the spectacle. After a while the chairman, R. T. Birge, was able to calm Lawrence down. Upon which the young man calmly said: "Sir, I don't know who you are, I work for the Telephone Company."

About 25 years later Rabi and his wife visited La Jolla. Norman Kroll (who, with ▶

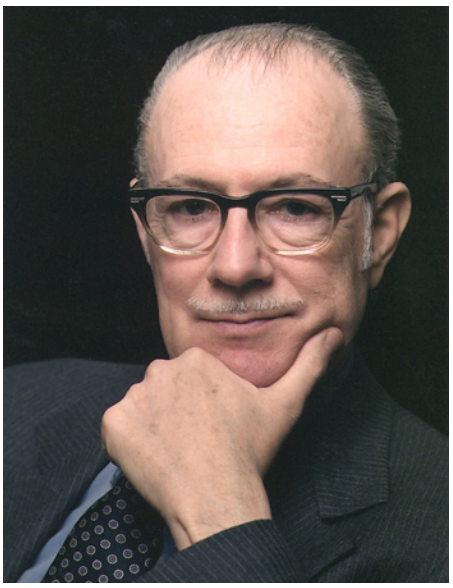
R. Karplus, calculated the *g*-value of 2.0023 for free electrons) invited the Rabi's and my wife and me for dinner. I thought that after 25 years surely it was safe to reminisce and so between the soup and main dish I told them the telephone story that helped me to endure his questioning during the colloquium. Rabi wasn't amused at all, but his wife was.

One puzzle remains. Why did Rabi dislike so intensely solid-state physics? After talking to some old timers about it, the following story emerged, which may provide a clue. I. I. Rabi and M. W. Zemansky (later of textbook fame) were graduate students in the nineteen twenties at Columbia University working for their PhD in – you won't believe it – Solid-State Physics. They were measuring the temperature dependence of the magnetic susceptibility of various materials. As expected they found the $1/T$ Curie susceptibility; except in alkali metals, where they found the susceptibility to be temperature-independent.

They were greatly puzzled by the temperature independence and decided to consult W. Pauli on the subject.* Zemansky traveled to Hamburg to see Pauli. Being a modest and shy man he knocked on the door and, when Pauli opened it, asked politely: "Excuse me, Professor Pauli, am I disturbing you?" Pauli simply said: "Yes" and closed the door in his face. Zemansky returned to the U.S. without accomplishing his mission. Some time elapsed before Rabi could collect enough money to travel to Hamburg himself and presented the problem to Pauli. Reportedly, Pauli said "Dummkopf, of course the susceptibility is temperature-independent. It is only the fraction of electrons at the fuzzy surface of the Fermi sphere that contribute to the paramagnetism. This fraction is T/T_F which when multiplied by $1/T$ results in the observed temperature independence." This phenomenon is now called Pauli paramagnetism. This must have upset Rabi no end; a phenomenon that he discovered ended up bearing the name of another person. Whether this incident made Rabi dislike solid-state physics and made him switch to the then new and exciting field of atomic beam spectroscopy is, of course, pure speculation. But if it is true, Rabi surely owes Pauli a great debt.

* In trying to verify this story, I was unsuccessful in finding any published reference to it. It may, therefore, be apocryphal. But then: "Si non è vero, è ben trovato" ("If not true, it is a good story").

George Edward Pake*



George Edward Pake, a distinguished condensed matter physicist and past president of the American Physical Society (APS), died on 4 March 2004 in Tucson, Arizona, from multiple system failure. He also was founder of the Xerox Palo Alto Research Center (now the Palo Alto Research Center, or PARC) and of the Institute for Research on Learning, both in Palo Alto, California.

George was born on 1 April 1924 in Jeffersonville, Ohio. He received a BS and an MS in physics from the Carnegie Institute of Technology (now Carnegie Mellon University) in 1945 and his PhD in physics from Harvard University in 1948. George was the second thesis student of Edward Purcell. He undertook a study of the proton resonance of water of hydration in a single crystal of gypsum. To his surprise, he found that the absorption line was a doublet rather than the narrow single lines of liquids.

It was not long before George showed that the doublet arose because each proton in the water molecule experienced not only the magnetic field of the laboratory magnet, but a magnetic field arising from the other hydrogen atom in the molecule. Because the proton has a spin-½, its orientations are quantized into two directions, up or down, in the external laboratory magnetic field. Thus the field of the neighbor either aided or opposed the laboratory field. George further showed that he could use nuclear magnetic resonance to measure the relative positions of the two protons (hence the spatial orientation of the water molecule) and the distance between the protons. That work attracted the interest of Herbert Gutowsky, a chemistry graduate student at Harvard, to learn NMR. The two men collaborated to characterize NMR absorption line shapes of singles, pairs, and triples of protons and to show that, at some temperatures, NMR revealed the presence of molecular rotations in the solid phase. Their research launched Gutowsky's career in NMR.

Between 1948 and 1956, George served in the physics department of Washington University in St. Louis, Missouri. In 1956, he was lured to Stanford University to replace Nobel laureate Willis Lamb, who had just left for Oxford University. During his time at Washington and Stanford, George wrote several important books, including *Notes on the Quantum Theory of Angular Momentum* (Stanford U. Press, 1953) with coauthor Eugene Feenberg; *Paramagnetic Resonance* (W. A. Benjamin, 1962); and, with Thomas Estle, *The Physical Principles of Electron Paramagnetic Resonance* (W. A. Benjamin, 1973). He began service as executive vice chancellor and provost, and professor of physics at Washington University in 1967. In 1969, the university named him Edward Mallinckrodt Distinguished University Professor of Physics.

From 1965 to 1969, George was a member of the US president's scientific advisory committee under Presidents Lyndon B. Johnson and Richard M. Nixon. He was active on panels of the National Academy of Sciences and NSF. As the chairman of the physics survey committee, which produced the so-called Pake Report, he directed the first of the NAS studies on the status of fields of science. In 1976, George was elected vice president and, in 1977, became president of APS. In 1983, APS established the George E. Pake Prize, an award that recognizes and encourages outstanding work by physicists who have combined accomplishments in original re-

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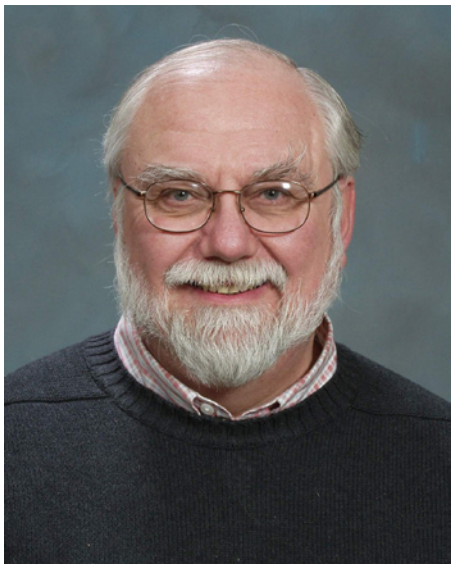
Robert Clarkson (1943–2004)



We are saddened to record the death of Robert Clarkson at the age of 61. He was a "man of all seasons", with very significant accomplishments and interest in many fields. His most recent research interests ranged from particles of coals and chars to in vivo EPR. Bob Clarkson was especially interested and successful in using a variety of powerful physical tools to obtain relevant information for understanding how magnetic resonance probes would function in vivo. His vast areas of interest and expertise were reflected by an extensive and varied bibliography and a wide range of collaborators. The importance of his work was recognized by his peers by his success in obtaining competitive grants. He also was a person who enjoyed life and enriched it for others. His was a superb singer, whose bass voice was lovely to hear. He played the guitar with great enjoyment for himself and his companions. He was always a wonderful companion before and after the meeting sessions, as well as during the scientific sessions. Bob Clarkson also was an exemplary family man, devoted to his children and his lovely wife Jean. He always spoke fondly and with great pride about them. We will greatly miss his excellent science and his excellent companionship.

Harold Swartz

search and leadership in managing research or development in industry.

George is perhaps best known for leading PARC from its inception in 1970 until 1978 and for overseeing Xerox corporate research from 1978 to 1986. Under his leadership, PARC gave birth to such innovations as laser printing, Ethernet, the graphical user interface, client–server architecture, object-oriented programming, bitmapped displays, and many other ideas that define modern computing. Under George's leadership, PARC became a major architect of the information age.

After retiring from Xerox in 1986, George founded the Institute for Research on Learning. In 1987, President Ronald Reagan awarded him the National Medal of Science.

George was a great person as well as a great scientist. In the words of one PARC technologist, Gary Starkweather, "Getting to know George Pake was one of the great experiences of my life. … PARC, as well as I, would not have been successful were it not for George's capable leadership and guidance. I always admired his friendly and gentlemanly manner and will always remember him fondly." George will be remembered as much for who he was as for what he did.

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Pro & Contra

Site-Directed Spin Labeling

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Introduction

The understanding of protein, DNA or RNA function and interaction requires the knowledge of the respective molecular structures and their conformational dynamics. In particular, crystallization of membrane proteins or protein complexes is still challenging and conformations found in the crystallized systems may not be biologically active. Therefore, the development of complementary spectroscopic techniques is necessary. Since the pioneering work of Wayne L. Hubbell in the late 80s site-directed spin labeling (SDSL) has emerged as a powerful method for studying the structure and conformational dynamics of proteins and nucleic acids under conditions relevant to function [1]. Spin label side chains are introduced at selected sites via cysteine substitution mutagenesis followed by modification of the sulfhydryl groups with a specific paramagnetic nitroxide reagent [2]. The continuous-wave (CW) EPR spectrum yields information about the nitroxide side chain mobility (for a recent review see, e.g., [3]), its solvent accessibility [4], the polarity of its immediate environment or the distance between two bound nitroxides. EPR data analysis of a series of spin-labeled variants of a given protein allows definition of elements of secondary structure, including their solvent exposure, to characterize protein topography and to determine orientations of individual segments of the protein. In doubly spin labeled samples the combination of CW and pulse EPR spectroscopy enables determination of inter- and intra-molecular distances in the range from 0.5 to 8 nm. A complete analysis thus leads to a model of the protein structure with a spatial resolution at the level of the backbone fold. One of the most powerful properties of SDSL is its sensitivity to molecular dynamics: protein equilibrium fluctuations and conformational changes of functional relevance can be followed on a wide time scale ranging from picoseconds to microseconds by combining CW and saturation transfer EPR spectroscopy. Time-resolved CW detection enables following conformational changes with submillisecond time resolution [5, 6].

Technical Aspects of SDSL

Cysteine residues may be modified with a variety of nitroxides to yield a spin label side chain. However, the methanethiosulfonate spin label (1-oxyl-2,2,5,5-tetramethylpyrroline-3-methyl)methanethiosulfonate (MTSSL) [2] is most often used in SDSL studies due to its sulfhydryl-specificity and its small molecular volume, similar to the phenylalanine or tryptophane side chains. In addition, the unique dynamic properties of this spin label side chain facilitate detailed structural information determined from the shape of its EPR spectrum. A general scheme of the spin labeling procedure proven to yield good labeling efficiency is as follows: After cysteine-substitution mutagenesis the purified protein is usually stored in the presence of DTT in order to prevent oxidation of the cysteines. Before spin labeling the protein solution has to be dialyzed against 100 mM sodium phosphate buffer, pH 7.0 to dilute the concentration of DTT. The protein (concentration adjusted to, e.g., 20 μM) is then incubated with 100 μM spin label at 4°C for 12 h. Higher concentration of the MTS spin label during the labeling reaction may lead to formation of unwanted biradicals. The unbound spin label is removed by gel filtration using Sephadex G-25 mini columns or dialyses. The spin labeled protein is concentrated to between 20 and 100 μM using centricons and filled into EPR quartz capillaries. Membrane proteins may require reconstitution into liposomes for proper folding and function. At X-band the use of loop-gap or dielectric resonators provide the necessary sensitivity to yield CW EPR spectra with good signal-to-noise ratio using less than 5 μl of sample within scan times of between 1 and 30 min. However, even concentrations of 10 μM may hardly be reached with reconstituted membrane protein complexes. A certain lipid-to-protein ratio may be required to provide the necessary lipid environment for protein function. So, further improvement of the sensitivity of EPR spectrometers is necessary. The spin-labeled cysteine-to-protein ratio can be finally determined by double integration of the EPR spectra followed by comparison with standard solutions of MTSSL and determination of the protein concentration. For inter-spin distance measurements this ratio has to be close to one. The following step is one of the most important: the function of the modified protein has to be checked with an independent method to rule out incorrect folding. Since the spin label side chain and the native amino acid side chain may be oriented differently, structural analysis should always be based on a set of different spin-labeled protein variants.

Specific Advantages and Limitations

During the last decade a large number of very successful applications of SDSL have been reported. Main goals have been the investigation of protein, DNA or RNA structures and conformational changes during function (see below). The method has its limits with proteins or protein complexes containing accessible native cysteines. In case these cysteines are of no functional importance they may be replaced by, e.g., serines or alanines, otherwise those systems cannot be studied with the technique outlined above. However, this problem can be overcome by recently developed techniques for the incorporation of unnatural amino acids into proteins. Tremendous progress has been achieved concerning the synthesis of unnatural spin-labeled amino acids [7] and techniques which facilitate the incorporation of unnatural amino acids into proteins using synthesized amino acylated t-RNA [8]. Whereas this technique is limited by the complicated synthesis and low yield, the chemical synthesis of peptides in combination with recombinant techniques provides the means to incorporate unnatural amino acids into proteins with reasonable effort [9]. The native chemical ligation of unprotected peptides has become a viable method for the preparation of a wide variety of biologically active proteins. Furthermore, the expressed protein ligation technique has enabled the production of polypeptide-

thioesters by recombinant means for use in native chemical ligation. It has now become possible to introduce an unnatural amino acid not only in the C- or N-terminal part of the protein but also in its center [10]. Taking advantage of tailor-made nitroxide amino acids (a) spin labeling is specific also in the presence of more than one SH group; (b) the spin label side chain can be designed to mimic and replace native amino acid side chains, like phenylalanine, histidine, tyrosine or tryptophane; (c) spin label amino acids with reduced residual motion can be designed to provide defined orientations with respect to the backbone. This will simplify discrimination between protein backbone dynamics and its modulation upon conformational changes and residual side chain dynamics. (e) The reduced residual motion and the defined orientation of the nitroxide side chain with respect to the backbone will improve measurements of inter-spin distances and relative orientations of the nitroxide side chains (molecular axes orientations). Using sets of doubly spin-labeled engineered proteins the determination of structural details and conformational changes with very high resolution will be achievable.

Applications

The relation between the nitroxide side chain mobility and the protein secondary and tertiary structure has been extensively reviewed [11]. The term "mobility" is used here in a general sense and includes effects due to the motional rate, amplitude and anisotropy of the nitroxide reorientation. Weak interaction between the nitroxide and neighboring side chain or backbone atoms as found for helix surface sites or loop regions results in a high degree of mobility. In this case the apparent hyperfine splitting and the line width are small. In turn, strong interaction of the nitroxide group with adjacent side chain or backbone atoms restricts its reorientational motion. Hence, tertiary contact or buried sites are characterized by an increased apparent hyperfine splitting and line width. Depending on the length and flexibility of the linker between the nitroxide and the protein backbone and possible hydrogen bonding of the nitroxide to the protein the flexibility of the protein backbone itself contributes to the overall nitroxide mobility. Numerous examples have shown that the analysis of the nitroxide dynamics of a series of spin labeled protein variants uncovers the secondary structure and provides important information about tertiary interaction [12].

The collision frequency of the nitroxide side chains with freely diffusing paramagnetic probe molecules provides additional structural information. The collision frequency of such a probe depends on the product of its translational diffusion coefficient and its local concentration. Molecular oxygen and water soluble paramagnetic Ni(II) complexes or chromium oxalate have been frequently used and are ideally suited because of their sizes and solubility properties [1, 4]. In a water/membrane system these molecules are partitioned between the water and the hydrophobic phase according to their polarity. Polar metal complexes preferentially partition into the aqueous phase, whereas apolar oxygen exhibits a maximum value of the product of concentration and diffusion coefficient in the centre of the membrane bilayer. The determination of the collision frequency of nitroxide side chains with these paramagnetic reagents in solution allows identification of the side chain orientations with respect to the protein-water or protein-lipid interface. CW power saturation has been shown to provide an easy and reliable means for the quantification of the collision frequencies [4].

The application of high-field EPR techniques with Lamor frequencies exceeding 90 GHz has considerably enhanced the Zeeman resolution of rigid-limit spectra of disordered spin labeled samples. The principal g-tensor components and their modulation due to solute-solvent interactions can be determined with high accuracy. The structural information is contained in the variation of the polarity in the spin label micro-environment. A polar environment shifts the tensor component g_{xx} of a nitroxide to smaller values whereas the hyperfine tensor component A_{zz} is increased. Thus, both tensor components can be regarded as polarity indexes. In addition, a plot of g_{xx} vs. A_{zz} allows discrimination between protic and aprotic environment due to the different sensitivities of these tensor components towards the influence of hydrogen bonding to the NO group [13]. In a sequence of a regular secondary structure with anisotropic solvation, the local water density in the vicinity of the spin labeled site and hence the polarity index values are a periodic function of sequence number, similar to the behavior of the nitroxide mobility or its accessibility for water soluble paramagnetic ions.

Distance measurements by magnetic resonance methods are based on the distance dependence of the dipole-dipole coupling between two spins. The static dipolar interaction leads to considerable broadening or dipolar splitting of the CW EPR spectrum if the inter-spin distance is less than 2 nm. In order to prevent motional averaging of the dipolar interaction samples have to be measured in solutions of high viscosity [14] or in the frozen state below 200 K [15, 16]. For unique orientations of the nitroxides relative to each other as found for spin labels introduced at buried sites a rigorous solution of the spin Hamiltonian can be obtained. Spectra simulations and fittings to experimental EPR data determined at 9 GHz, 35 GHz and 95 GHz, yield the distance between the nitroxides and the Euler angles describing their relative orientation and that of the inter-spin vector relative to the magnetic field [17]. For surface sites, the nitroxides usually adopt a statistical distribution of distances and relative orientations within a restricted distance and orientation range. In a frozen solution or powder sample an isotropic orientation distribution of the distance vectors between the interacting spins can be assumed. The corresponding dipolar spectrum of such a macroscopically disordered sample may be approximated by a Pake pattern. The range of inter-spin distances arising from a distribution of protein conformations or orientations of the spin label side chains may be accounted for by a Gaussian distribution [16]. The final dipolar broadened EPR spectrum is then calculated from the nitroxide powder spectrum by convolution with a superposition of Pake patterns. Values of inter-

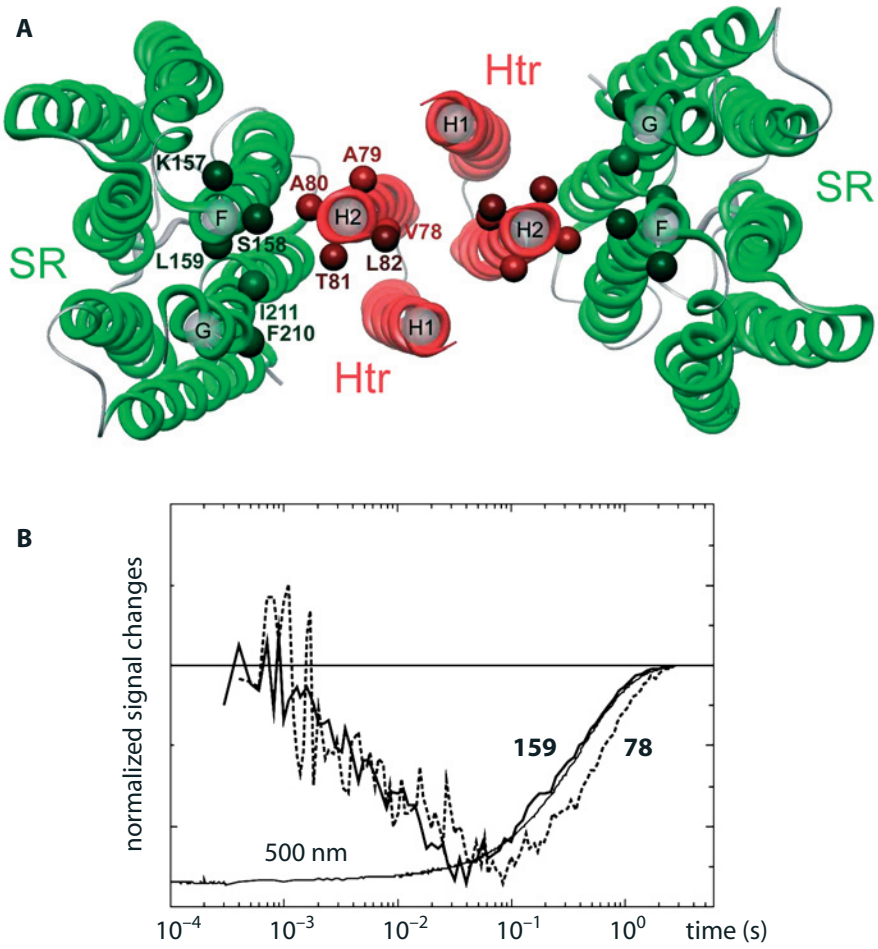

Fig. 1. A Transmembrane portion of the 2:2-complex of the sensory rhodopsin pSRII with its transducer pHtrII (view from the cytoplasm). This protein complex serves as a light sensor controlling the flagella motor of archaea. Inter-spin distances determined for 26 combinations of site-directed spin-labeled (positions marked as spheres) pSRII and pHtrII variants enabled modeling of the location and relative orientations of the transmembrane helices of the transducer, H1 and H2, and of helices F and G of the receptor [36]. The topology of the complex agrees with the later determined crystal structure [37]. **B** Optical absorption changes of the retinal (550 nm) and EPR transients (noisy lines, determined at fixed B field) of the receptor – transducer complex pSRII – pHtr with a spin label attached to position 78 (broken line) and to position 159 (continuous line) after light activation ($T = 293$ K). The transient increase of the mobility of the spin label side chain at position 159 indicates a transient movement of the cytoplasmic moiety of helix F of pSRII. This leads to a conformational change of the transducer as revealed by the transient EPR signal changes of the spin labels bound to position 78 and 78′ in H2 and H2′ (modified, [37]).

spin distances are finally determined by fitting of the calculated EPR spectra to the experimental spectra [16, 18]. Alternatively, the dipolar broadened experimental spectrum of the doubly spin-labeled protein may be deconvoluted with the superposition spectra of the corresponding singly labeled proteins. The inter-spin distance is extracted from the shape of the resulting Pake pattern [15]. The lower limit for reliable distance determination using the above methods is given by the increasing influence of exchange interaction for inter-spin distances less than 0.8 nm due to partial overlap of the nitrogen π-orbitals of the two interacting nitroxides. In this distance regime, half field transitions, which are not sensitive to exchange interaction, have been successfully applied [19]. A detailed discussion of the influence of exchange coupling on the above approximations can be found in a recent review [20]. Since the spin labeling efficiency may be less than 100% a variable fraction of singly spin-labeled protein has to be accounted for in the above approaches. The spectra simulation and deconvolution methods for inter-spin distance determination have been successfully validated by comparison of EPR data with crystallographic data of spin-labeled insulin [16], lysozyme [14], bacteriorhodopsin [18] or with synthesized spin-labeled peptides of known secondary structure [15]. The line width of the spectra is a steep function of the inter-spin distance. Hence, empirical or semi-empirical parameters as spectral amplitude ratios or spectral second moment values are often sufficient to answer structural questions [18, 21]. Besides nitroxide-nitroxide interaction also metal ion-nitroxide interactions in metallo-proteins or engineered copper-ion-binding sites allow estimation of intra-molecular distances [22, 23].

For inter-spin distances exceeding 2 nm the line broadening due to dipolar interaction is much less than the influence of other homogeneous and inhomogeneous contributions, hence CW EPR is not sensitive for dipolar interaction. However, the application of pulse double resonance EPR methods increases the accessible distance range up to 8 nm. These techniques have been recently reviewed by Gunnar Jeschke [24].

Since the first quantitative analysis of dipolar splitting observed in a spin-labeled protein [25] the method of inter-spin distance determination by CW EPR spectroscopy has been successfully applied to reveal domain structures and conformational changes of membrane proteins, including rhodopsin [26, 27], lac permease [28], the KcsA potassium channel [29] and the prokaryotic voltage-dependent K^+ channel (KvAP) [31]. In addition, changes in dipolar interaction result in large spectral changes, making it straightforward to monitor conformational changes. Recent notable successes include the evaluation of the gating mechanism of the mechanosensitive channel MscL [30]. Changes of the protein secondary structure, protein tertiary fold or domain movements can be followed with up to 0.1 ms resolution with conventional CW EPR instrumentation and detection schemes. Interesting examples are the detection of rigid-body helix motion in both rhodopsin and bacteriorhodopsin [18, 32–35], and conformational changes during signal transfer from sensory rhodopsin pSRII to the transducer pHtrII [36] (see also Fig. 1).

Conclusion

The combination of SDSL with multifrequency CW and pulse EPR spectroscopy allows elucidation of macromolecular structures and functional mechanisms involving conformational changes like transport, signaling, gating or gene regulation. Application to membrane proteins, which represent one of the most significant frontiers in the field of structural biology and molecular biophysics, the developments of new methods of spin labeling using artificial amino acids and the application of multifrequency and pulse EPR spectroscopy with magnetic fields ranging from 0.1 to 13 T will give this technique a bright future.

photo of the issue

TAKEN BY ARTHUR SCHWEIGER

A hello from EPR spectroscopy

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A Joint Conference of the 11th **In Vivo EPR Spectroscopy and Imaging** and the 8th **International EPR Spin Trapping**

The Ohio State University College of Medicine and the Dorothy M. Davis Heart and Lung Research Institute, Columbus, Ohio, USA
September 4–8, 2005
heartlung.osu.edu/epr
epr2005@medctr.osu.edu
(previously announced in *newsletter* 14/3)

Spin Chemistry Meeting 2005 9th International Symposium on Spin and Magnetic Field Effects in Chemistry and Related Phenomena

St. John's College, Oxford, UK
September 11–17, 2005
physchem.ox.ac.uk/~scm2005
(previously announced in *newsletter* 14/4)

4th International Conference on Nitroxide Radicals: Synthesis, Properties and Implications of Nitroxides **SPIN-2005**

Akademgorodok, Novosibirsk, Russian Federation
September 20–24, 2005
www.nioch.nsc.ru/conf2005
spin2005@nioch.nsc.ru
(previously announced in *newsletter* 15/1)

High Field / High Frequency ESR/EPR Workshop

The Center for Advanced ESR Technology (ACERT) at Cornell University, Ithaca, USA
November 4–6, 2005
jkt27@cornell.edu
(previously announced in *newsletter* 15/1)

The 38th Annual International Meeting **Advanced Techniques & Applications of EPR**

University of Bath, Bath, UK
March 20–24, 2005

At this meeting plenary lectures were presented by: John Davies (University of Bath) who described some optical methods for the detection of ESR in semiconductors. Yuri Tsvetkov (Institute of Chemical Kinetics, Novosibirsk) described the application of Pulsed Double Electron-Electron Resonance (PELDOR). Edgar Groenen (Huygens Laboratory, Leiden) described recent advances using pulsed EPR and ENDOR at 95 and 275 GHz. Motiji Ikeya (Osaka University) described studies of interdisciplinary ESR from Earth to planetary science: dating and radiation dosimetry. Zbigniew Sojka (Jagellonian University, Poland) described a computational approach combining a hybrid genetic algorithm for optimization of EPR spectra with DFT calculations of magnetic parameters. Joshua Telser (Roosevelt University) took us on a journey across the first row transition metals with high field/frequency EPR. Jack Peisach (Albert Einstein College of Medicine) brought us up to date with his long interest in EPR spectroscopy of transition metal proteins, from his early studies on copper and iron, up to the present with his on-going work on prion proteins. Heinz-Jürgen Steinhoff (University of Osnabruck) showed how multifrequency EPR spectroscopy and site-directed spin labelling reveal the structure and conformational dynamics of membrane bound proteins complexes. Sabine Van Doorslaer (University of Antwerp) described The Echo of Life – Pulse EPR analyses of paramagnetic metalloproteins and related macrocyclic metal complexes, concentrating on globin proteins.

The 2005 Bruker Lecture was presented by Professor Klaus-Peter Dinse of Darmstadt University of Technology who posed the question: EPR – an interesting topic? In an elegant and amusing lecture we were ably persuaded that the subject has not only an interesting past and present but that a bright future awaits us.

Neil Connelly (Bristol University) introduced the session dedicated to the memory of the late Phil Rieger (Brown University, Providence USA). Phil's interest in EPR spectra from systems with non-coincident *g* and *A* tensors and his long-standing ties with the group were elaborated. His wife and collaborator Anne Rieger was able to join us (see also EPR newsletter 14/3, p.17 (2005)).

The JEOL prize for the best oral presentation by a young scientist saw Malika Bouterfas (École Polytechnique Fédérale de Lausanne) received the JEOL Prize Medal for her talk entitled: Longitudinally detected ESR (LODESR) using miniaturized Hall sensors. Joint runners-up were Katerina Pirker (ARC Seibersdorf Research GmbH) and Evi Vinck (University of Antwerp). All three student talks were of a very high calibre but sadly there was only one medal to be won. All three received cheques for £100 from JEOL.

This year there were 33 posters and as usual one was selected to win the poster prize with the traditional bottle of whisky going to Stephan Stoll (ETH Zurich) for his poster on – A Suppression Effect in ESEEM spectra of multinuclear spin systems.

Full abstracts of the plenary lectures, short talks and posters are on the website: www.esr-group.org.uk.

Shirley Fairhurst

Market Place

POSITIONS

Postdoctoral Positions Available at Davis Heart and Lung Research Institute, The Ohio State University

A position is available for a scientist with experience in magnetic resonance instrumentation development and application. The candidate should have experience in EPR/MR hardware or software development and applications to chemical or biological systems. Salary commensurate with experience. Please reference PA06 in your application.

A position is available for a scientist with experience in cardiac NMR spectroscopy or imaging research to perform isolated heart and in vivo studies of alterations in myocardial energetics and metabolism in the post-ischemic heart. Salary commensurate with experience. Please reference PA07 in your application.

The Ohio State University is an equal opportunity/affirmative action employer. Qualified women, minorities, Vietnam era veterans and individuals with disabilities are encouraged to apply.

Send CV to: Dr. Jay Zweier, 473 West 12th Avenue, Room 110, Columbus, Ohio 43210 or zweier-1@medctr.osu.edu.

Full Time Research Associate Position at The University of Chicago

The University of Chicago's Department of Radiation and Cellular Oncology is seeking a qualified applicant for full time Research Associate position. The primary activity of a Research Associate is academic research in associate with faculty member or team. An opening currently exists in a research team studying and developing of novel Electron Paramagnetic Resonance based images of aspects of the physiologic milieu, particularly quantitative images of oxygen concentrations in the tissues of a living animal. This is part of the Center for EPR Imaging in Vivo Physiology, a federally funded multi-institutional research resource. The person selected for this position will work with a consortium of medical physicists, chemists, engineers and biologists. The work will involve developing

- instrumentation for novel EPR spectral spatial imaging strategies using both continuous wave and pulsed acquisition techniques at radiofrequency (~250 MHz)
- new magnet technologies for rapid and flexible projection acquisition
- novel projection acquisition strategies to increase the image resolution and sensitivity per unit time
- novel analysis strategies to optimize the extraction of information per unit time
- novel resonant structures for enhancement of EPR imaging with continuous wave and pulsed instrumentation
- standards and novel phantoms to test both spatial and physiologic parameter resolution of images

and coordination and the obtaining of animal MRI studies to correlate anatomic images with the physiologic images obtained with EPR.

Qualified applicants must possess:

- A doctorate degree in physics, physical chemistry, or related field
- Expertise in EPR instrumentation and theory
- Expertise in programming and, in particular, in development of acquisition and analysis algorithms
- Expertise in computer control of data acquisition
- The ability to supervise and manage up to ten researchers involved in a wide variety of EPR experiments is crucial
- Willingness to work with animal subjects
- The ability to maintain a calm but critical work environment

Desirable attributes for applicants include:

- Experience with tomographic image acquisition and analysis
- Experience with MatLab programming environment
- Experience with National Instruments Labview
- Experience in the obtaining of MRI images

Applicants should fax (773)702-5940 or e-mail h-halpern@uchicago.edu a letter and Curriculum Vitae to Dr. Halpern, Department of Radiation and Cellular Oncology, The University of Chicago. The University of Chicago is an Affirmative Action/Equal Opportunity Employer.

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Please send enquires to:
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We have some excess EPR accessories and supplies that might be of use to other labs. For example, we have a lot of chart paper, pens and ink for order recorders, and some spare parts and accessories such as VT Dewars for older spectrometers. If you need something for an older-style Varian or Bruker spectrometer, ask us – we might be able to help. Most items are available for shipping costs.

Gareth R. Eaton geaton@du.edu

Available: Isotope-Containing Spin Probes

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For SWAP: Fast Digitizers EG&G 9825-200

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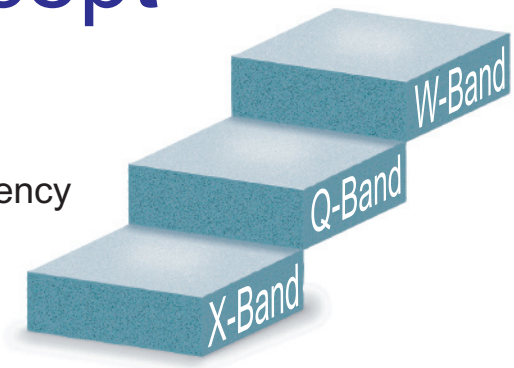
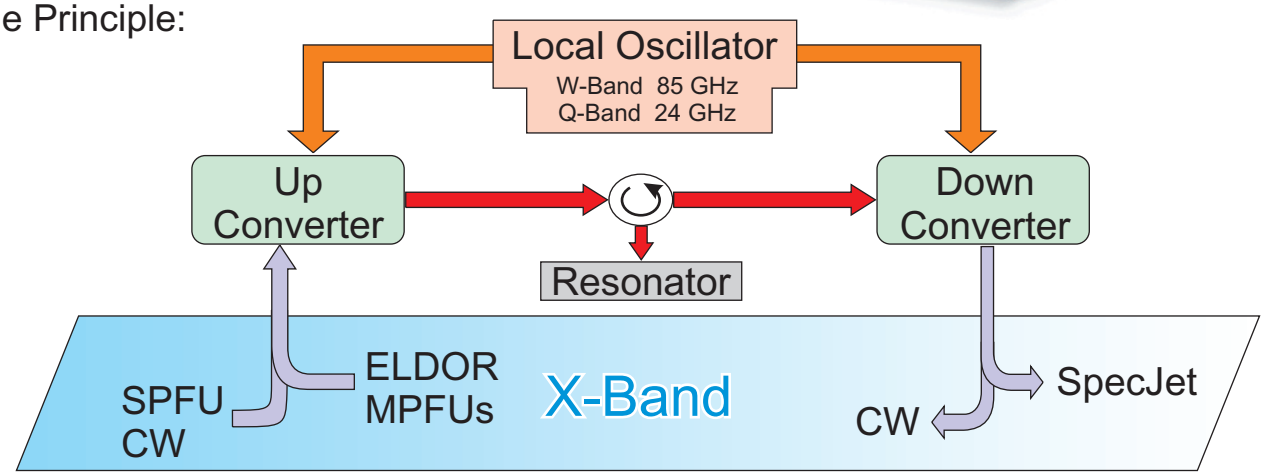


- The Principle:

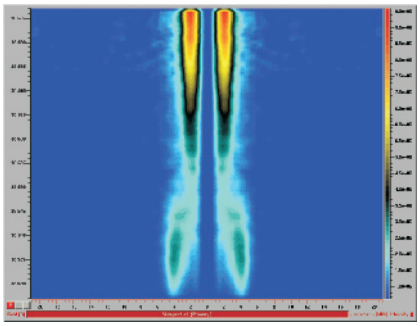


Up Conversion: X-band excitation (CW/Pulse) combined with LO results in Operational frequencies

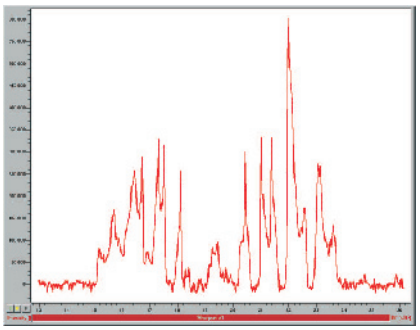
Down Conversion: Operational frequency combined with LO results in X-band frequencies for detection

- The Implementation:

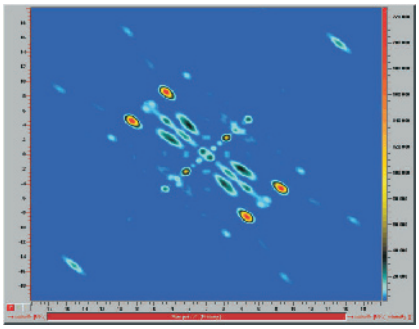
W-Band Field Sweep 4p-DEER



Q-Band Sn-ENDOR



X-Band ^{14}N-HYSCORE



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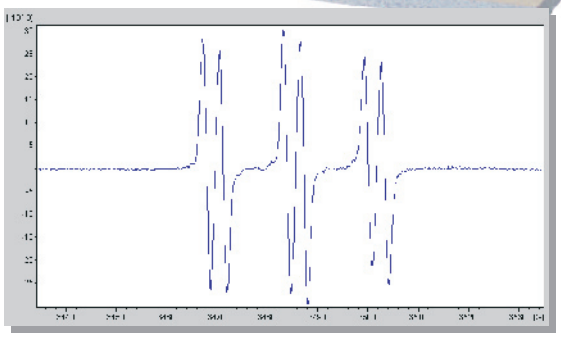
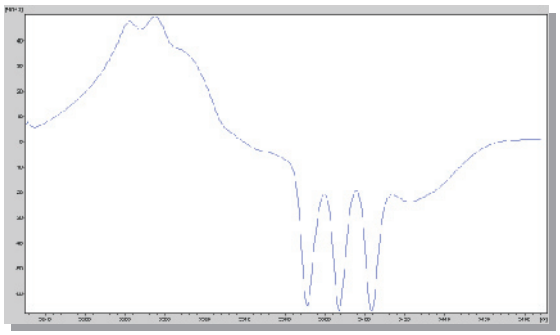
- *Spin-Label EPR for Pharmacokinetics, Membrane Research*
- *Nitroxide (NO) Detection at 77 K*
- *Antioxidant Status & Activity*



Spin Trapping of ROS



***Spectrum of NO bound to Haemoglobin.** (Recorded at 77 K using the Finger Dewar Accessory)*

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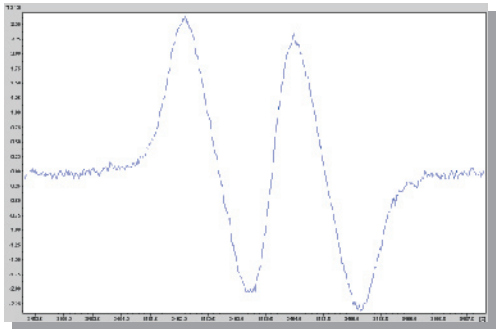
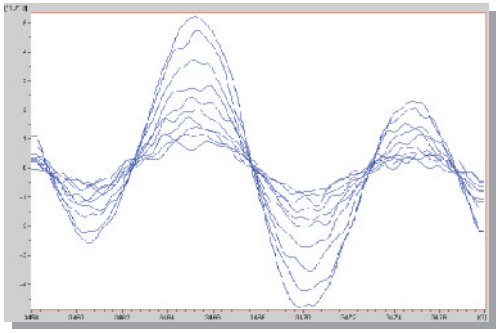


Spectrum of the Ascorbic Acid Radical In Blood Plasma



Antioxidant Activity Assessed with the DPPH-Assay

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