Research Highlight #157

Extruded dielectric sample tubes of complex cross section for EPR signal enhancement of aqueous samples

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Introduction: This highlighted work[1] builds on our previous published work[2] and that of Sidabras, Mett, and Hyde,[3] where multiple flat aqueous sample cells placed perpendicular to electric fields in microwave cavities were used to reduce the radio frequency losses and increase the EPR signal. In this study, we present three novel sample holders for loop-gap resonators (LGRs) and cylindrical cavity geometries.

Results: By careful observation of the electric field profiles, the design of custom sample holder geometries has led to a reduction in the electric field intensities within a sample, yielding significant experimental EPR signal gains. The goal set in the design of the sample holders was to maximize EPR saturable signal intensity in a two-loop–one-gap LGR with a 1.2 mm diameter sample loop. The design takes into account breaking up the tangential electric field (Type I), minimizing edge effects (Type III), and maximizing the perpendicular surface to the electric field (Type II). Two of the designs for sample holders were commissioned and built using polytetrafluoroethylene (PTFE) extrusion techniques: a 1 mm O.D. capillary with a septum down the middle, named DoubleDee tubing, and a 3.5 mm O.D. star-shaped sample holder, named AquaStar tubing. Simulations and experimental results at X-band show that the EPR signal intensity increases by factors of 1.43 and 3.87 for the DoubleDee and AquaStar, respectively, over the current TPX 0.9 mm O.D. sample tube in a two-loop–one-gap LGR. Finite-element simulations were performed with ANSYS High Frequency Structure Simulator (HFSS, Version 17.0; Canonsburg, PA) at 9.5 GHz (X-band). Experimental EPR sensitivity values were recorded at peak values of $P_{1/2}$, which produced a constant H_1 (a saturable sample); then, the ratio of these intensities was calculated and compared with finite-element ratios. All tests were performed on a standard Bruker E500 X-band spectrometer. Additionally, by combining the insights gained from the constructed sample holders and finite-element solutions, a third multi-lumen sample holder for a cylindrical TE011 cavity is optimized, named AquaSun, where simulations result in an EPR signal intensity increase by a factor of 8.2 over a standard 1 mm capillary. The three sample holder geometries are illustrated in Figure 1.

Implications: The DoubleDee and the AquaStar designs were built and tested, showing factors of 1.4–3.9x improvement over conventional geometries. A factor of nearly 4 is a fantastic improvement and uniquely enabled experiments to be carried out in the Klug lab that required low concentrations of protein for protein-ligand binding studies that otherwise would not have been possible. In addition, further innovations, as proposed by the AquaSun design, illustrate how flat geometries in a perpendicular electric field can produce an eightfold increase in the EPR signal. This is a very exciting development that will enable an array of biological studies to be carried out that previously were not possible by EPR. Plus, even further improvements beyond this may result when considering the concept of a line dipole, which is well known in the field of electricity and magnetism but apparently not yet in EPR sample tube design.

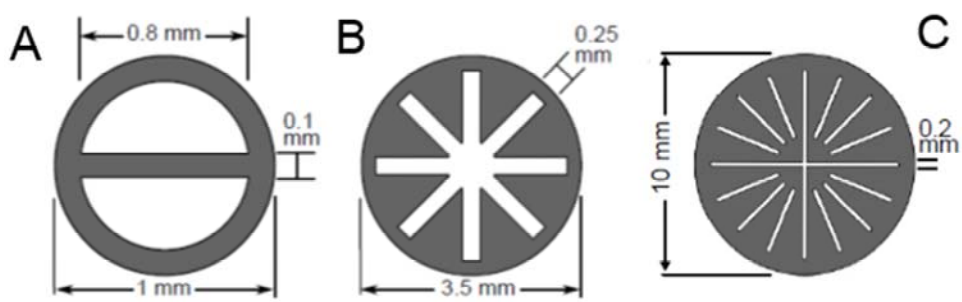

Figure 1. Illustration of the three new sample holder geometries: A) DoubleDee, B) AquaStar, and C) AquaSun

[1] Sidabras JW, Mett RR, Hyde JS. (in submission) Extruded Dielectric Sample Tubes of Complex Cross Section for EPR Signal Enhancement of Aqueous Samples. [submitted to J Magn Reson]
[2] Mett RR, Hyde JS. (2003) Aqueous Flat Cells Perpendicular to the Electric Field for Use in Electron Paramagnetic Resonance Spectroscopy. J Magn Reson. 165:137-152.
[3] Sidabras JW, Mett RR, Hyde JS. (2005) Aqueous Flat-Cells Perpendicular to the Electric Field for Use in Electron Paramagnetic Resonance Spectroscopy. II: Design. J Magn Reson. 172:333-341.