Summer 2019 Final Report: Designing NMR Devices

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Contents

1 Introduction

This report outlines the work that I have conducted over the summer of 2019. This report should serve as a guide to anyone looking to continue the work that I have conducted. This report will be divided into two, unequal sections; one will outline the brief work with the 3 magnet array while the second will outline the design and construction of smaller, "dunkable" probes made using small disc magnets. With this document, one should be able to quickly replicate the results achieved this summer and continue this work.

2 3 Magnet Array with Constant Gradient along B_0

Simulations of a large, three magnet array based off the

3 Dunkable NMR Devices

Figure 1: A completed device. The exterior is coated in epoxy. The coaxial cable connecting the device to the external tuning and matching network can be seen on the right.

A very small, cheap, robust, and submersible NMR device has tremendous potential. One such application for these devices is the speciation of solutions. Many relaxation-based measurements can be made using these devices, including: CPMG, Saturation recovery, $T_1 - T_2$ mapping, and diffusion. Each of these measurement types will give insight into the composition of the substance being measured.

3.1 Design and Construction

The final device consists of a small box constructed using double clad PC board with two pairs of magnets on either side. Between the magnets is a rectangular cutout, within which is a rectangular RF probe. The PC board box is filled with epoxy to give structural rigidity and ensure that no solution comes into contact with the electronics within the box.

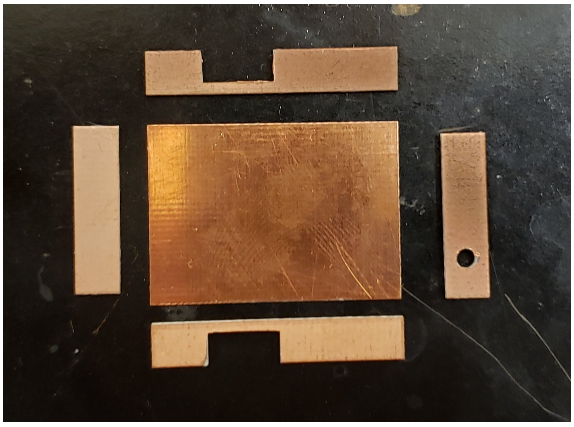

Figure 2: The circuit board pieces used to fabricate the box.

The construction of these small NMR devices is quite simple:

1. A small box with rectangular cutouts is constructed using PC board.
2. The RF coil is wound around a 3D printed former and then inserted into the rectangular cutout of the box
3. A 2.5mm outer diameter coaxial cable is connected to the RF coil through a hole drilled in the end of the box.
4. The box is filled with Loctite EA M-31CL epoxy and allowed to cure.
5. The former for the coil is removed using a heat gun.
6. The box is then completely coated in epoxy, protecting the external surfaces from corrosion.

3.1.1 Outer Box

The first consideration for the design of the device is the dimensions and the material of the outer box which will house the RF coil. This box should be fabricated using unperforated, double-clad (copper on both sides) circuit boards available from sources such as Digi-Key or McMaster Carr. The width and length of the box are quite flexible however, the thickness of the box is not; it is determined by the strength of the permanent magnets used.

N35 grade Disc magnets with a diameter of 25mm (1”) and a thickness of 3.25mm (1/8”) (available from Lee Valley) were used to create the B_0 field. The separation of the magnets is crucial in generating a homogeneous field. A good simulation of the magnetic field between two disc magnets separated by some amount is available on K&J Magnetics’ website, https://www.kjmagnetics.com/gap.calculator.asp.

For a pair of N35 disc magnets with a gap of 9mm (which gives optimal field homogeneity), the resonant frequency is 8 MHz. This field is too low for any rapid measurements. The solution is to use a pair of magnets on each side of the outer box. With 4 magnets, two on either side, the resonant frequency will be 16 MHz. However, the increased magnetic field strength requires that the gap between the magnets be narrowed to 7mm, according to K&J Magnetics' simulation software. This is configuration with 4 magnets in two pairs with a 7mm gap in the suggested configuration.

With the magnet gap decided, the next consideration is the thickness of the PC board used in the construction of the box. The outer wall should be constructed using 0.8mm (1/32") double clad PC board. This additional thickness allows for a simpler construction. The top and bottom of the box, the surfaces that the magnets will be pressed against should be constructed using 0.4mm double clad PC board. This very thin board allows for the RF coil to occupy as much volume as possible. This thickness of PC board is not usually readily available from McMaster Carr, however, Digi-Key sells pre-sensitized 0.4mm PC board (https://www.digikey.ca/product-detail/en/mg-chemicals/698/473-1022-ND/559716). The pre-sensitized layers can be removed and the board can be used.

The final outer dimensions of the box are 25 mm $\times$ 35 mm $\times$ 7 mm. The dimensions of the rectangular cutout for the RF probe are 10 mm $\times$ 3 mm and the diameter of the hole for the coax cable is 2.5 mm. Once the pieces of PC board have been cut (have the machinists do this) the box must be soldered together. This ensures that the pieces are securely connected both mechanically and electrically.

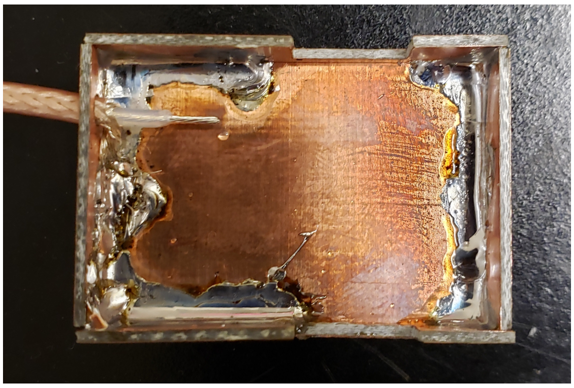

Figure 3: Box soldered together with the coaxial cable attached.

More details of the final dimensions can be found in the 3D model of the box.

3.1.2 Probe Construction

The next consideration is the RF probe itself. The probe consists of two components: the coil and the tuning and matching network.

The geometry of the RF coil is an important consideration. The probe must fit within the 7mm gap between the magnets while providing sufficient measurement volume for sample. A rectangular coil is used as the B_1 field generated from it is still quite homogeneous but, unlike a cylindrical coil, it has a much larger sample volume. This probe must be wound around a square former. This former can either be hollow and left in place once the epoxy has cured or it can be removed. While removal is more difficult, it allows the sample volume of the probe to be much larger hence, it is the suggested option. The probe former was 3D printed using PLA plastic and wrapped in teflon tape before 0.8 mm magnet wire is wrapped around it. The teflon tape ensures that the epoxy will not stick directly to the PLA plastic. Approximately 20-25 turns of wire are possible.

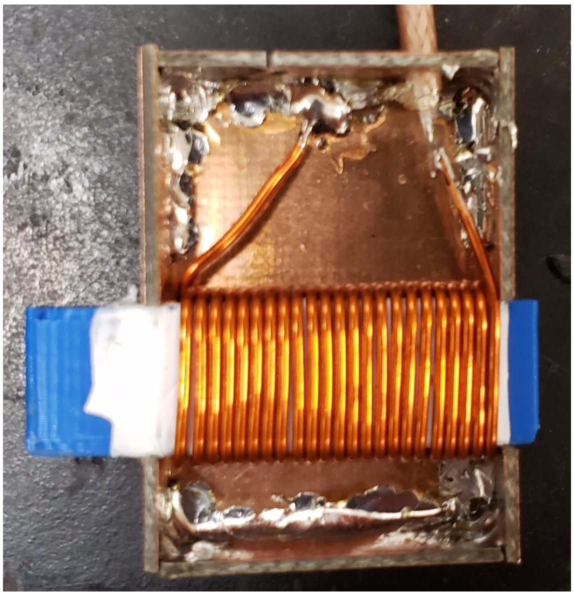

Figure 4: The with the probe and former connected.

Once the coil is wound around the former, both can be placed into the rectangular cutouts of the box. Next, the coaxial cable's core is connected to one end of the coil while the shielding around the core is soldered to the inside of the box. The opposite end of the RF coil is also soldered to the inside of the box. This grounds the interior of the box, creating a faraday cage and shielding the probe from external RF signals. Be cautious when soldering the RF probe to anything as heating it too much will melt the PLA former, deforming it.

Next, the opposite end of the coaxial cable is attached to a tuning and matching network with a variable tuning capacitor. The input of the tuning and matching circuit is then connected to a BNC cable.

Once the probe is in place, the box is filled with Loctite EA M-31CL epoxy. This ensures that the box will remain rigid once that magnets are in place and that no liquid is able to get inside the box; the epoxy should be left for 24 hours to fully cure.

Finally, the exterior of the box is coated with epoxy, preventing corrosion and keeping the sample free from contaminate. This will also prevent the magnets from moving and distorting the field within the sample volume. Once this epoxy has been left to cure for 24 hours, the device can be submerged in liquid and measurements can be done.

3.2 Hardware/Software Configuration

Once the probe, box and electronics have been finished, testing the probe can begin. The probe is first connected directly to the transcoupler/duplexer to the output labeled "Probe". The connector labeled "Tx" is connected to "RF out" on the amplifier and "Rx" is connected to the pre-amplifier which is in turn, connected to the "Rx in" on the console. An appropriate quarter-lambda cable must be connected across the connectors marked "Quarterwave". Note that the 15 MHz quarterwave cable is suitable for frequencies between 13-17 MHz.

3.2.1 Pulse Parameters

Once the device is connected to the amplifier, the correct RF pulse must be sent to the probe in order to get signal. This pulse has 3 main parameters: amplitude, frequency and duration.

The frequency of the RF pulse sent to the probe should be equal to the frequency that the probe is tuned to. The tuning frequency is determined by the resonant frequency of hydrogen in the B_0 field and hence the strength of the B_0 field. This can be measured using the thin-tipped gaussmeter.

The amplitude and duration of the RF pulses both control the tip angle of the magnetization by:

$$\theta_t = 2\pi B_1 t_p \tag{1}$$

Here, t_p is the pulse duration and B_1 is the magnitude of the magnetic field generated by the RF pulse which is proportional to the amplitude of the pulse. The pulse amplitude can be changed by increasing the attenuation of the signal being sent from the console to the amplifier. It can also be done in software in the apollo.ini file that can be found C:/TNMR /config. Two variables in apollo.ini are important: *Attenuator* and *Attenuator2*. The *Attenuator* variable is the attenuation of the pulse when the "AHOP" line in TNMR pulse program is not activated. When the "AHOP" line is activated, the attenuation switches to *Attenuator2*. This is done when performing CPMG measurements where the first pulse should be a 90° pulse and the following should be 180° pulses. For the first pulse, the AHOP line is turned on and *Attenuator2* is set to 6dB fewer than the *Attenuator* variable, halving the pulse amplitude of the first pulse, making it only 90° rather than a 180° pulse.

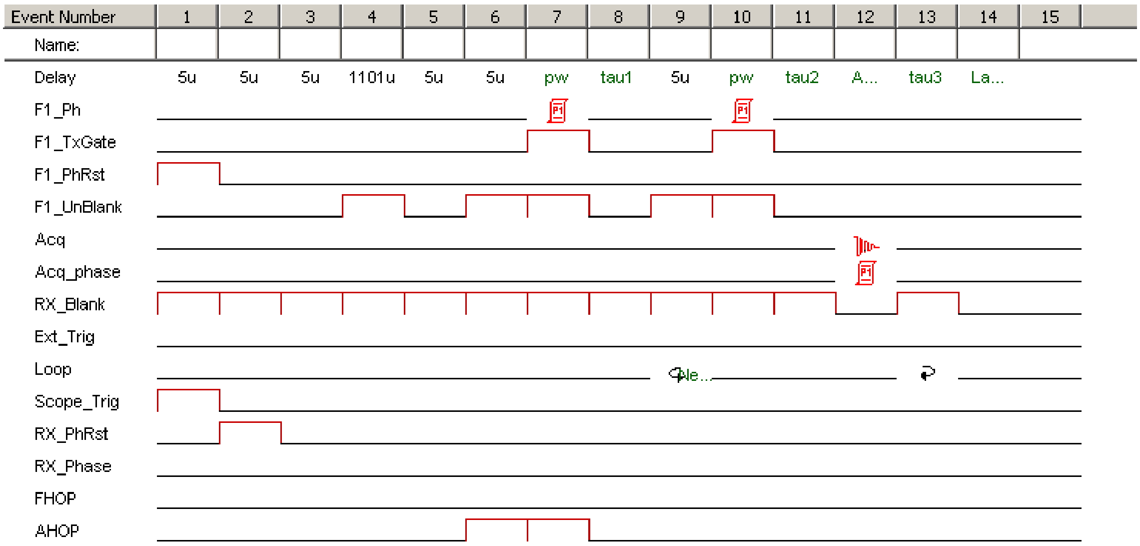

Figure 5: A CPMG sequence in TNMR. Note the AHOP line activating when the 90° pulse is applied and deactivating when the 180° pulse is applied. This changes the transmit attenuation from 33 dB (90° pulse) to 26 dB (180° pulse)

While changing the pulse amplitude changes very little other than tip angle, the pulse duration does. As the pulse duration is increased, the frequency bandwidth (fourier transform) of the pulse becomes more narrow meaning that, in an inhomogeneous field, the slice thickness will become smaller and fewer spins will be excited. This effect may or may not be desired but it should be considered when choosing a pulse width. A reasonable pulse width is between 4 and 12 microseconds. pulse width can be controlled using the "pw" variable in TNMR.

It takes quite some time to dial-in the 90° pulse parameters exactly but beginning with 33 dB of attenuation for a 90° pulse and 26 dB of attenuation for a 180° pulse and a 90 μs and varying both slowly should be a good starting point.

3.3 Measurement Types

3.3.1 T_2 Measurements

Measuring the T_2 of the sample can be achieved using a simple CMPG measurement. Note however that with an inhomogeneous field, the T_2 observed will be dependent on the echo time, t_e, due to diffusive effects. The intrinsic T_2 can be determined using a technique similar to the methods used for determining the diffusion coefficient.

3.3.2 T_1 Measurements

A T_1 measurement can be obtained using a saturation recovery measurement. Here, sequential CPMG measurements are made and the last delay is varied. Ensure that after the CPMG is completed that the magnetization is completely destroyed and that no echoes are observed. By waiting longer last delays, the saturated magnetization will have more time to relax back to equilibrium and, when the subsequent

CPMG begins, there will be more magnetization along the z-axis when the measurement begins. The magnitude of the second or third peak of each CPMG is then used as a gauge of net magnetization for the given last delay. T_1 can be determined by fitting M_0 vs $T_{\text{last delay}}$ to:

$$M_0 = 1 - e^{-t_{\text{last delay}}/T_1} \tag{2}$$

This data can also be processed to generate a $T_1 - T_2$ map using the software provided by Schlumberger.

3.3.3 Diffusion Measurements

In an inhomogeneous field, the effective T_2 that is observed in a CPMG measurement is composed of multiple components:

$$\frac{1}{T_{2,\text{ effective}}} = \frac{1}{T_{2,\text{ intrinsic}}} + \frac{1}{T_{2,\text{ diffusion}}} \tag{3}$$

where the diffusion contribution is:

$$\frac{1}{T_{2,\text{ diffusion}}} = \frac{D(\gamma G t_e)^2}{12} \tag{4}$$

Here, D is the diffusion coefficient of the substance, t_e is the echo time, and G is the effective gradient of B_0. By repeating CPMG measurements and varying t_e and observing the effect on T_2, the diffusion coefficient can be determined by fitting $\frac{1}{T_2}$ vs t_e^2 linearly. The slope will depend on the diffusion coefficient and the gradient and the intercept will depend on the intrinsic T_2 of the sample.