

Editorial Review

Gerd Buntkowsky*, Konstantin Ivanov* and Hans-Martin Vieth* From Free Radicals and Spin-Chemistry Over Spin-Dynamics and Hyperpolarization to Biology and Materials Science

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This special issue of ZPC is dedicated to the 80th anniversary of Kev Salikhov, a prominent scientist who has made seminal contributions to spin chemistry, chemical kinetics, spin hyperpolarization and magnetic resonance. Altogether there are 41 scientific papers; for this reason, the guest editors have decided to publish three subsequent issues. Each issue is focused on a particular field, which is closely related to research interests of Salikhov or was even seeded by his work. As the following papers clearly demonstrate, the initial seed of Salikhov fell on a very fruitful soil and has grown to a huge and impressive scientific tree with branches ranging from fundamental science to applications in biochemistry and materials science.

The papers in the first issue are mainly dealing with fundamental aspects of spin chemistry and studies of free radicals and radical pairs. The investigation of the magnetic field effects on chemical reaction rates has been one of the major fields of research by Salikhov. Accordingly, there are several contributions in this special issue, which address this important topic from various angles of observation.

In the paper by Doktorov [1] “On relations between singlet and triplet recombination yields for singlet and triplet precursors” general relations for radical pair recombination yields have been obtained, which are valid in arbitrary external magnetic fields and for arbitrary reactivity. Interestingly, the yields of singlet

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and triple state recombination are inter-related: relations between these yields are established and proven in the most general situation.

The paper by Lukzen et al. [2] gives a Liouville-equation quantum-dynamic treatment of the magnetic field dependence of the charge recombination reaction dynamics in radical pairs, which considers both coherent and incoherent processes. With this sophisticated approach the authors are able to faithfully reproduce the experimentally observed field dependencies.

The paper by Purtoev [3] “Reaction operators for radical pairs. The exactly solvable model” is dealing with computation of the reaction operator, which describes spin-selective recombination of radical pairs. In this work the consideration is extended to treat reversible processes by using the results of an exactly solvable model. The results of previously proposed models can also be deduced from this treatment in the limiting case of irreversible recombination.

The paper by Melnikov et al. [4] “Estimation of the Fraction of Spin-Correlated Radical Ion Pairs in Irradiated Alkanes Using Magnetosensitive Recombination Luminescence from Exciplexes Generated upon Recombination of a Probe Pair” outlines a study of magnetic field-dependent luminescence of exciplexes generated by radical pair recombination in non-polar media. Large magnetic field effects on luminescence reaching 20% allow one to estimate the contribution of spin-correlated radical pairs to the overall recombination.

In the paper by Nasibulov et al. [5] “Theoretical description of pulsed RYDMR: refocusing zero-quantum and single quantum coherences” methods are described for generating spin echos in radical pairs, which can be detected by monitoring the radical pair recombination yield. These methods are using refocusing of zero-quantum or single-quantum spin coherences.

Properties of spin polarization are used to characterize donor–acceptor complexes in the report “A Transient EPR Study of Electron Transfer in Tetrathiafulvalene-Aluminum(III) Porphyrin-Anthraquinone Supramolecular Triads” by Poddutoori et al. [6]. Analysis of light induced electron transfer (ET) in a donor-porphyrin-acceptor dyad reveals a two-step ET process with negligible singlet-triplet mixing and shows how the observed charge separation can be stabilized.

Borovkov et al. [7] in their work “Solvent radical anions in irradiated aliphatic ketones and esters as observed using time-resolved magnetic field effects in the recombination fluorescence” make use of the quantum beats in recombination luminescence to detect elusive radical pairs and to determine their magnetic parameters. By using this method, it is shown that radical anions of some liquid ketones and esters can be stabilized by additions of alcohols.

The paper by Shelaev et al. [8] “Effect of dehydrated trehalose matrix on the kinetics of forward electron transfer reactions in Photosystem I” reports a time-resolved EPR and optical spectroscopy study of forward electron transfer

in Photosystem I. Effects of a trehalose matrix are studied in detail: whereas the primary electron transfer step remains unaffected by the matrix, a strong reduction of the secondary transfer step is found.

The contribution by Zill et al. [9] studies the photochemically induced nuclear polarization of the photosynthetic reaction center (RC) of the diatom *Cyclotella meneghiniana* by ^{13}C MAS NMR. Since the CIDNP-effect is only active in the direct neighborhood of the RC the authors are able to resolve and assign fourteen of the carbon NMR resonances of the chlorophyll A of the RC, which would be completely impossible without CIDNP, owing on the one hand to the low sensitivity and on the other hand to the background of the cell of the diatom.

EPR and the hyperpolarization method of CIDNP are applied in the work by Markova et al. [10] entitled “Light-stimulated generation of free radicals by quinone-chelators”. The effect of chelators on the formation of free radicals in photo-induced oxidation serving as a model for light-stimulated redox activity in anticancer drugs is examined. A strong increase in the reaction yield of reactive species upon chelation is found with the prospect to be utilized for anti-cancer therapy.

The interactions between solute and ionic liquids (ILs) is the topic of Ivanov et al. [11] in their study “Influence of C_2 -Methylation of Imidazolium Based Ionic Liquids on the Photoinduced Spin Dynamics of Dissolved ZnTPP Studied by Time-Resolved EPR”. Differences in spectral shape and relaxation indicate strong distortions of the photoexcited porphyrin molecule when solved in methylated ILs in contrast to their protonated analogs and other media and show the sensitivity of time-resolved EPR to such solvent-solute interactions.

The work by Zinovieva et al. [12] “ESR study of electron states in Ge/Si heterostructures with nanodisc shaped quantum dots” is dealing with applications of EPR methods to electron states in Ge/Si heterostructures in the dark conditions and generated by light irradiation. It is demonstrated that EPR signals are originating from the electrons localized due to the strain at the top edges and base edges of the nanodiscs, and from the electrons localized in the Si layer above (or under) the central parts of the nanodiscs.

In their investigation “Novel anthrathiophene-based small molecules as donor material for organic photovoltaics: synthesis and light-induced EPR study”, Shatrova et al. [13] focus on new materials with improved properties for photovoltaic application. Synthesis and analysis by various techniques including optical absorption and EPR are described demonstrating that their material indeed shows efficient light-induced electron transfer and is a good candidate for use in future solar cells.

An approach to obtain and characterize one-dimensional periodic spin chains for use in quantum computation is described in the contribution by Yamamoto et al. [14] “Structural determination of a DNA oligomer for a molecular spin

qubit Lloyd model of quantum computers”. In a combination of pulsed electron-electron double resonance (PELDOR) and molecular mechanics calculations DNA duplexes spin-labeled with two pairwise radicals are studied as potential electron spin qubit systems.

The second issue is dedicated to studies of various aspects of spin dynamics, notably, in non-thermally polarized spin systems. Owing to the strong interest of Kev Salikhov in electron-spin pair dynamics and electron-nuclear spin dynamics the majority of these papers is related to these fields and their application, e.g. the transfer of thermally or photo-induced electron-spin polarization to nuclei.

Morozova et al. [15] in their paper entitled “Intramolecular Electron Transfer from Tryptophan to Guanosyl Radicals in a Linked System as a Model of DNA Repair” applied the CIDNP method to study intramolecular electron transfer in a model compound, which mimics a DNA repair system. Quantitative assessment of the CIDNP time dependence enabled determining the electron transfer rates, revealing a high efficiency of chemical repair of the guanosyl radical by means of electron transfer from the amino acids.

Atsarkin and Dzheparov [16] in their paper “Spin dynamics and establishing of internal quasi-equilibrium in dilute paramagnetic solids” briefly review the theoretical description and experimental verification of the two-temperature quasi-equilibrium concept in EPR of dilute paramagnetic solids. This discussion leads to a reconsideration of experimental data on spectral diffusion in dipolar broadened EPR lines. The discussion marks clearly the applicability range of the two-temperature quasi-equilibrium model in electron spin systems with random distribution of paramagnetic centers.

The paper by Schöps et al. [17] “Multi-frequency Pulsed Overhauser DNP at 1.2 Tesla” is dealing with developing pulsed DNP techniques. Specifically, a fast arbitrary waveform generator is used to excite all three EPR lines of a ^{14}N -nitroxide with the aim to increase the DNP-derived NMR enhancement. This method is advantageous in DNP experiments with radicals having a multi-line EPR spectrum.

The contribution “Triplet Charge Recombination in Heliobacterial Reaction Centers Does Not Produce a Spin-Polarized EPR Spectrum” by Ferlez et al. [18] reports on the spin polarization patterns and optically detected magnetic resonance (ODMR) spectra of Heliobacteria. It is shown by transient EPR and ODMR that for triplet states with $D=3E$ exact cancelation of the polarization occurs when the triplet state is formed by charge recombination, a situation that is found in heliobacterial reaction centers.

Khramtsova et al. [19] in their paper “Photoinduced Electron Transfer in Dyads with (R)-/(S)-Naproxen and (S)-Tryptophan” describe a CIDNP and optical spectroscopy study of electron transfer in a dyad system containing naproxen and tryptophan amino acid. A thorough analysis of the experimental data

reveals that the intramolecular electron transfer between the naproxen and tryptophan fragments is efficient and the electron transfer rates demonstrate stereo-differentiation.

The study by Stadler et al. [20] entitled “Toward Matching Optically and NMR Active Volumes for Optimizing the Observation of Photo-Induced Reactions by NMR” reports on a setup that can be utilized to enhance the scope of NMR-based experiments for the investigation of photo-induced reactions. By adjusting the photo-active volume to the NMR-detectable portion of the sample more realistic kinetic information, background suppression and reduction of thermal and diffusional effects are achieved.

Bowman et al. [21] in their work “Pulsed EPR Signals from Triplets” present a theoretical study of the spin dynamics of triplet states in pulsed EPR experiments. The dependence of the spin echo and stimulated echo signals on the microwave phase and flip angle of the EPR pulses is studied in detail for selective as well as non-selective EPR excitation. An emphasis is put on spin polarized triplet states.

The paper by Matveeva et al. [22] “Double Electron-Electron Resonance Study of Biradical Conformations with Monte Carlo Search for the Pair Distance Distribution” presents a new method for quantitative analysis of PELDOR data, aimed at extracting the distance distribution for a pair of EPR spin probes. The method makes use of approximation of the solution by a set of Gaussian functions; their parameters are determined by a Monte Carlo search. The method is applied to new flexible nitroxide biradicals based on trolox being a synthetic analog of α -tocopherol.

Three manuscripts are devoted to the usage of parahydrogen for the enhancement of NMR signals:

In his work “SABRE hyperpolarization of dipyriddy stabilized Ir-complex at high, low and ultralow magnetic fields”, Pravdivtsev [23] demonstrates a new approach to perform SABRE experiments, namely SABRE using stabilized Ir-complexes. With this method it is feasible to generate spin hyperpolarization in a continuous way and in particular to reach ^{15}N polarization of up to 3%. Different methods for optimizing the polarization level are developed.

The paper by Skovpin et al. [24] presents the development of a novel heterogeneous iridium catalyst, which is created by immobilization of Vaska’s Complex on silica and its application in gas-phase hydrogenation with parahydrogen.

The paper by Gutmann et al. [25] presents the development of a novel heterogeneous iridium nanocatalyst in the form of a silica-polymer core-shell nanoparticle and its application in hydrogenations with parahydrogen. It is shown that the catalyst is very stable, exhibits no detectable leaching and has good catalytic activity.

The remaining three papers in this issue describe methodological improvements of NMR methodology:

Bochkin et al. [26] in their paper “Relaxation of Multiple Quantum NMR Coherences in Quasi-One-Dimensional Spin Systems” present a study of the evolution of multiple-quantum coherences generated in a one-dimensional chain of ^{19}F spins. Dipolar relaxation of the coherences is studied experimentally and modeled theoretically. It is demonstrated that the coherence of the zeroth order is not subject to dipolar relaxation in the ‘ZZ model’, in contrast to the second order coherence.

The paper by Carnevale et al. [27] “Exciting wide NMR spectra of static solid samples with weak radiofrequency fields” outlines an application of the DANTE method to exciting a broad NMR line, namely, the Pake pattern of protons of water molecules trapped in a static powdered sample of barium chlorate monohydrate. It is shown that this method allows one to obtain undistorted NMR lineshapes; the selectivity of NMR excitation provides single-crystal like spectra, which are ‘slices’ of the original Pake pattern.

Tseytlin [28] suggests in his contribution “Concept of Phase Cycling in Pulsed Magnetic Resonance Using Sinusoidal Magnetic Field Modulation” a method in pulsed EPR employing modulation of the external field that is expected to improve suppression of unwanted signals such as cavity ring-down overlapping with the echo free induction decay.

The focus of the third issue is on applications of magnetic resonance, both EPR and NMR, to timely problems in chemistry, material science and biology.

Taguchi et al. [29] in the paper “Basic and Combination Cross-features in X- and Q-band HYSCORE of the ^{15}N Labeled Bacteriochlorophyll a Cation Radical” present a study of the bacteriochlorophyll cation radical. To probe hyperfine couplings in the ^{15}N -labeled compound the HYSCORE method is used. The nitrogen hyperfine interaction tensors were resolved in the squared-frequency representation of the HYSCORE spectra; simulations enable precise determination of the hyperfine coupling constants.

Cini et al. [30] study magnetic iron oxide nanoparticles (MNPs) of the spinel type mineralized inside the internal cavity of a mini ferritin-type protein by electron magnetic resonance (EMR) spectroscopy as a function of temperature and orientation.

The paper by Krupskaya et al. [31] “Magnetic resonance study of the spin-1/2 quantum magnet $\text{BaAg}_2\text{Cu}[\text{VO}_4]_2$ ” is dealing with high-field/high-frequency EPR and NMR studies of Cu-spin chains in the complex oxide $\text{BaAg}_2\text{Cu}[\text{VO}_4]_2$. Low-temperature EPR data indicates that short-range magnetic correlations are present in the system. The study reveals the superposition of ferromagnetic as well as antiferromagnetic spin chains in the studied material.

Kuzhelev et al. [32] in their paper “Triarylmethyl Radicals: EPR Study of ^{13}C Hyperfine Coupling Constants” performed an EPR study of a series of

triarylmethyl radicals and determined for the first time their ^{13}C hyperfine couplings. Additionally, it is demonstrated that PELDOR experiments with observation performed at the ^{13}C satellite line are useful for distance measurements at room temperature and at natural carbon abundance.

Jackalin and Dvinskikh [33] employ natural abundance ^{15}N NMR to study thermotropic liquid crystals with cyano-groups. They demonstrate that high resolution ^{15}N NMR spectra can be measured with high sensitivity in mesophases, employing a standard solution NMR setup. This opens up a new way to study orientational effects or reorientational dynamics for example via the ^{15}N chemical shift anisotropy in liquid crystalline systems.

Aluminium oxide surfaces are among the most important technical catalysts. The paper by Khabibulin et al. [34] study the local structure and in particular the catalytically relevant surface defects by an NMR crystallographic approach, employing a combination of NMR, XRD, and DFT calculations. They show that the surface of $\eta\text{-Al}_2\text{O}_3$ is formed primarily by the (111) facets, while the $\gamma\text{-Al}_2\text{O}_3$ surface is a combination of (111) and (110) facets.

Okubo et al. [35] report in their paper “THz ESR study of Spinel Compound GeCo_2O_4 ” a high-field high-frequency EPR investigation of a spin frustrated spinel compound GeCo_2O_4 . The study reveals the existence of new temperature-magnetic phases below 3 K; the critical fields of these phases were determined and estimates for the zero-field gap were obtained.

The self-aggregation of smaller organic molecules to supramolecular complexes and structures is studied by Petrova et al. [36] employing glycyrrhizic acid as model compound. They follow the micelle formation by observing the spin-spin relaxation and diffusion properties of the glycyrrhizic acid.

The paper by Pravdivtsev et al. [37] “A site-specific study of the magnetic field-dependent proton spin relaxation of an Iridium N-Heterocyclic Carbene Complex” reports a study of proton NMR relaxation of an Ir-based complex, which is frequently used in SABRE hyperpolarization experiments. Relaxation measurements performed over a wide field range enable determination of the motional correlation times for individual protons. Additionally, in the field dependence of relaxation rates features are found originating from ‘strong coupling’ of spins at low fields.

The study by Lohmiller et al. [38] “Multifrequency multiresonance EPR investigation of halogen-bonded complexes involving neutral nitroxide radicals” applies CW and pulse EPR spectroscopies, including ENDOR and ELDOR-detected NMR (EDNMR) for characterizing such complexes in liquid and frozen solution. The authors show that non-covalent interaction with the halogen-bond donor affects a variety of EPR parameters of the stable nitroxide radicals demonstrating the coexistence of several complexes that differ in donor-acceptor binding geometries.

In their contribution “Electron Paramagnetic Resonance and DFT Analysis of the Effects of Bulky Perfluoroalkyl Substituents on a Vanadyl Perfluoro Phthalocyanine” Moons et al. [39] describe an EPR and DFT work on $F_{64}PcVO$ complexes studied in solids and frozen solutions. In their analysis, the authors also illustrate the use of so-called Czjzek plots, known from Differential Perturbed Angular Correlation spectroscopy, for the interpretation of ^{14}N nuclear quadrupole coupling parameters derived from ESEEM or ENDOR spectroscopy.

Jakes et al. [40] investigate the local coordination of the manganese ions in $LiCo_xMn_{1-x}O_2$ cathode materials for lithium-ion batteries employing a combination of XRD and EPR characterization. By EPR the authors could show that the manganese ions are in the high-spin Mn^{4+} -oxidation state.

With the prospect for application to *in vivo* EPR oximetry Shi et al. [41] give in their report on “Triarylmethyl Radical: EPR Signal to Noise at Frequencies between 250 MHz and 1.5 GHz and Dependence of Relaxation on Radical and Salt Concentration and on Frequency” a detailed analysis of the relaxation behavior and pulsed EPR detection sensitivity at various low frequencies.

In conclusion, the variety of exciting papers collected in these three issues of *Zeitschrift für Physikalische Chemie* clearly demonstrates the tremendous impact the work of Kev Salikhov had on the magnetic resonance, the chemical physics and the physical chemistry community.

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