New Apparatus for Double Nuclear Magnetic Resonance Experiments

Introduction

Nuclear Magnetic Resonance (NMR) is a phenomena utilized in medicine for commonly known magnetic resonance imaging (MRI). Similarly, NMR spectroscopy is a widely used technique in chemistry that gives an insight into structure of molecules, properties of materials, and dynamics of molecular systems. Previous attempts to do solid state Signal spectroscopy have been thwarted by the spectrum broadening that occurs because the molecules are close together, tightly bound, and thus interact easily. We wish to show that using a combination of dynamic nuclear polarization (DNP) and cross polarization of nuclei can result in successful static solid state experiments, as well as design a probe that will be effective in doing these kinds of experiments.





We wish to create a specially designed NMR probe that will give us the ability to do dynamic nuclear polarization on solid state systems, double resonance experiments, and single resonance NMR experiments on a variety of nuclei.

NMR spectroscopy works by putting a sample



that contains the nuclei that you want to detect in a strong, static magnetic field. This polarizes the nuclear spins (most of them aligning with the magnetic field). After the spins are polarized, we run a pulse sequence through an inductor coil, which tips the spins perpendicular to the original field into the plane where we can detect them. When they are pulsed down, they precess and emit detectable signal in the form of electromagnetic radiation. The sinusoidal signal versus time graph can be fourier transformed to give the NMR spectrum for that particular sample like the example proton spectrum to the left for ethanol. Each signal peak corresponds to the location of protons in the structure of ethanol.





First, let us take an unknown surface like some kind of polymer or surface containing carbon that we wish to classify. We then submerge our sample in a radical solution, which contains free electrons and protons.

Methods

Next, we irradiate our sample with microwaves to polarize the spins of the electrons.

The electrons in the solution can interact with the spins of the protons, thus transfering polarization from elctron to protons.

Finally, we can run a sequence of pulses through the inductor coil that surrounds our sample. This gives us the nuclei cross polarization from the protons in the solution to the carbons in the surface. When the carbon spins relax, they will emit detectable electromagnetic radiation that we can fourier transform to get interpretable data.



Future Plans

Since the probe is not finished yet, we do not have data from it. However previous DNP experiments have shown that when irradiating the sample with microwaves, you can see a drastic increase in signal (possibly orders of magnitude larger). For instance, the aluminum spectrum, which can be hard to get signal from, has been amplified to 3 times the original signal.

Microwaves off

400

Aluminum NMR spectrum

²⁷Al (ppm)

Microwaves on

tor poper los

-400

Once the coil is done, we plan to run double resonance and cross polarization experiments using adamantane (an NMR standard) to confirm that it is operational and as high preformance as we have designed it to be. In addition to this, we shall run single resonance experiments on several different nuclei including but not limited to Chlorine 35, Flourine 19, Boron 5, Lithium 7, Hydrogen 1, and Carbon 13 to demonstrate the versatility of our design.