

IMPROVED NMR SENSITIVITY WITH AN INTEGRATED EPR-NMR SPECTROMETER

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Technology Description

Researchers in Dr. Alexander Barnes's lab at Washington University have developed a low-noise integrated EPR-NMR spectrometer with improved NMR sensitivity and faster acquisition times. This patented device can increase sensitivity in NMR experiments (potentially by a factor of 20,000) and acquisition speeds (potentially 200 million times faster) by using a frequency-agile gyrotron with the ability to rapidly modulate its output microwave frequency. By altering the gyrotron anode voltage, short pulses of microwaves not only sweep through the EPR line width, but also control the EPR spins simultaneously with a broad excitation bandwidth. Using this device, researchers can perform electron and nuclei coupling (to increase NMR sensitivity), electron decoupling (to reduce noise), time-domain DNP (to increase speed), and capture long-range EPR-NMR measurements (for characterizing large molecules).

Stage of Research

- **Theoretical analysis and simulations** to determine the time constants required to quickly tune the gyrotron output frequency and control EPR spins
- **Inventors have assembled 3 gyrotrons** with a fourth one in production
- **Magic-angle spinning (MAS) DNP experiments** show that the device reduces noise and increases NMR sensitivity: electron decoupling reduces line widths, lengthens transverse relaxation times, and increases the intensities of resonances in NMR spectroscopy

Applications

- **EPR-NMR spectroscopy** with end-user applications such as:
 - MAS DNP NMR spectroscopy
 - Time domain DNP NMR spectroscopy
- **Coupling and decoupling of electrons and nuclei**
- **Characterization of molecules in their natural state:** device can measure long range nuclear electron distances

Key Advantages

- **Improved NMR sensitivity**
 - Reduced line widths, lengthened transverse relaxation times, and increased intensities of resonances in NMR spectroscopy
 - Sensitivity can be increased potentially by a factor of 20,000
- **Reduced paramagnetic broadening:** electron and nuclei coupling followed by electron

decoupling can reduce paramagnetic broadening

- **Easily modulate microwave frequency output:** gyrotron output is controlled by the NMR spectrometer interface for synchronized microwave and RF irradiation
- **Faster acquisition times:** EPR-NMR experiments can potentially be 200 million times faster
- **Enhanced precision:** device can potentially monitor the dephasing of rotational Hahn-echoes to enhance precision of electron-nuclear distance measurements

Publications

- [Hoff DEM, Albert BJ, Saliba EP, et al. Frequency swept microwaves for hyperfine decoupling and time domain dynamic nuclear polarization. *Solid State Nucl Magn Reson.* 2015;72:79-89.](#)
- [Saliba EP, Sesti EL, Scott FJ, et al. Electron Decoupling with Dynamic Nuclear Polarization in Rotating Solids. *J Am Chem Soc.* 2017;139\(18\):6310-6313.](#)

Patents – Patented

- [Integrated EPR NMR with frequency agile gyrotron](#) (US Patent No. 10,113,984; additional application pending)

Website – [Alexander Barnes Lab Website](#)