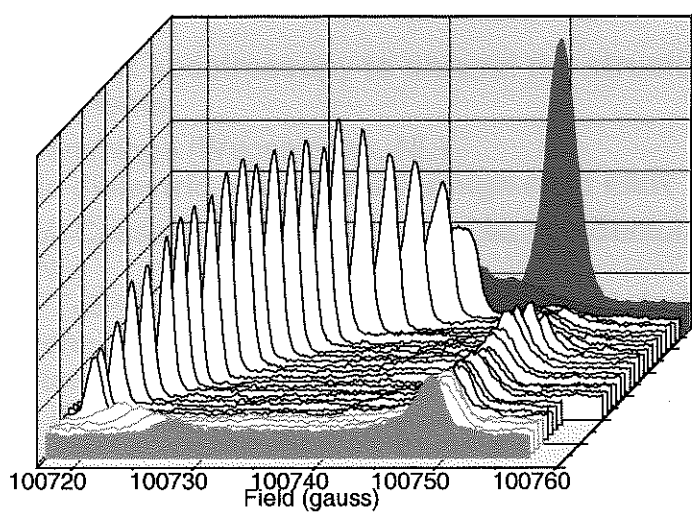


11th Meeting of the Benelux EPR Society 2003



University of Nijmegen,  
High Field Magnet Laboratory HFML



April, the 16<sup>th</sup>. 2003

# 11<sup>th</sup> Meeting of the Benelux EPR Society 2003

## HFML-16 April

Faculty of Science  
University of Nijmegen  
Toernooiveld 7, 6525 ED Nijmegen

**10:00 meeting at HFML – 02-20**

**10:30 welcome Jan Kees Maan**

**10:45 Site-directed ESR spectroscopy to unravel the structure of membrane proteins**

Marcus A. Hemminga (Wageningen, NL)

**11:15 Single-mode dielectric resonators for high-field EMR**

G. Annino, M. Cassetari, M. Martinelli, P.J.M. van Bentum (Nijmegen, NL)

**11:45 Ab initio DFT-EPR study of diatomic chalcogen defects**

Franky Stevens (Ghent, B)

**12:15 Fifty years later: copper acetate dimer revisited**

Fred Hagen (Delft, NL)

**13:00 LUNCH – followed by tour of the new installation**

**14:30 ESR Spectroscopy in Combination with Spin Trapping Techniques: Applications for Detection of Free Radical Formation by Food Components and Human Environmental Pollutants.**

J.J. Briedé (Maastricht, NL)

**15:00 Spectral Sensitizers in Photographic Systems: Molecular Structure and Intermolecular Interactions vs. Aggregation**

Jan Van Nysten (Antwerp, B)

**15:30 Coffee break**

**16:00 Single-ion versus molecular zero-field splitting tensor in an iron-(III)-oxo dimer studied by means of single crystal W-band EPR**

Peter ter Heerdt (Antwerp, B)

**16:30 ZnO at 275 GHz**

J. Schmidt, J.A.J.M. Disselhorst, H. Blok, S.B. Orlinski, (Leiden, NL)

**17:00 Cocktail time**

## Site-directed ESR spectroscopy to unravel the structure of membrane proteins

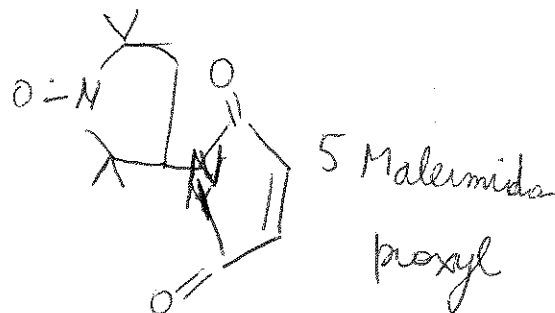
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One of the most challenging problems in structural biology of the 21st century will be to unravel the structure and function of membrane proteins. Among the spectroscopic methods, site-directed ESR spin labelling and fluorescence labelling are promising tools in the hands of a biophysicist. The techniques are based on site-directed mutagenesis in combination with specific labelling and provide specific information about the local environment, structure and dynamical properties of the labelled sites. In this presentation, an overview will be given of the current state of the art of site-directed ESR applications at various microwave frequencies to get a detailed insight into the topology, structure and dynamics of membrane proteins.

ESR + fluorescence

ESR spin label:



W-band

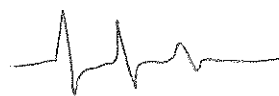


$g_x$  well resolved

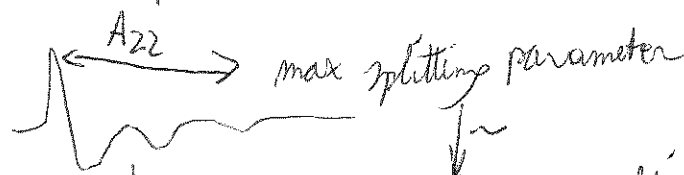
$A_2$  well resolved



these values as a function of substitution on the protein.



↓ free motion



↓ hindered motion

membrane or packing value

$$f = \sqrt{\sum \frac{m_i}{r_i d_i^2}}$$

X-band

→ Molecular Dynamics!

## Single-mode dielectric resonators for high-field EMR

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The development of a single-mode dielectric resonator specifically designed for high-frequency paramagnetic resonance applications is discussed. The use of dielectric resonators is expected to give better performances in comparison to metallic cavities, as well established at low frequency. The relatively low dielectric constant of the common good quality materials at high frequency requires the presence of a metallic shielding in order to obtain an efficient single-mode operation. The configuration proposed in this talk concerns a partially open structure in which the confinement of the radiation is guaranteed by the forbidden propagation around the dielectric region. By this way a single-mode resonator can be obtained for arbitrary values of the employed dielectric constant. The complex resonance frequencies and field distributions of the proposed non-radiative device are obtained in terms of the complex permittivity of the resonator and of the finite conductivity of the employed conductors. The actual field distribution and intensity can then be obtained even in presence of a lossy sample. An efficient excitation scheme, fully compatible with the common electron paramagnetic resonance setups, has been developed exploiting the non-radiative nature of the proposed device. Preliminary measurements around 90 GHz and 186 GHz have been then successfully realized. The specific benefits of the proposed resonator will be discussed and compared with the performances of a conventional metallic cavity.

## Ab initio DFT-EPR study of diatomic chalcogen defects

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Density functional methods are used to calculate the EPR parameters of  $X_2^-$  and  $XY^-$  ( $X, Y = S, Se$ ) lattice defects in various alkali halide lattices ( $MZ, M = Na, K, Rb$  and  $Z = Cl, Br, I$ ).

In the literature, two models have been proposed for these diatomic chalcogen defects. In the first model – monovacancy model – the  $X_2^-$  or  $XY^-$  molecular ion replaces a single halide ion. In the second model, the  $X_2^-$  or  $XY^-$  molecular ions replace two adjacent halide ions. They represent the divacancy model. In both models, the bond axis of the diatomic molecular ion lies along a  $\langle 110 \rangle$  direction.

Although a lot of EPR and ENDOR data are available, some questions remain unanswered. Firstly, two  $S_2^-$  centers with different ground states have been encountered. Second, the occurrence of different ground states for different lattices containing the same lattice defect has not been fully understood.

The answer to these questions and the validation of the DFT methods for the description of paramagnetic defects in ionic lattices were the main stimulations for the present DFT study.

For all alkali halide lattices considered, the calculated EPR parameters for the  $S_2^-$ ,  $Se_2^-$  and  $SSe^-$  defects are in very good agreement with experiment when a monovacancy model is assumed. In KCl, the DFT results suggest that one of the  $S_2^-$  defects resides in a halide monovacancy, while the other occupies a divacancy site.

Beide opletten met dat divacature / <sup>extra</sup>vacature  
verhaaltje.  
→

## Fifty years later: copper acetate dimer revisited

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PR  
In 1952 Bleaney and Bowers published their 'EPR classic' on the analysis of single crystal data from tetrakis( $\mu$ -acetato-*O,O'*)diaquadicopper(II), or copper acetate monohydrate [1]. Previous susceptibility and EPR work by others had shown the compound to behave anomalously, where simple  $S = 1/2$  paramagnetism was anticipated. Bleaney and Bowers proposed a model in which the Cu(II) ions occur in pairs subject to strong antiferromagnetic exchange coupling resulting in a singlet ground state and an  $S = 1$  triplet at approximately  $300 \text{ cm}^{-1}$  with a zero-field splitting,  $D$ , approximately equal to the X-band quantum. Subsequent structural and magnetic studies until now have been confirmative with slight refinement of the numbers [2]. Today the study of Bleaney and Bowers stands as the textbook case of exchange interaction in molecular metal clusters.

In previous high-frequency EPR studies on  $S = 1$  Ni(II) salts with  $D \approx 1 \text{ cm}^{-1}$  we [3] and others [4] have observed double-quantum transitions within equidistant triplet energy levels, and with unusual linear dependence on the microwave power, where quantum mechanics predicts a quadratic dependence [5].

We have now re-investigated copper acetate as a low-frequency alternative to the Ni(II) salts for the study of linear double-quantum transitions in powder EPR. A double-quantum spectrum has been found with Q-band and dual-mode X-band EPR. A data analysis and interpretation of the double-quantum spectrum will be presented.

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- [3] P. J. van Dam, A. A. K. Klaasen, E. J. Reijerse, and W. R. Hagen, Application of high frequency EPR to integer spin systems: unusual behaviour of the double-quantum line, *J. Magn. Reson.* **130**, 140-144 (1998).
- [4] D. Collison, M. Helliwel, V. M. Jones, F. E. Mabbs, E. J. L. McInnes, P. C. Riedl, G. M. Smith, R. G. Pritchard, and W. I. Cross, Single and double quantum transitions in the multi-frequency continuous wave electron paramagnetic resonance (cwEPR) of three six co-ordinate nickel(II) complexes:  $[\text{Ni}(\text{EtL})_2(\text{Me}_5\text{dien})]$  and  $[\text{Ni}(\text{5-methylpyrazole})_6\text{X}_2]$ ,  $\text{X}=(\text{ClO}_4)^-$  or  $(\text{BF}_4)^-$ . The single crystal X-ray structure at room temperature of  $[\text{Ni}(\text{5-methylpyrazole})_6](\text{ClO}_4)_2$ , *Faraday Trans.* **94**, 3019-3025 (1998).
- [5] M. Göppert-Mayer, Über Elementarakte mit zwei Quantensprüngen, *Annalen der Physik* **9**, 273-294 (1931).

## **ESR Spectroscopy in Combination with Spin Trapping Techniques: Applications for Detection of Free Radical Formation by Food Components and Human Environmental Pollutants.**

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Free radical formation is involved in the initiation and progression of multi-stage diseases such as cancer and atherosclerosis. The development of these diseases is modified by nutritional components as well as environmental compounds that influence the formation of short-lived reactive species like carbon- and oxygen-centered free radicals. These radicals are able to damage biological important macromolecules including DNA. We use X-band continuous wave electron spin resonance (CW-ESR) spectrometry in combination with spin-trappings techniques to detect and identify free radical formation during metabolism of the food-related carcinogenic and atherosclerotic compounds like the model polycyclic aromatic hydrocarbon benzo(a)pyrene (B[a]P) heterocyclic aromatic amines (HAA's) and beta-carotene. Moreover, this technique is also used to determine free radical activity of ambient particulate air pollution (particulate matter (PM)). The spin traps DMPO, DEPMPO and POBN react with short living free radical species, e.g. reactive oxygen species (ROS), yielding longer living species which offers the ability to measure their formation with ESR. In this way, the ESR technique offers more fundamental insight into the mechanisms involved in the induction of (oxidative) DNA damage by food components and environmental pollution that is related to the occurrence and progression of several diseases.

## Organic Sensitizers in photographic systems: molecular structure and intermolecular interactions vs. aggregation

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In photographic systems organic molecules are used to spectrally sensitize the silver halide (AgHal) emulsion. These spectral sensitizers can be combined on the same AgHal crystals to get the desired effect of supersensitization<sup>1</sup>. In the supersensitization mechanisms the molecular and intermolecular structure or aggregation play a crucial role. In this study two sensitizers that are structurally closely related are being studied: the first one (G1) is a green sensitizer, while the second one (R3) is a red sensitizer. With this combination we aimed to study the hole trapping supersensitization mechanism<sup>2,3</sup> because of the relative positioning of their energy levels.

For hole trapping on R3 it is essential that the HOMO level of the R3 molecules is positioned above the HOMO of G1.<sup>4</sup> Further it is necessary that both sensitizers are in close contact with each other, so that hole transfer can occur between them. From the absorption spectra we learn that in the combined system R3 is adsorbed on the AgHal in a monomeric state, indicating that it is distributed between the G1 aggregates rather than forming an aggregate on its own.

Light-induced EPR experiments, in which G1 is optically excited, clearly show that hole transfer from G1 to R3 occurs and as a result R3 radicals are formed in all the systems with R3. The G1 and R3 radicals could be discriminated on the basis of a small but significant difference in g-value. For the first time the hole trapping supersensitization mechanism in these photographic systems could be validated by means of EPR spectroscopy.

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P.B. Gilman and T.D. Koszelak, *J. Photogr. Sci.*, **21**, 53 (1973)

J.P. Zheng, A.G. DiFrancesco, R.K. Hailstone, et al., *Imaging Sci. J.*, **50**, 63 (2002)

Work supported by the IWT: the Flemish Institute for the Encouragement of Scientific and Technological Research in Industry.



## Single-ion versus molecular anisotropies of an Fe(III)-oxo dimer studied by single crystal W-band EPR.

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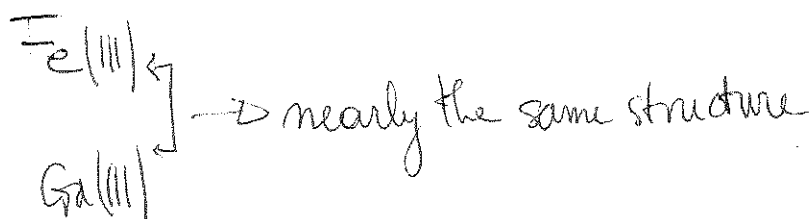
Typical *single molecule magnet* behaviour is observed in a series of Fe<sup>III</sup>-oxo complexes. Two dimers of this family are studied by single-crystal W-band EPR.

The first complex is a Ga<sup>III</sup> dimer doped with a small amount of Fe<sup>III</sup>-ions. The second complex is an isomorphous Fe<sup>III</sup> dimer.

The first compound is used to derive the single-ion *zero field splitting* tensor of the Fe<sup>III</sup>-ion. This FeGa dimer can be thought of as an Fe<sup>III</sup> monomer in which the basic single-ion properties can be studied. The next step is to use the single-ion properties of the Fe<sup>III</sup> center and separate them from other major interactions, like dipolar contributions.

The principal values of the Fe<sup>III</sup> ZFS tensor and the orientation of the principal directions relative to the crystalline axes are determined. The resulting ZFS parameters are consistent with the parameters derived from high-frequency powder EPR measurements at 525 GHz. The principal axes are pointing in arbitrary directions with respect to the directions of the different bonds.

From the Fe<sup>III</sup> spectra the principal values and axes of the complete anisotropic spin-spin interaction are determined. The principal values and directions demonstrate that the dipolar interaction is not the dominant contribution to the spin-spin interactions. The anisotropic exchange interaction therefore has a significant contribution to the overall magnetic anisotropy of these specific molecular magnets of the Fe<sup>III</sup>-oxo family.



$$\text{Fe}^{3+} \quad S=5/2 \quad \rightarrow \quad \mathcal{H}_Z + \mathcal{H}_{ZFS}$$

$$\downarrow$$

$$SOS$$

$$D = \frac{g\mu_B}{2k_B} \left( \frac{1}{2} \right)$$

$$\hookrightarrow \text{Fourth order terms? } B_{4,e}?$$

## ZnO at 275 GHz

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G. Baranov  
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By using EPR and ENDOR spectroscopy at 95 GHz it has been shown by us that the EPR signal observable in nominally undoped *n*-type crystals of ZnO is related to the presence of interstitial H atoms that act as a shallow donor [1]. In a more recent EPR study of the same crystals at 275 GHz it appears that at 5 K a hole can be burnt this EPR line. It is even possible to displace the resonance line by as much as 28 G by subjecting the EPR line to resonant microwave irradiation. These phenomena are thought to be caused by the  $^{67}\text{Zn}$  ( $I=5/2$ ) nuclear spins that have a hyperfine interaction with the unpaired electron spin and that become polarized via the Overhauser effect.

We will show that this remarkable effect finds its origin in two aspects of EPR at 275 GHz and 5 K. First the Boltzmann distribution over the electron spin levels is extremely large, i.e., we start with an almost completely polarized system of electron spins. Second the spin-lattice relaxation between the electron spin levels is dominated by spontaneous-emission processes which are caused by the zero-point fluctuations of the phonon field. It is thought that these zero-point fluctuations in the phonon system also induce the cross-relaxation rate necessary for the Overhauser effect that polarizes the nuclear spins.

In subsequent experiments on ZnO nanoparticles with dimensions of only 3.2 nm we observe EPR signals of a shallow donor that are related to Li. Again hole-burning effects are observed that point to a high polarization of the  $^{67}\text{Zn}$  nuclear spins. Remarkably the spin-lattice relaxation rate in these nano particles is much slower than in the bulk crystals of ZnO. This observation may be explained by assuming that the receiving modes in the phonon spectrum at 275 GHz are absent in these nano particles.

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*Shallow donors in ZnO*

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Senior guest researcher: National Institute for Materials Physics, Boekarest, Roemenie)

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