# Low-E Magic Angle Spinning Probe for Biological Solid State NMR at 750 MHz

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## Introduction

Crossed-coil NMR probes have been a useful tool for reducing sample heating for biological magic angle spinning (MAS) NMR. In a crossed-coil probe, the higher frequency $^1$H field, which is the primary source of sample heating, is produced by a separate low-inductance resonator. Because a smaller driving voltage is required, the electric field across the sample and the resultant heating is reduced. In a commercial implementation, the $^1$H coil is placed inside the low-frequency solenoid [1]. Last year we successfully developed a MAS probe based on the alternative approach of placing the $^1$H resonator outside the solenoid (Figure 1). Because the solenoid can then be smaller and better separated from the $^1$H resonator, we expect that this approach may give improved sensitivity and allow higher $B_1$ fields without arcing between the two coils. This dual resonator approach, named "Low-E," was originally developed to reduce heating in samples of mechanically aligned membranes [2], where it allowed the use of larger samples that were required to obtain sufficient NMR signal. The study of inherently dilute systems, such as proteins in lipid bilayers, via MAS techniques also requires large sample volumes at high field to obtain spectra with sufficient signal to noise ratio under physiologically relevant conditions. This year we tested the probe under a variety of conditions and fine tuned design elements, particularly to improve proton decoupling fields. The probe currently outperforms commercial probes we have tested as well as results published for other designs. In particular, due to the larger volume of the rotor, we are able to examine structure in membrane associated peptides at physiologically relevant concentrations.

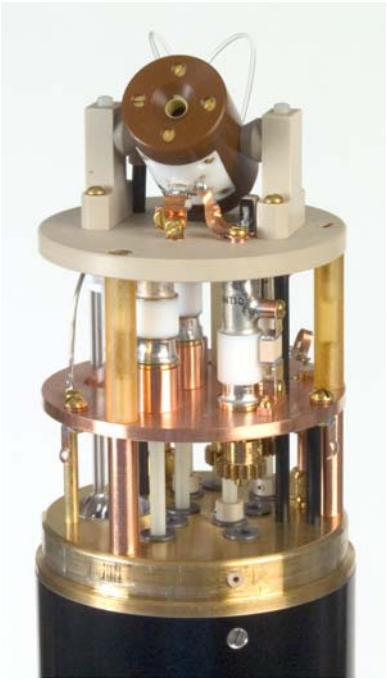

Figure 1. Low E probe showing stator and tuning network.

## Methods

The probe was thoroughly characterized in order to determine maximum achievable $B_1$ fields, homogeneity of the $B_1$ fields, RF efficiencies, RF heating, frictional heating under MAS, spectral linewidths, and signal/noise on standard compounds. Working with Revolution NMR (Ft. Collins, CO), we were able to obtain rotors which have thinner walls and spin stably at 12 kHz, allowing us to pack more sample into the rotors. To increase robustness on the proton channel, capacitors were added in parallel to reduce voltages at individual points. This caused a small decrease in RF efficiency but allows us to operate for long periods of time with high proton decoupling.

## Results and Discussion

Under normal operating conditions, a $^1$H $B_1$ field of 93 kHz and homogeneity (810/90) of 93% can be obtained. With a higher power amplifier, we should be able to reach 110 kHz based on bench measurements. $^{13}$C $B_1$ fields of > 71 kHz homogeneity (810/90) of 80% are obtainable with a full rotor . Measurements on a more restricted samples (9-10 mm in length or 43-48 μL in volume) achieve well over 95% homogeneity. The power handling characteristics, $B_1$ fields, and homogeneities make this an ideal probe for the full range of solid state NMR experiments, including sequences which use extended periods of continuous RF pulsing on both channels. Details of design considerations as will as some initial results was recently published [3]. A more detailed manuscript demonstrating the probe's capabilities with a variety of samples and pulse sequences is nearing completion.

## References

[1] Doty, F.D., *et al*., J. Magn. Reson. **182** (2006) 239-253.
[2] Gor'kov, P.L., *et al*., J. Magn. Reson. **185** (2007) 77-93.
[3] McNeill, S.A., Gor'kov, P.L., Struppe, J., Brey, W.W., and Long, J.R., Magn. Reson. Chem. **45**(2007) S209–S220.