



# ELEXSYS-II E 500

- The Research Platform for Electron Paramagnetic Resonance

# ELEXSYS-II E 500

Electron paramagnetic resonance is a versatile tool used in all natural sciences. The intrinsic high sensitivity of this method enables detection of paramagnetic centers down to the nano molar concentration range. This non-destructive technique is the ideal research tool for chemical, biological, physical, and material science studies.

## Past, Present and Future

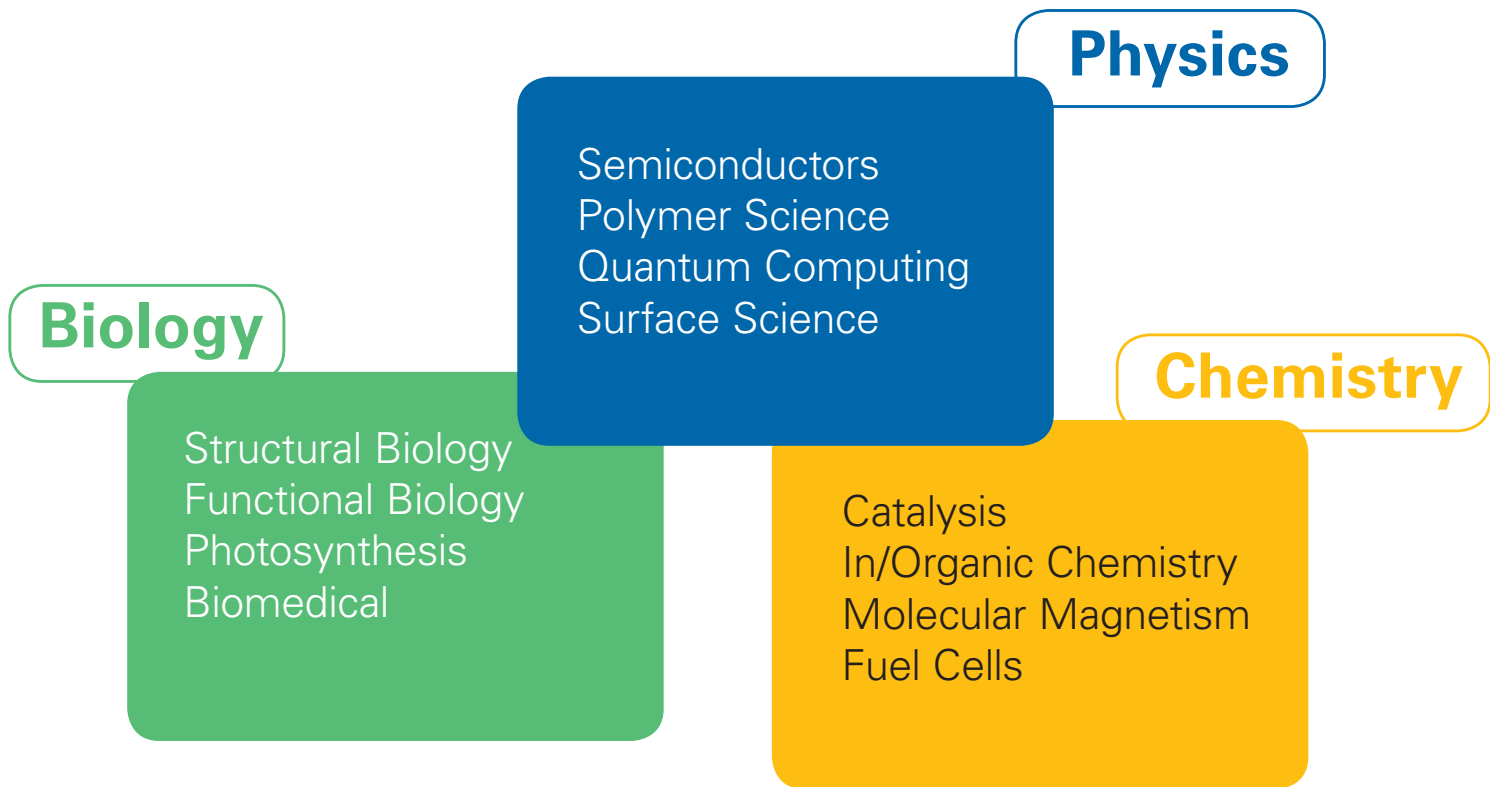
The ELEXSYS EPR Series was introduced at the Rocky Mountain Conference on Analytical Chemistry in 1996 and is the research platform for modern, advanced EPR. In close cooperation with the leading research institutes of the world Bruker pushed the technology of the ELEXSYS series to the highest level. The ELEXSYS covers nearly all known applications in the field of EPR.

The second generation pulse devices, SpecJet-II and PatternJet-II, were launched in 2006. Just recently the DICE-II ENDOR unit debuted with an expanded variety of new features.

Major changes in system architecture were developed and implemented in order to introduce the new ELEXSYS-II series. The OS9 hardware acquisition server has been replaced by a software server and the SuperX microwave bridge has undergone a redesign with improved specifications.

A new multi-purpose signal processing unit (SPU) plays a central role in the expanded capabilities of the ELEXSYS-II. The SPU replaces the signal channel, fast digitizer, and rapid scan with a single integrated unit offering unprecedented performance, specifications and newly developed acquisition mode flexibility.





If your instrument is accessible outside your network the system can now be fully remote controlled for instrument checks and enhanced assistance from the EPR home office.

The expectations for a high level EPR research instrument go far beyond the capabilities of the individual units. It is always several devices working in concert that contribute to a successful experiment. It is part of the ELEXSYS design philosophy that individual units are integrated together for the needs of EPR and matched to each other for optimum performance.

The Xepr front-end software underlines the philosophy of the ELEXSYS II and guides the inexperienced as well as the most experienced user through simple or very complex experiments. The legendary stability of our instruments allow routine 24/7 operation even in low temperature mode with the highest precision.

The ELEXSYS-II is another major milestone in EPR instrumentation, designed with today's application in mind but open to tomorrow's challenges.

# The ELEXSYS-II Platform

ELEXSYS-II provides the platform on which all modern EPR techniques are built. The system supports multi-frequency EPR from 1 to 263 GHz in CW and FT modes as well as EPR imaging for animal research or material science. Multi-resonance is the domain of the recently introduced DICE-II unit, a powerful add-on for Electron-Nuclear Double and Triple Resonance (ENDOR).

## E 500 highlights & standard features

- SuperX microwave units for world record sensitivity
- Ultra high resolution field controller
- Digital rapid field scan module
- Stationary and time resolved experiments
- Multi purpose signal processing unit
- Reference free spin counting
- Xepr software package



E 580



E 780 quasi optical front end

## The platform members

- E 540 imaging system
- E 500T transient EPR
- E 580 CW/FT spectrometer
- E 600/680 W-band instrument
- E 780 mm-wave EPR at 263 GHz



E 540



E 680

	L-Band	S-Band	X-Band	K-Band	Q-Band	W-Band	mm-wave
CW-EPR	■	■	■	■	■	■	■
FT-EPR	■	■	■	■	■	■	■
CW-ENDOR			■		■		
Pulse-ENDOR			■		■		■
Pulse-ELDOR	■	■	■		■	■	■
Imaging	■	■	■				
Saturation recovery	■	■	■		■		
Rapid scan	■	■	■				
Transient EPR	■	■	■		■	■	
ODMR			■		■		

# The SuperX Microwave Units

The X-Band ELEXSYS-II instruments are equipped with the SuperX feature, the package for the ultimate sensitivity. SuperX comprises a selected high power, ultra-low-noise Dual-Gunn source, and the super-high-Q cavity. The combination of these devices has achieved an order of magnitude increase in CW-EPR sensitivity in X-band. For the traditional sensitivity measurement mode we specify a weak-pitch signal-to-noise ratio of 3000:1 for the E 500 CW-EPR spectrometer.

## Super-high-Q probehead (SHQE)

- Optical window, standard
- Cooling side-plates for temperatures up to 700 K, optional
- Single line reference marker, optional

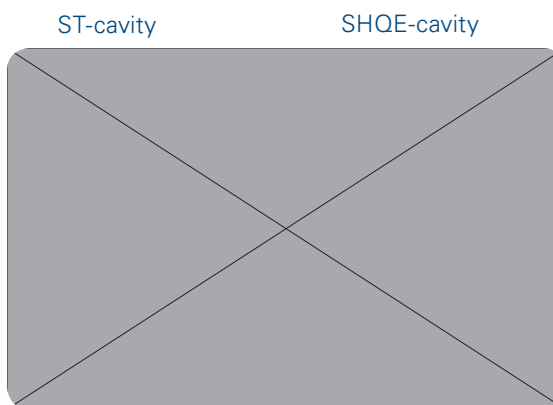


## SuperX microwave bridge

- Integrated controller
- Integrated frequency counter
- 90 dB attenuation standard
- 6.5 MHz receiver bandwidth (30 ns time resolution)
- Fully calibrated signal amplitude, a prerequisite for reference free spin counting
- 80 MHz wide electronic tune picture with zoom function for optimum control of high and low Q resonators
- High fidelity AFC

## Microwave bridge options

- Absorption and dispersion detection
- Receiver for transient detection with 200 MHz bandwidth



Cu<sup>2+</sup> Histidine, 0.5 mM at 20 K and 20 dB

# Signal Processing Unit (SPU)

The SPU is a highly integrated device offering all acquisition modes in modern CW-EPR. Even EPR experiments outside the standard repertoire can be easily mastered with the 8 input channels and many trigger modes. The enormous dynamic range on the X- and Y-axis simplifies user interactions as many fine adjustments of the past are no longer necessary.



Rapid scan coils mounted on the magnet pole face

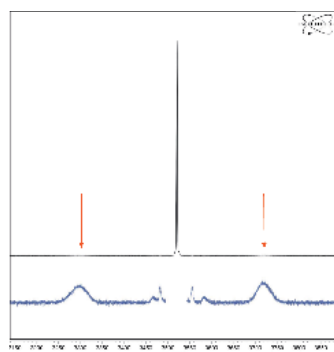
The rapid scan module features up to 200 G sweep range and 200 Hz scan rate at 100 kHz modulation frequency. In rapid scan vs time mode fast dynamical evolution of full spectra can be monitored

## Summary of SPU features

The application suite is a full set of programs and protocols optimized for a wide range of applications.

- Digital lock-in with up to 5 simultaneously detected harmonics
- Simultaneous detection of 0 and 90 degree modulation phase
- Modulation frequencies up to 1 MHz
- Dual channel for simultaneous absorption and dispersion measurement
- Rapid scan ramp generator and detection system, modulated and direct detection
- Transient recorder with 14 bit single shot amplitude resolution and 8 ns time resolution
- Predefined and user defined channels
- Input channels with AC and DC coupling
- Internal and external triggers
- 256,000 points on field and time axis
- 65,536 points on transient time axis

## Amplitude resolution



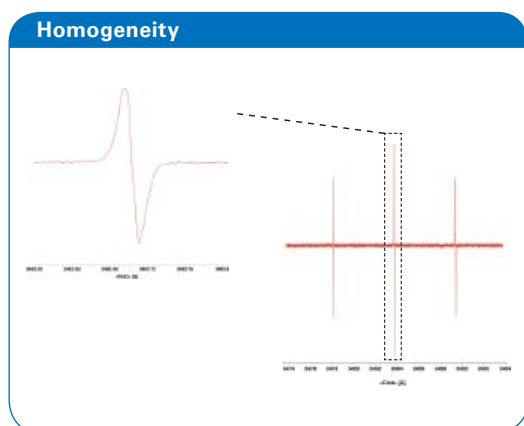
The 27 bit amplitude resolution allows the measurement of very small and very large signals in a single scan. Digital zooming of the spectrum reveals all details. The example shows a spectrum of the E' center in quartz where the ratio of the largest to the smallest line is 750.

# Magnetic Fields

A magnet system for EPR comprises an electromagnet, a power supply and a field controller with Hall sensor to measure and regulate the field. Our magnet systems are optimized for EPR acquisition. This means that specifications like homogeneity, homogeneous volume, stability and sweep range are the parameters in focus during development, production and test. The magnet homogeneous volume, for example, is optimized for sample tubes and flat cell (the most common sample containers in EPR).

## Magnet homogeneity

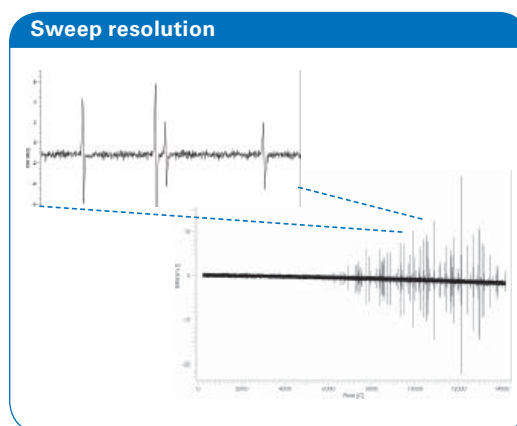
The 10" magnet has a homogeneity of 10 mG over a length of 22 mm along the sample tube axis



$^{14}\text{N}@C_{60}$  in solution with dimensions of  $\varnothing = 3$  mm and  $L=15$  mm showing a line width of 15 mG

## Sweep range and resolution of Hall controller

The Hall controller is calibrated from -18 kG to +18 kG with a precision better than 500 mG. It can sweep the full range with a single scan with a resolution of up to 256,000 points



Q-Band spectrum of  $\text{O}_2$  in air at reduced pressure. 14 kG sweep range with 80 mG sweep resolution

## ER 036TM Teslameter,

The highest precision in magnetic field measurement is achieved by an NMR Teslameter. The Teslameter is a compact  $^1\text{H}$  NMR spectrometer which has a resolution of 1 mG and operates from 1.5 to 15 kG with a single probe. It is fully integrated into the Xepr measuring routines.

## ER 033D FF-lock

The ER 033D field-frequency lock uses the Teslameter and the frequency counter to establish a g-factor stable measuring condition. It is recommended for narrow lines, long acquisition times and ENDOR experiments.

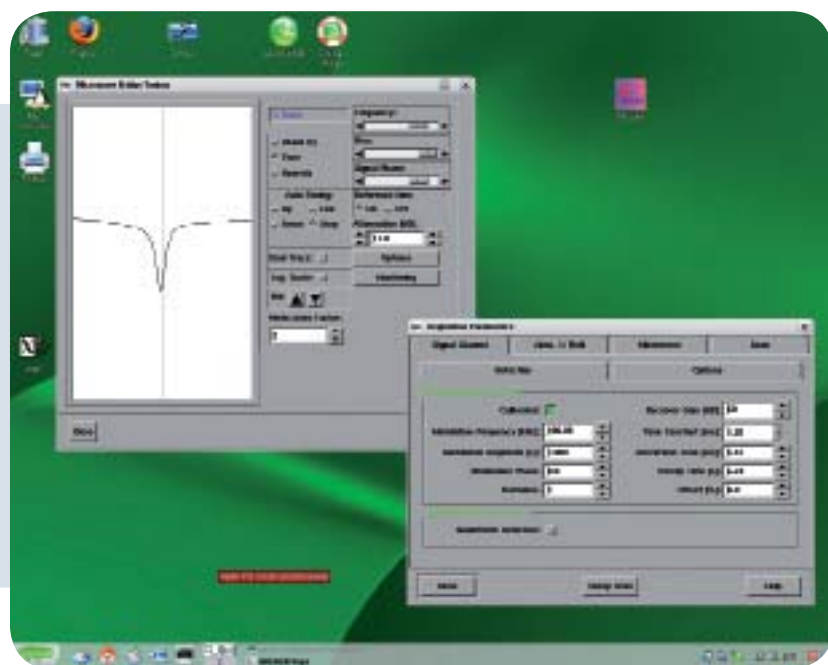
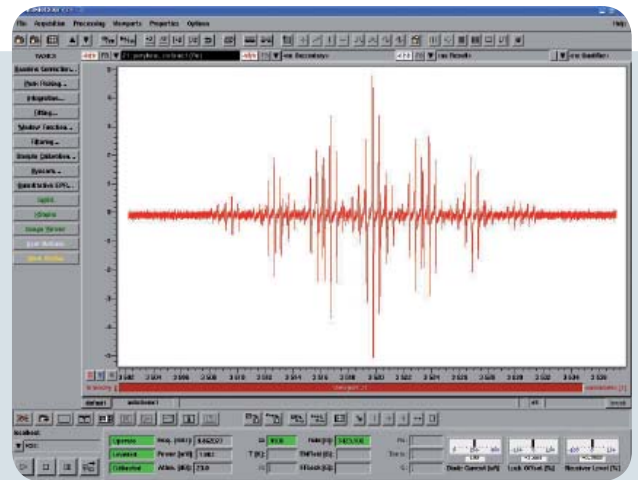
Various combinations of magnets and power supplies are available to match different needs and budgets. The magnet systems can be chosen based on maximum field, air gap, homogeneity and weight. All ELEXSYS-II magnets are equipped with rapid scan coils. The 9.5" and 10" magnet can be both equipped with the new 12 kW bipolar power supply. Together with the fully bipolar calibrated Hall controller the magnets can be swept from - max field to + max field in one scan.

# Xepr for Instrument Control and Acquisition

The Xepr software suite for experiment design and system control provides unprecedented flexibility and ease of use. Whether you are dealing with a simple CW experiment or a complicated multiple-resonance 2D experiment, the graphical user interface of Xepr ensures easy instrument control, experiment definition and execution.

The status of all instrument parameters is constantly monitored and all parameters of the experiment are stored together with the data set. Any experiment can therefore be reproduced and shared with others worldwide.

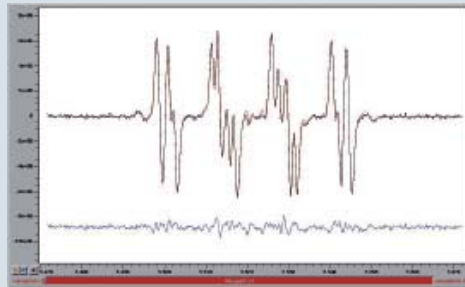
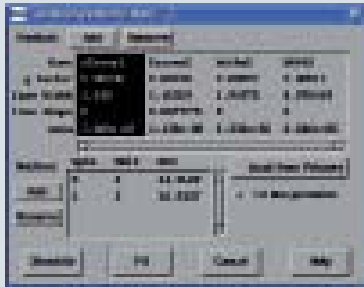
Many times recording just a single EPR spectrum is the first step in a sequence of experiments. Xepr provides numerous fully automated 2D experiments for a deeper insight into the sample properties. For example, 2D power saturation can be performed with automatic tuning between successive power steps to assure correct instrument conditions throughout the complete experiment. The various acquisition features of Xepr are depicted in the diagram on the backcover.



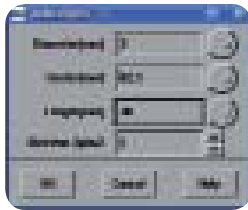


# Xepr for Data Processing and Analysis

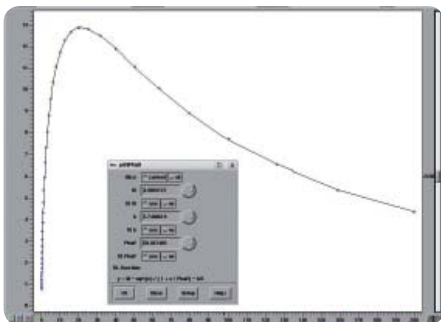
Xepr offers a vast range of tools to process and analyze all kinds of experimental data. No matter whether you want to do a base line correction, a clean double integral, a peak-picking, a  $P_{1/2}$  fit or count the number of spins in your sample, all analysis tools are always at hand and can be executed without interfering with a running experiment. In addition, user defined processing macros can be generated to minimize tedious work in repetitive experiments.



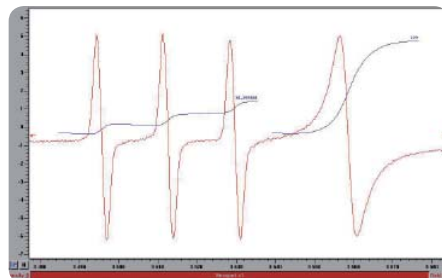
The new spin trapping program “SpinFit” allows the disentanglement and quantification of spin adducts by fitting input parameter from a database to the experimental spectrum. The example shows the fit of 4 spin adducts to one spectrum. The residual (exp-fit) is shown as well.



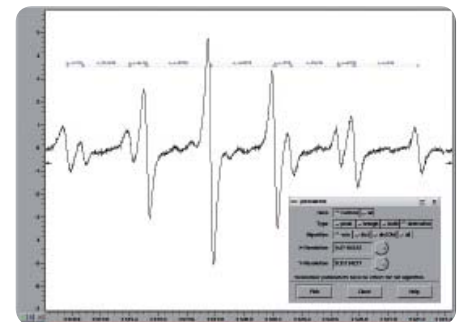
With only 4 user input numbers the spin counting module reports precise values of the number of spins and concentration.



$P_{1/2}$  fit for the analysis of saturation curves



The processing task for quantitative EPR handles, for example, the E 4100MK single line reference marker. Relative quantitation can be performed based on peak-to-peak or double integral analysis.



Peak-picking with distance measurement

# Specialized Probes

The standard super-high-Q resonator is an excellent choice for all sample classes and spin systems. Nevertheless, there are good reasons why a dedicated resonator may be a better option in certain situations. For example, a large volume cavity is not optimum if only a small sample quantity is available.

## Specialized X-Band probes

**ER 4123D**, spin label resonator which requires only 3  $\mu$ l sample volume

**ER 4116DM**, dual mode resonator for the investigation of forbidden transitions of spin systems with  $S > 1/2$

**ER 4117MX**, mixing resonator with 3  $\mu$ l active volume

**ER 4103TM**, cylindrical cavity for large flat cells

**ER 4108TMHS**, compact TM mode cavity with only 20 mm width. Specifically designed for the 2D high power gradient system

**ER 4102ST**, standard rectangular cavity which allows 70 ns time resolution in transient EPR

**ER 4105DR**, double resonator for quantitative EPR

**ER 4104OR**, optical transmission resonator

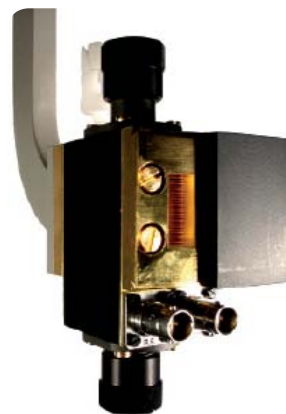
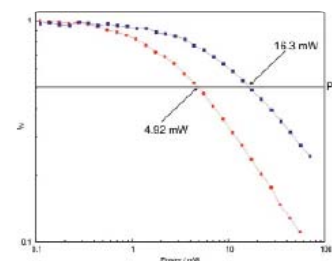
**ER 4114HT**, high temperature cavity for T up to 1300 K

**Flexline**, the product line for high filling factor resonators from L- to X-band

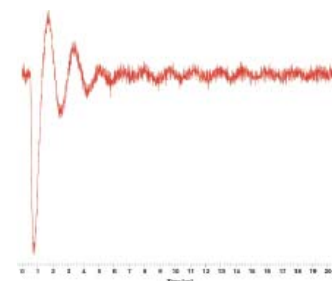


ER 4123D

Saturation curves of 10  $\mu$ M TEMPOL in water measured with the ER4123D resonator. Air or  $N_2$  atmosphere results in  $P_{1/2}$  of 16.3 mW and 4.92 mW, respectively.



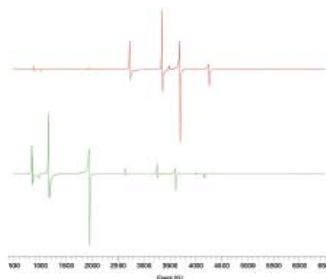
ER 4102ST



Rabi oscillation of an optically excited triplet state observed in a transient EPR experiment with the ER 4102ST cavity at 9 dB microwave attenuation



ER 4116DM



Separating allowed (top) and forbidden transitions (bottom) with the ER 4116DM resonator at low temperature

# Multi Frequency Accessories

Multi-frequency EPR is a tool to disentangle magnetic field dependent and independent terms, to increase the resolution for a certain interaction, to simplify spectra or to change the sensitivity window for motional effects. Bruker BioSpin's EPR product range encompasses two orders of magnitude in microwave frequencies, from 1 GHz to 263 GHz. For each band specific resonators are available dedicated to the typical applications.

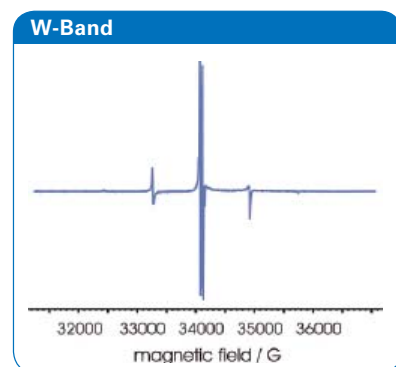
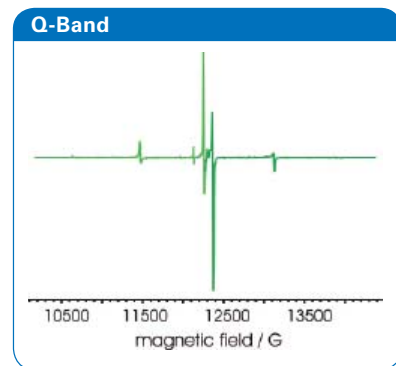
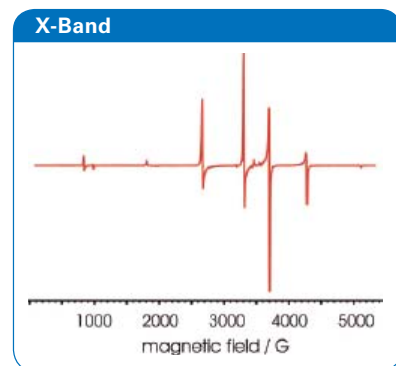
- L-Band at 1 GHz with probes for animal research and spectroscopy
- S-Band at 3.6 GHz with Flexline probe for spectroscopy
- X-Band: most common frequency at ca. 9.8 GHz
- K-Band at 24 GHz with a VT probe for spectroscopy
- Q-Band at 34 GHz with VT probes for EPR/ENDOR and optical excitation
- W-Band at 94 GHz with VT probes for EPR/ENDOR and optical excitation
- mm-wave at 263 GHz with resonant and non-resonant VT probes



Q-Band probehead



E 600-1021H  
W-Band probehead



Multi frequency EPR of 1%  $\text{Cr}^{2+}$  in  $\text{CsAl}(\text{SO}_4)_2$  showing the progressive simplification of the spectrum with increasing frequency

# ENDOR Accessory

ENDOR (Electron Nuclear DOuble Resonance) is an extremely valuable technique to determine electron-nuclear hyperfine interactions. It can greatly simplify very complicated EPR spectra. Furthermore, the resolution is enhanced with ENDOR if an EPR spectrum appears structureless due to inhomogeneous broadening.

## E 560D DICE-II

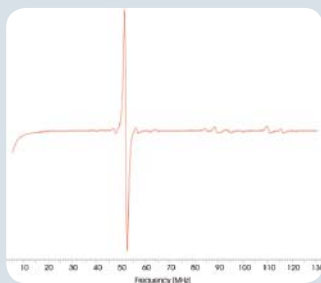
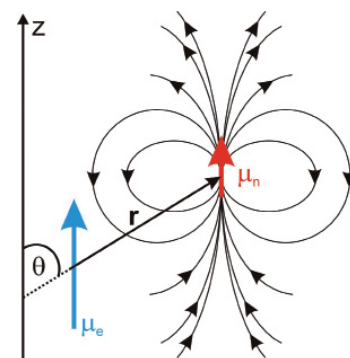
The newly developed ENDOR system for the ELEXSYS family covers a frequency range from 1 to 650 MHz.

The system comprises

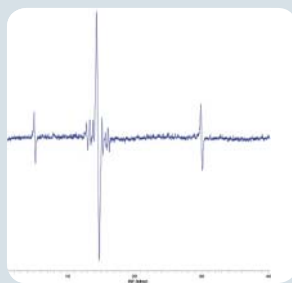
- ENDOR/TRIPLE RF generator
- ENDOR low temperature dewar
- CW-ENDOR resonator (X- or Q-Band)
- Acquisition software for FM and AM, ENDOR, TRIPLE, EIE
- PNT ENDOR test sample
- Water cooled 50 Ohm load
- Various options for RF amplifier
- Optional FF-lock



Rack with DICE-II RF unit and ENDOR amplifier



Alanine low temperature powder ENDOR spectrum at Q-Band



Malonic acid single crystal X-Band <sup>1</sup>H ENDOR spectrum



EN 801 X-Band CW-ENDOR cavity



EN 250VTDJ  
N<sub>2</sub> ENDOR dewar



EN 260HVDJ  
Helium ENDOR dewar

# Accessories for Sample Conditioning

Different sample categories and various types of investigation require additional accessories. A variable temperature system, for example, for the analysis of a temperature dependent sample property or a computer controlled goniometer for an automated and precise single crystal rotation. All accessories are seamlessly integrated into the ELEXSYS-II hard- and software.

## ER 4131VT

Nitrogen based variable temperature system for the range 100 to 1300 K.



Controller for ER 4131VT system



Controller for ER 4112HV system

## ER 4112HV

Helium based variable temperature systems for the range 1.8 to 300 K.

The system can be configured as well with cryostats for Flexline probes, for K-, Q-, W-Band and mm-wave resonators.

Our N<sub>2</sub> and Helium based VT units are fully integrated into the Xepr software and are characterized by low consumption and high stability

## ER 4110AX AquaX

Multi capillary (4 or 19) sample cell for quantitative EPR with aqueous solutions



## ER 203UV

150 W UV irradiation system with mercury or xenon lamp, with optional light guide, shutter, chopper and water filter



Helium cryostat with SHQE cavity mounted



Aqueous solution cells and holder

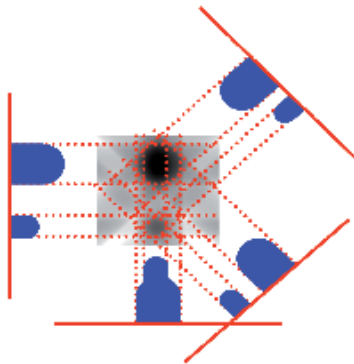


ER 218PG1 programmable goniometer for single crystal studies with 1/8 deg resolution

# Magnetic Field Gradients

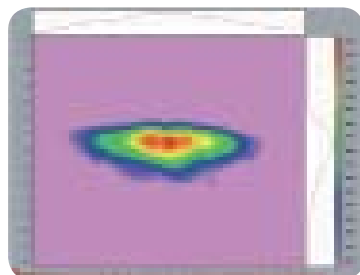
In standard EPR the magnet is optimized for the best possible homogeneous field. For imaging a well defined linear field gradient is superimposed onto the homogeneous field to achieve a spatial encoding. EPR imaging can provide the spatial distribution of paramagnetic species and their EPR parameters. For example, we can measure the EPR line width within each voxel to determine local oxygen pressure in a tissue. The imaging accessory comprises 2D or 3D water cooled gradient coils, powers supplies, gradient controller and acquisition and processing software for up to 4D imaging.

In an imaging experiment spectrum profiles are recorded for different gradient directions. The profiles are then used in a back projection routine to reconstruct the object



## E 540GCL

- 3D gradients with 40 G/cm for X-and L-Band
- Compatible with ER 073W and E 540M magnets



Sensitivity profile of L-Band surface coil

## E 540GCX2

- 2D gradients with 200 G/cm for imaging in X-Band
- Compatible with ER 073 magnet
- 25 mm air gap
- ER 4108TMHS resonator
- Compatible with ER 4112HV Helium system



ER 4108TMHS

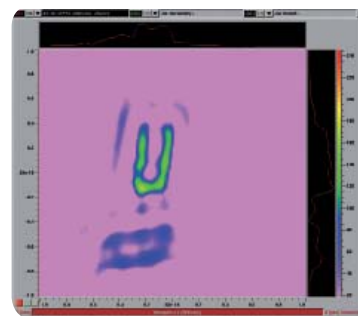


Image of two DPPH crystals with 25 um pixel resolution

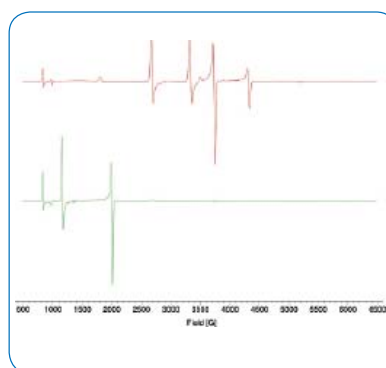
# XSophe: Spectra Simulation and Optimization

XSophe provides scientists with an easy-to-use research tool for the simulation and analysis of isotropic, randomly orientated and single crystal CW-EPR spectra.

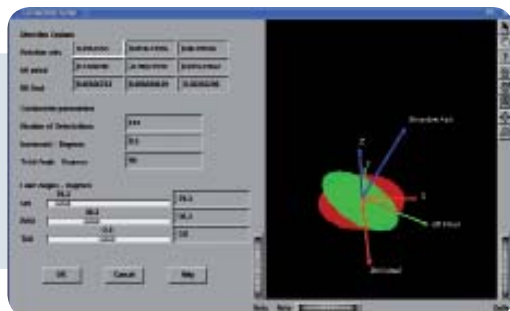
The XSophe graphical user interface allows the definition of the spin system, setting up instrument parameters and selecting an optimization algorithm. The simulated spectra are automatically transferred to Xepr for further analysis and processing.

## Magnetic and other properties of the Sample

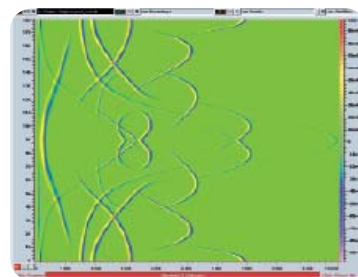
- G tensor
- Hyperfine coupling(s)
- Linewidth models
- Single crystal road-maps
- Temperature variation



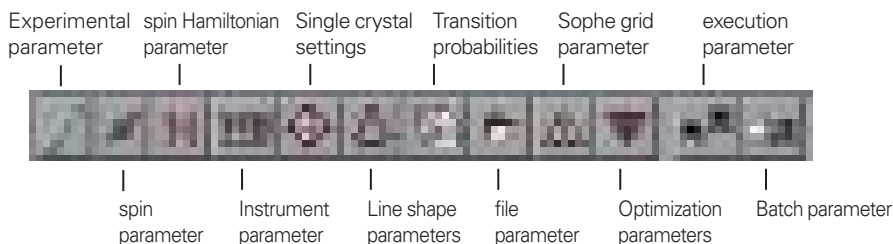
Separating allowed (top) and forbidden transitions (bottom) of a  $S=3/2$  powder sample with an XSophe simulation (for experimental data see page 10)



Menu for the input of single crystal simulation parameters



Simulation of a single crystal roadmap of a spin system with  $S=5/2$ ,  $g=2$  and  $D=-4.5$  GHz



A convenient icon bar walks you through the steps towards a successful simulation

# The ELEXSYS Family: A Myriad of Features

