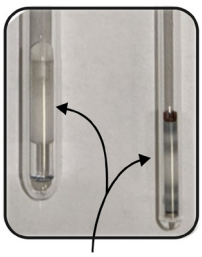
MAGNETIC RESONANCE

Magnettech ESR5000

Evaluating DNP Agents with the Bruker Magnettech ESR5000 Benchtop EPR Spectrometer

Innovation with Integrity

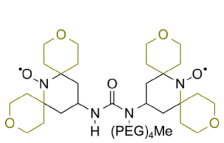


3.2, 1.3 mm MAS rotor in 5, 3 mm EPR tube

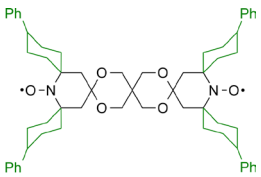
Introduction

Dynamic Nuclear Polarization (DNP) is a method to transfer spin polarization from unpaired electrons (polarizing agents, PA) to hyperpolarize nuclear spins. The result is up to 300x sensitivity gain in solids NMR, and often more than 10,000x in solution NMR using the related technique of dissolution DNP (d-DNP). Developments of the past 10+ years have advanced DNP via rational design of PAs such as binitroxides and hetero biradicals, as well as use of various narrow-line monoradicals, paramagnetic metal ions or other endogeneous unpaired electrons. Likewise, for d-DNP, a wide range of radical systems have been developed as PAs suited to specific applications.

The best DNP (and d-DNP) depends on optimal radical concentrations, as well detailed magnetic and structural properties of the PA, including exchange and dipolar interactions, geometrical orientations of radical pairs, or similar interactions among monoradicals as a function of concentration or aggregation. Furthermore, electron spin-relaxation behavior also determines DNP quality. The Bruker Magnettech ESR5000 is an essential tool to investigate such characteristics and ensure sample integrity for best DNP NMR results.



AMUPol



TEKPol

Key Features

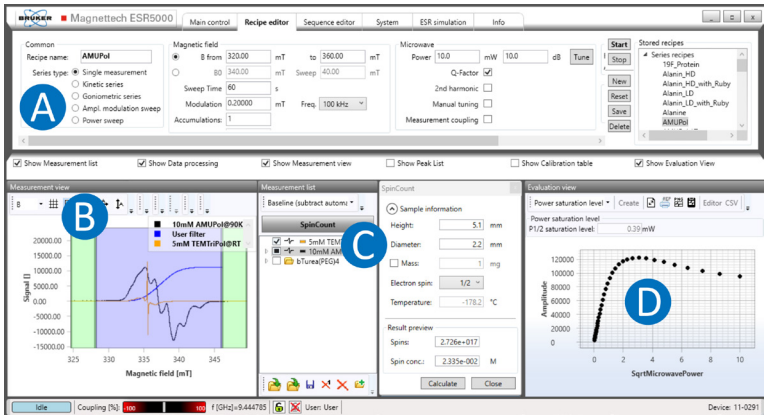
- ESRStudio user-friendly software
- SpinCount module to quantify radical concentrations without an internal reference
- Cavity auto-tuning range for study of samples in both aqueous and organic solvents
- Measurement of samples in various MAS rotors
- Compatible with cryogenic temperatures
- Automatic power sweep and fitting to obtain $P_{1/2}$, a predictor of DNP performance
- Compact design (40 x 28 x 19 cm, 45 kg)

ESRStudio: Execution, Display & Analysis

The ESR5000 spectrometer comes with a powerful, user-friendly software for acquisition, processing, and analysis, all facilitated by clear, well-thought GUI workflow. Even with limited EPR experience, researchers may utilize a full suite of sophisticated spectroscopic tools on the ESR5000, including some ideally suited to DNP.

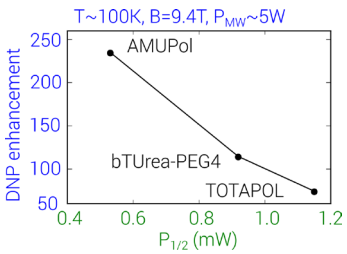
As shown in the layout overview (below), ESRStudio includes (A) clear parameter definitions, and one-click execution for automatic tuning and data acquisition. The spectral display in (B) Measurement view shows an overlay of spectra from two DNP samples prepared and analyzed in 3.2 mm MAS rotors held in a standard EPR tube. This works for all Bruker MAS DNP rotors ≤ 3.2 mm when packed with either solid or liquid samples, including in aqueous or organic solvents.

The SpinCount module in (C) allows reference-free determination of electron spin concentrations. This is a key quality check for freshly prepared DNP samples or to ensure the integrity of older samples.

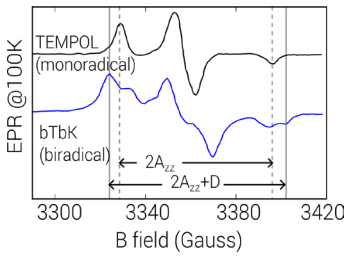


ESRStudio example layout for DNP applications, with (A) Acquisition Parameters, (B) Measurement View, (C) SpinCount module and (D) Power Sweep features highlighted.

DNP efficacy partly depends on electron spin relaxation (T_{1e}). Thus, DNP gain has a strong linear correlation with $P_{1/2}$, the microwave power at half saturation. An example $P_{1/2}$ determination is shown in (D) above, and here resulting correlation of DNP gains vs. $P_{1/2}$ are shown for various common DNP radicals. In under an hour, this approach can assess suitability of newer generation radicals or endogenous PAs for DNP.



ESRStudio also allows evaluation to assess dipolar and exchange coupling. In DNP literature, these have been used to screen biradicals for suitability in various applications and at various magnetic fields.



Low-Temperature Accessories

Two different low-temperature accessories (below) are available to enable experiments on the ESR5000 at an either fixed 77 K (cold finger) or for 93 to 473 K (variable temperature system). This can be valuable to ensure relevance of EPR measurements to DNP experiment typically near 95 – 100 K. The finger dewar maximizes convenience and affordability, while the VT with 50 L LN2 dewar and T-controller offers a robust in-line design for maximum flexibility.

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Magnettech ESR5000
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