NMR Measurements

for Solid Polarized Targets

J. Maxwell

for the Jefferson Lab Target Group

Jefferson Lab

23rd International Spin Symposium
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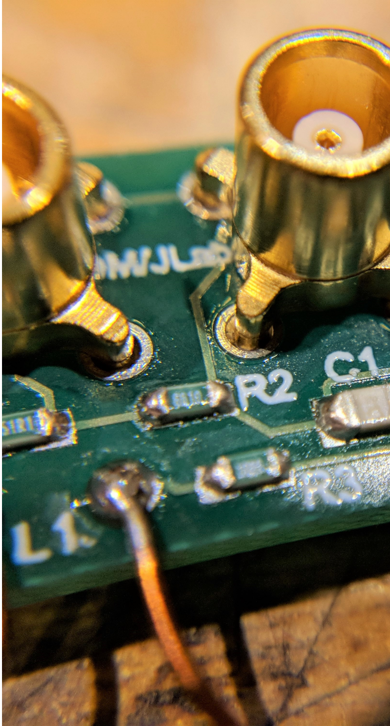
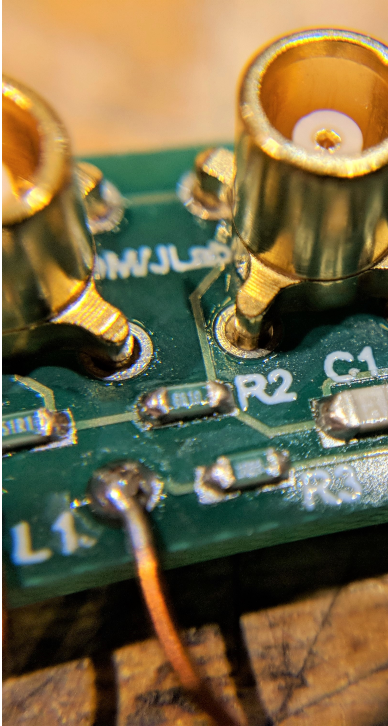
September 10th, 2018



Outline

1. NMR and JLab Solid Targets
   - Introduction
   - New Challenges
2. Progress
   - Cold Tank Circuit NMR
   - 77 K NMR Test Bed
3. Looking Forward
   - Improving Hardware
   - Next Steps



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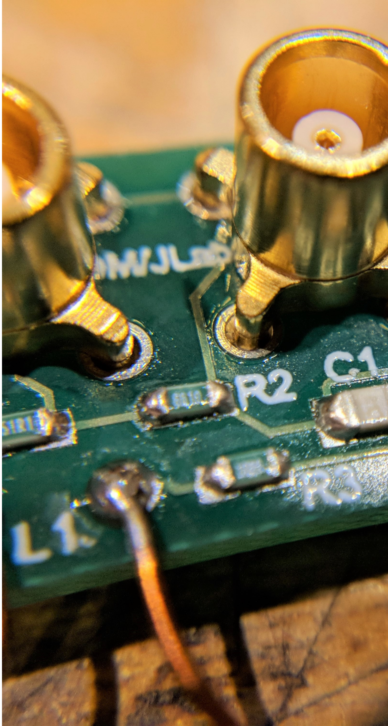
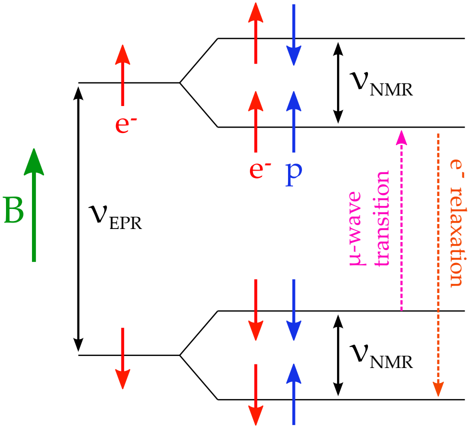


Dynamic Nuclear Polarization (Solid Effect)

- Take advantage of $e{-}p$ spin coupling
- Induce "forbidden" transitions with μ-waves to match energy gaps
  - $\nu_{\mu+} = \nu_{\text{EPR}} - \nu_{\text{NMR}}$
- Relaxation times key
  - $e \approx$ milliseconds
  - $p \approx$ 10s of minutes
- Choose polarity without changing magnetic field
  - $\nu_{\mu-} = \nu_{\text{EPR}} + \nu_{\text{NMR}}$



Measuring Polarization in Polarized Solids

- NMR in field B_0 at ω_0, apply RF field to material
- Coil of L_0 perpendicular to B_0 to induce spin flip
- Material polarization modifies the inductance of a coupled coil, with filling factor η:

$$L(\omega) = L_0(1 + 4\pi\eta\chi(\omega))$$

- Polarized nuclei give the target material a complex susceptibility, a function of applied frequency (ω):

$$\chi(\omega) = \chi'(\omega) + i\chi''(\omega) \quad \text{and} \quad P = K\int_0^\infty \chi''(\omega)d\omega$$

- $\chi(\omega)$ is non-zero only close to the Larmor frequency ω_0

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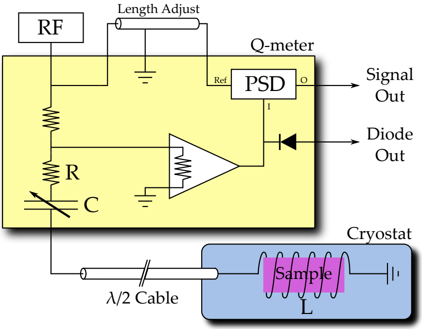
Continuous-wave NMR Electronics: Q-meter

- Choose L, C of circuit so that $\omega_0 = 1/\sqrt{LC}$
- Complex impedance of circuit $\sim i\omega L_0 \Delta L(\omega)$
- Compare signal to reference with BRM to get real portion, must match phase

- Away from ω_0, coil impedance has reactive components, makes Q-curve
- Sweep frequency around ω_0 to integrate in ω
  - For 5 T protons, 213 MHz $\pm$ 400 kHz



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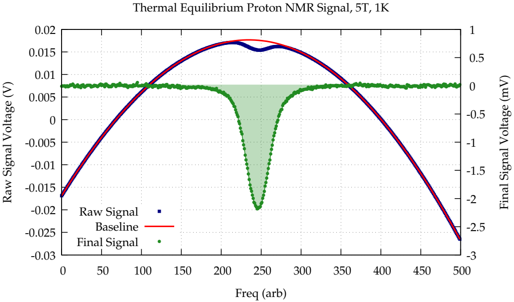
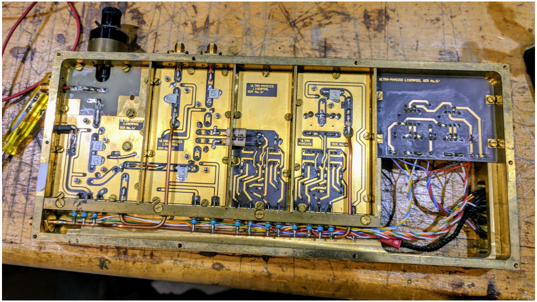


- Liverpool Q-meter used for decades. Carefully designed for excellent RF performance for DNP applications.
  - G.R. Court, NIM A324 (1993)



Traditional Tuning for Continuous-wave NMR

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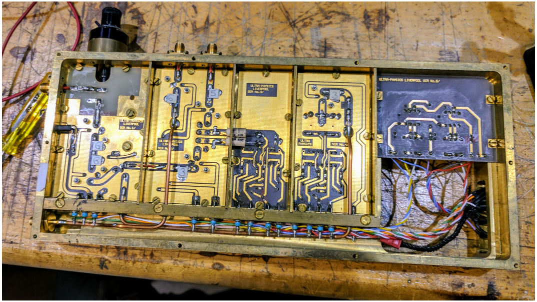

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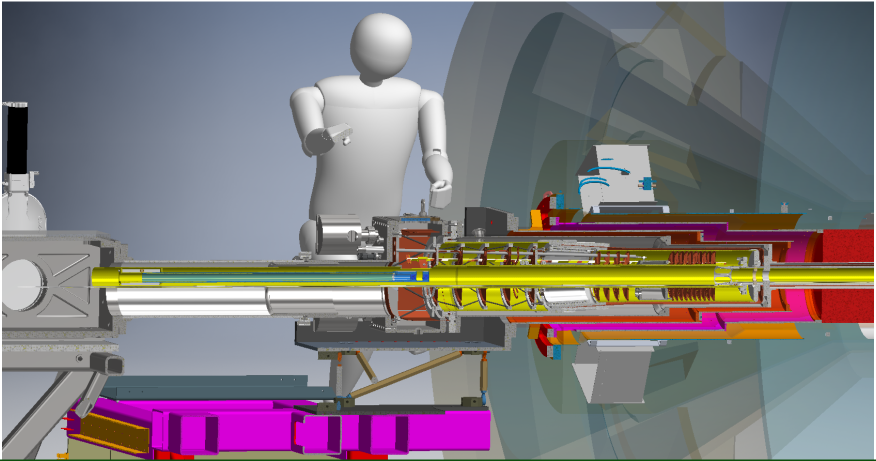
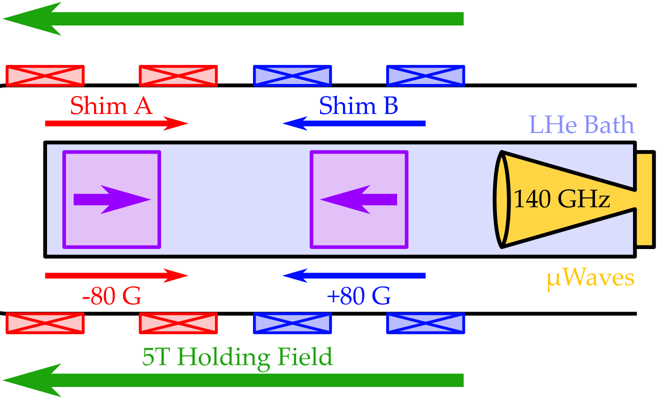
Hall B's CLAS12 Polarized Target

- Requires external NMR coils with polarized protons
- Requires shim magnets, possibility of 2 cells

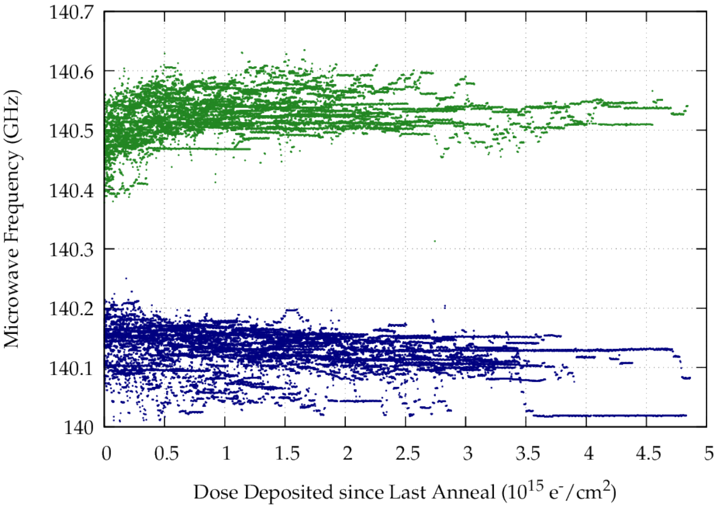


Double Cells with Opposing Polarization

- Access $\pm$ polarization with $\nu_{\mu\pm} = \nu_{\text{EPR}} \pm \nu_{\text{NMR}}$ in a single holding field (COMPASS)
  - OR change local fields so that $\nu_{\mu+} = \nu_{\mu-}$
- Microwave freq static, must change shim fields, NMR tune



Excitation Frequency Change with Radiation Dose



JLab NMR Wishlist

- Cold circuit NMR
  - Noise reduction, Non-resonant cable circuit
- Remote capacitor tuning
  - Accommodate 2 opposing cells, Synchronous Tuning
- Electronic phase tuning
- Optimize external NMR coil design
- Address aging of Liverpool Q-meter

Most not revolutionary, but many previous innovations combined for the first time

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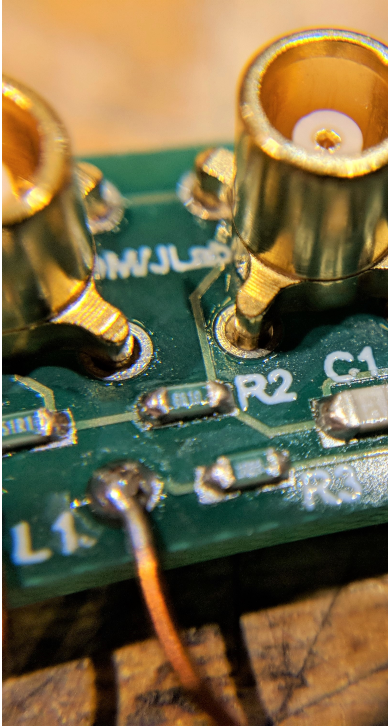
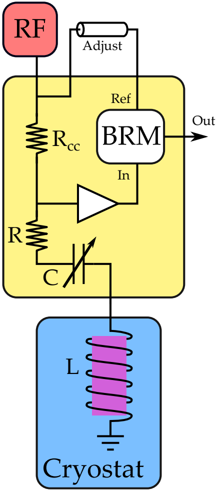


Move the tank circuit inside the cryostat?

- Traditionally, R and C inside Q-meter, L in the cryostat
- Moving R and C into the cryostat, $\lambda/2$ no longer separates L
    - Q-curve shallower
    - Thermal noise reduction
    - Requires components that can handle cold, microwaves, radiation
- How can we tune a capacitor inside the cryostat?
- Varactor diodes to vary capacitance via bias voltage



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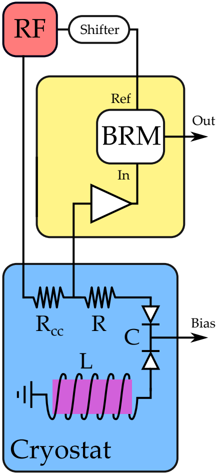

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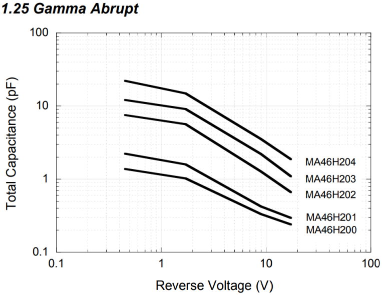
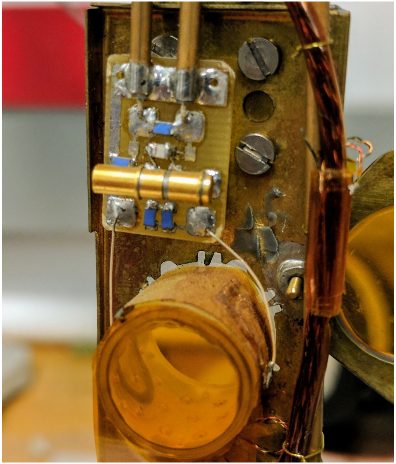


Cold NMR Boards

- Cold NMR method most recently used at JLab for EG1-DVCS deuteron
  - Trim cap and high C for 5 T D: 32.7 MHz
  - Tune performed warm, anticipating the change with temperature
- Introducing GaAs varactor diodes
  - Tested at 213 MHz to 3.2 K
  - Minimal changes seen at high B, low T
- Miniturized, connectorized



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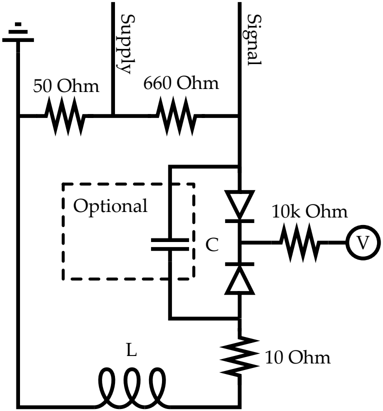

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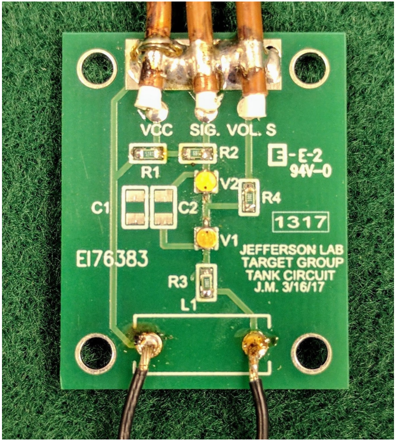

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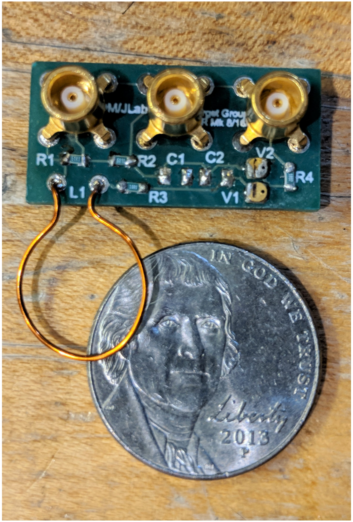
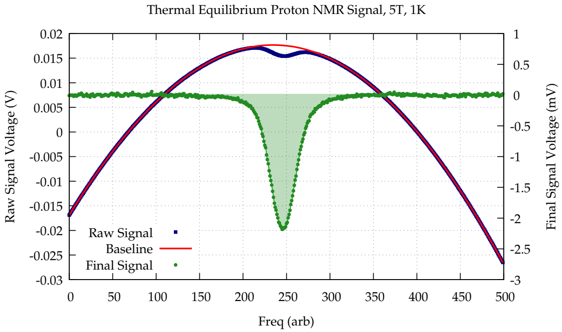


Testing NMR Methods at 77 K

- Traditionally test NMR at 5 T & 1.2 K
- Expensive, and no fridge currently available.
- At 77 K, we expect polarization to be over 50 times lower
- LN bath with 5 T warm bore solenoid
- Still testing at 213 MHz, but with **small** signals
- Embed coils in epoxy for protons



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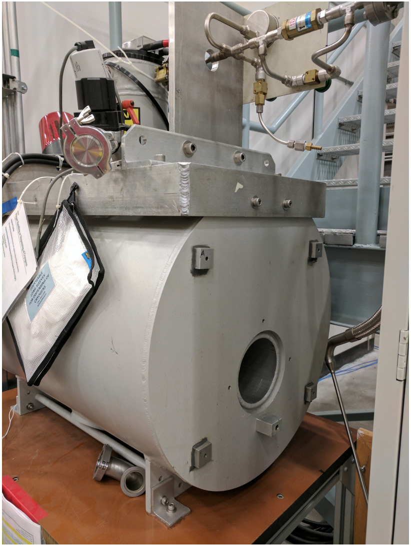

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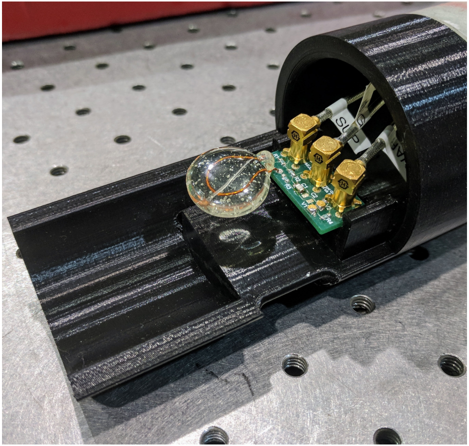

Testing Shim Coils

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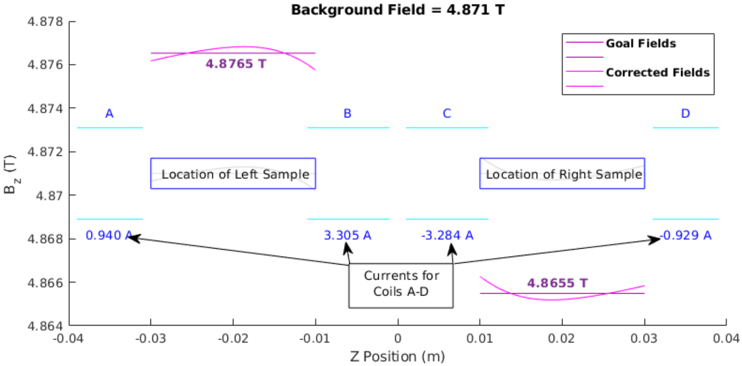

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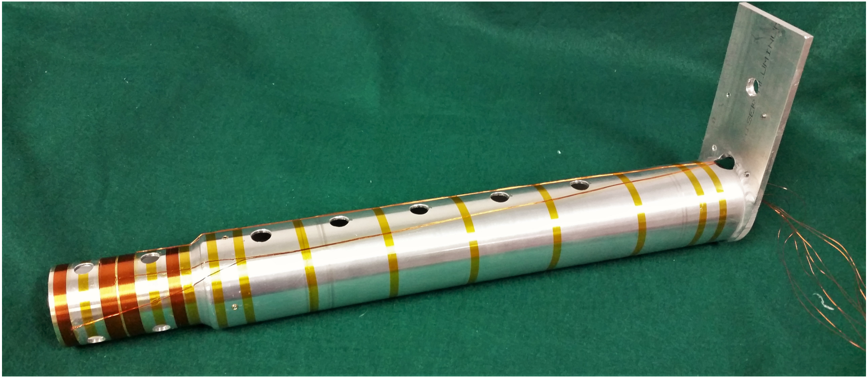

Enhancing Polarization at 77 K, 5 T?

- Microwaves aimed through the kapton windows in 77 K bath
- EIO tube in test has center frequency is 136 GHz, so field lowered to 4.9 T, NMR at 207 MHz
- Dope epoxy with TEMPO
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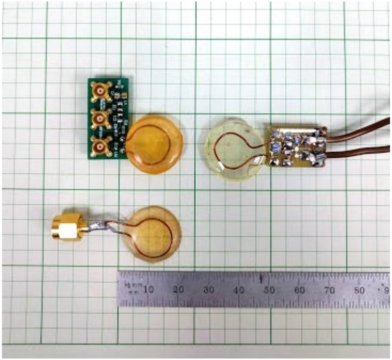
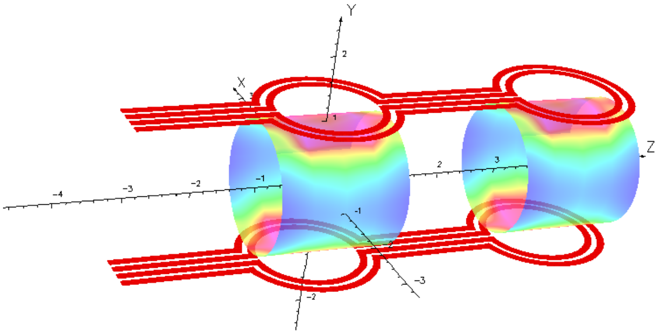


External NMR Coils

- Coils must be outside target material: easy for D, tricky for P
- Need to keep L low to keep C feasible (above 10 pF)
- Tested a number of coil geometries, covering both cells



V.Lagerquist

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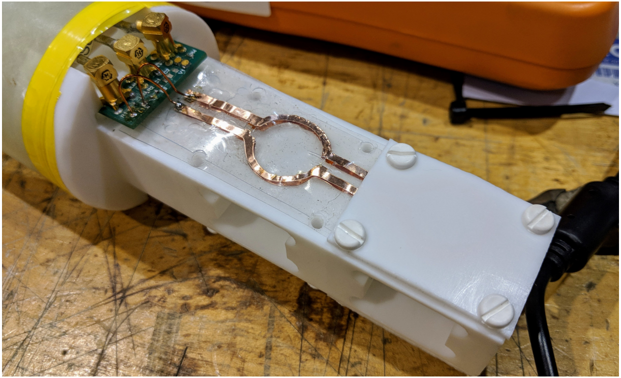

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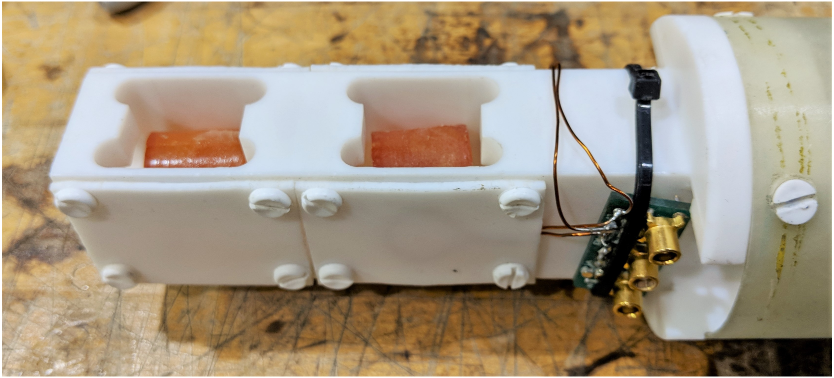

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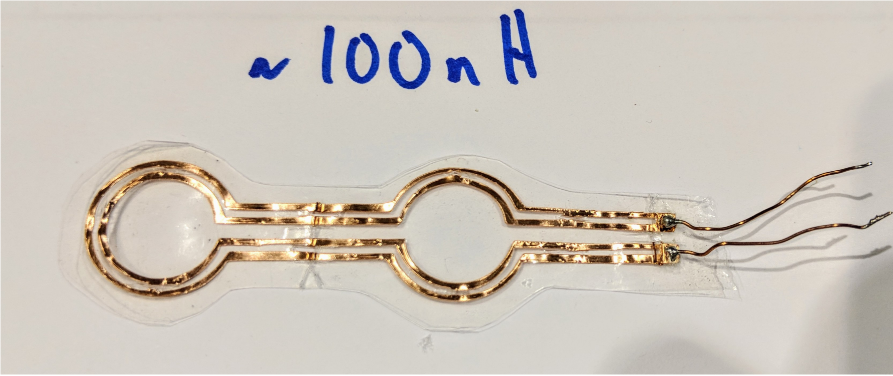
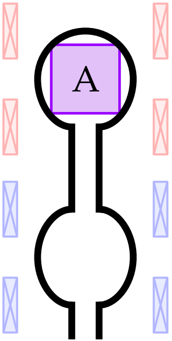
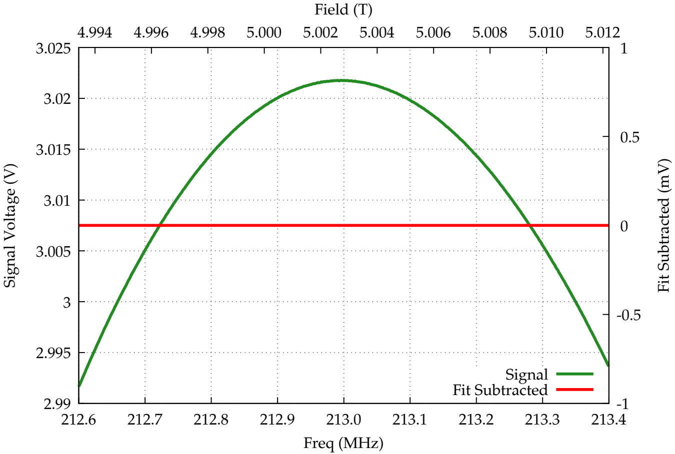


NMR Signals with a Single Coil, Double Cells





Baseline NMR Q-curve

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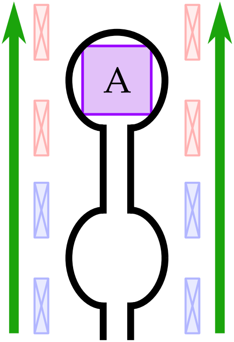

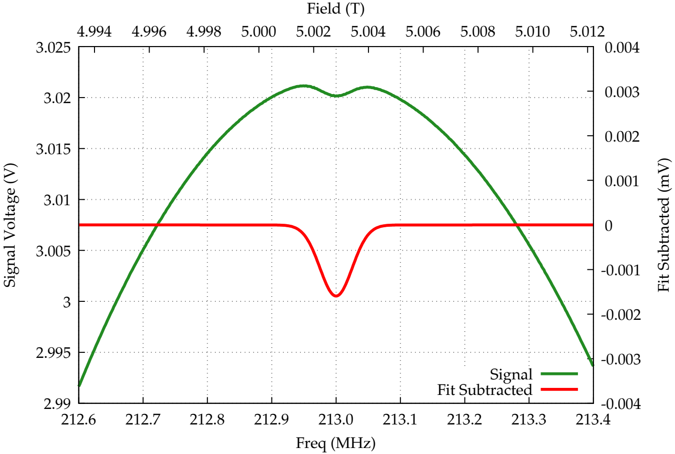

Polarized Proton Signal

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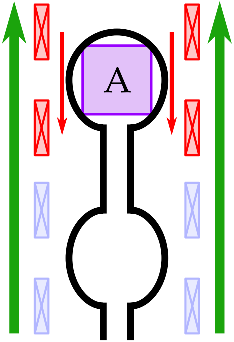

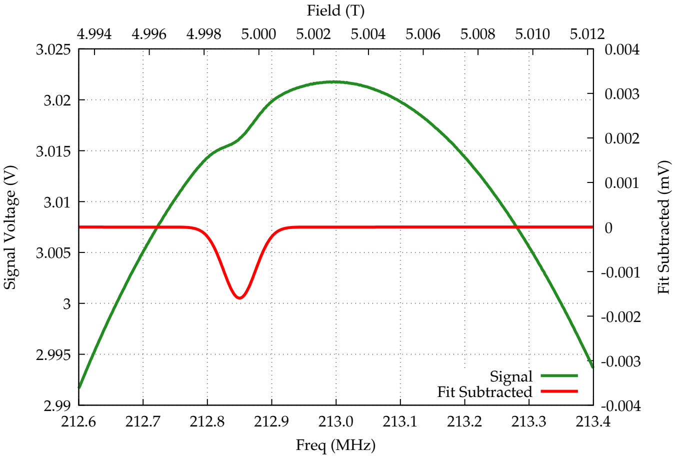

Shim A decreases field

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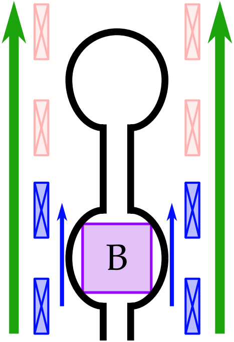

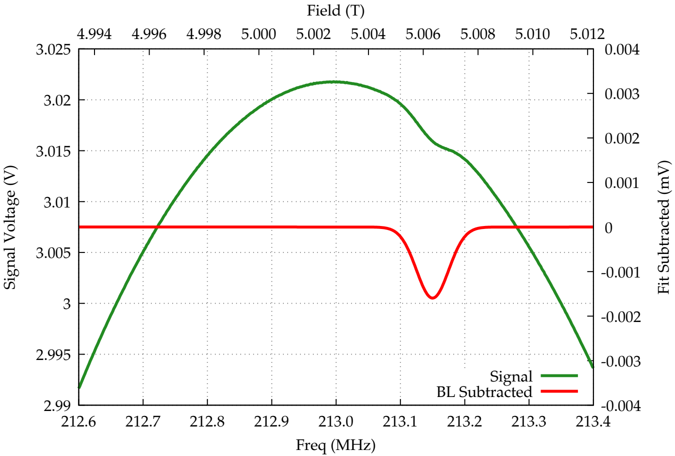

Shim B increases field

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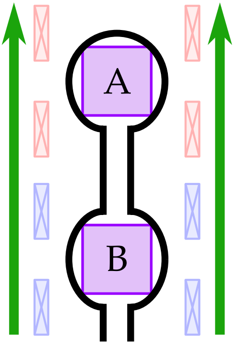

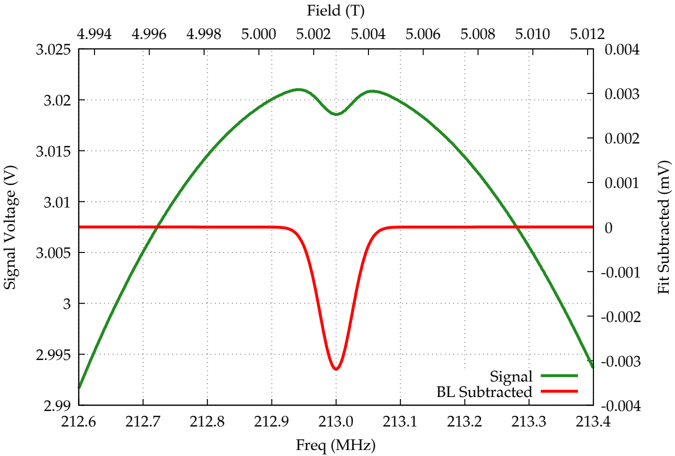

2 cells with shims off

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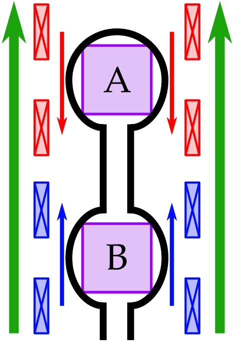

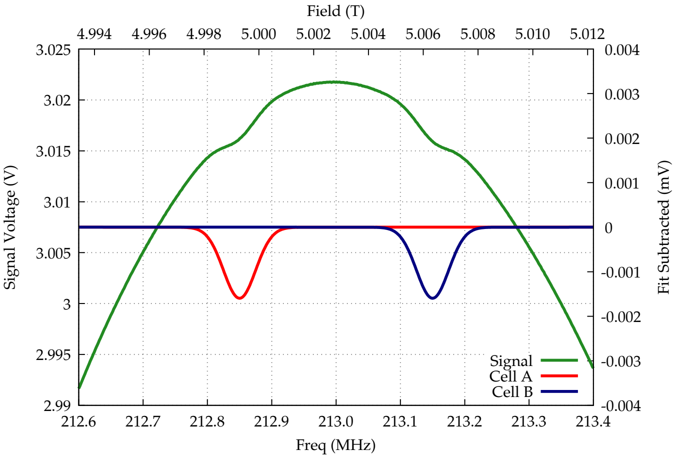

2 cell with shims separating signals from each cell

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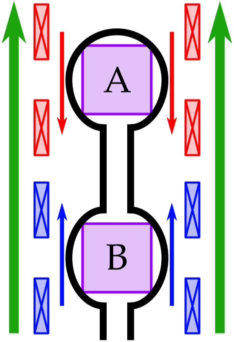
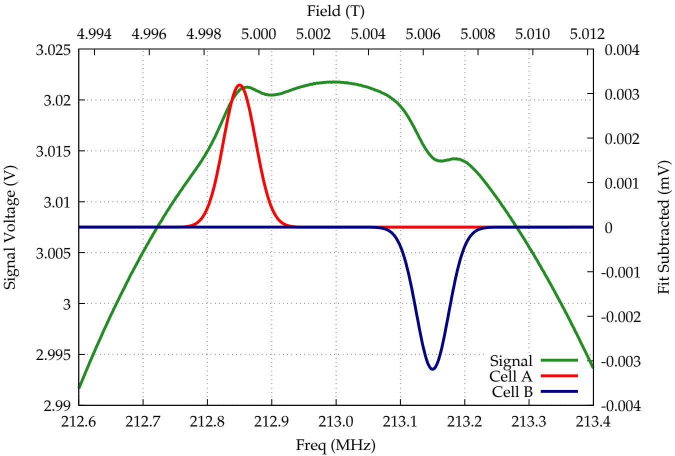
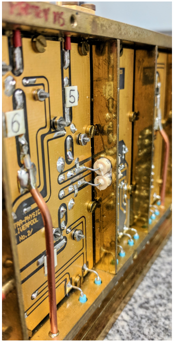
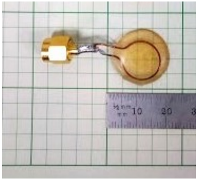


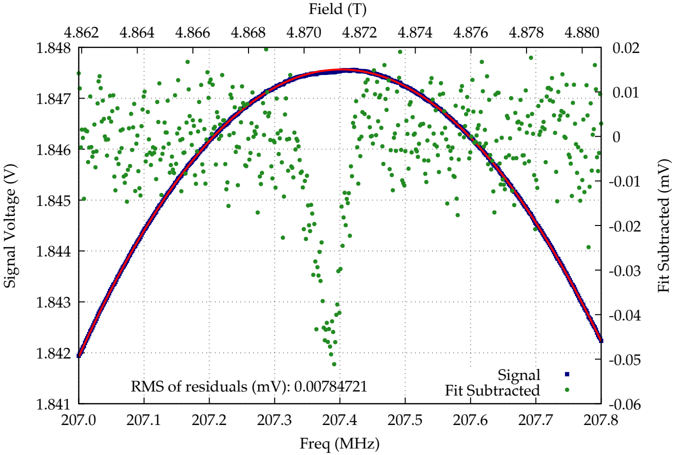


Single microwave frequency applied to polarize

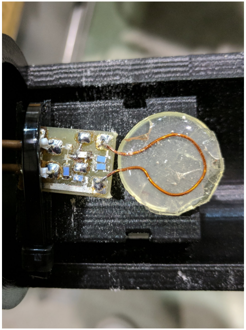
Traditional Liverpool NMR

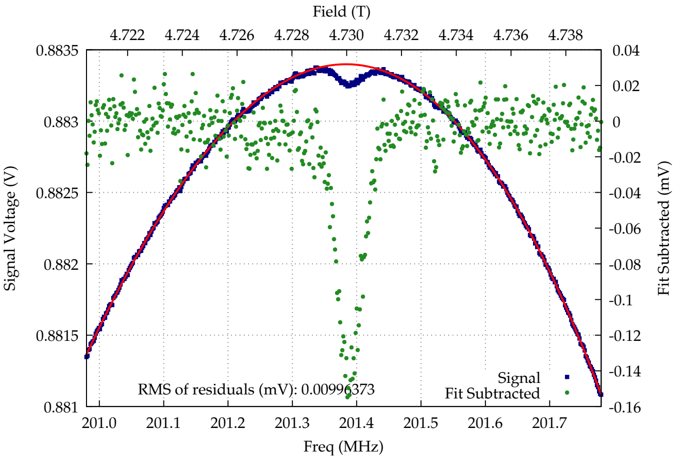




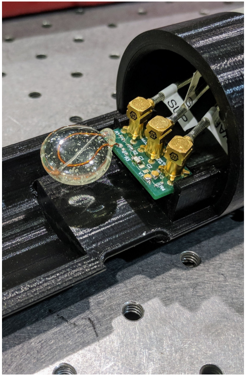


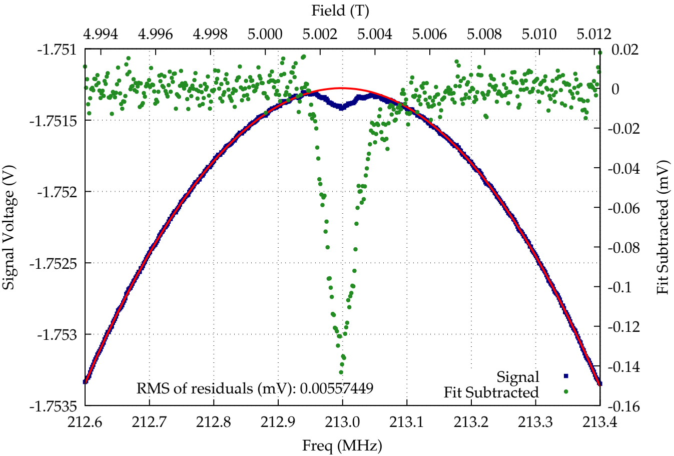
Testing Legacy Cold Board



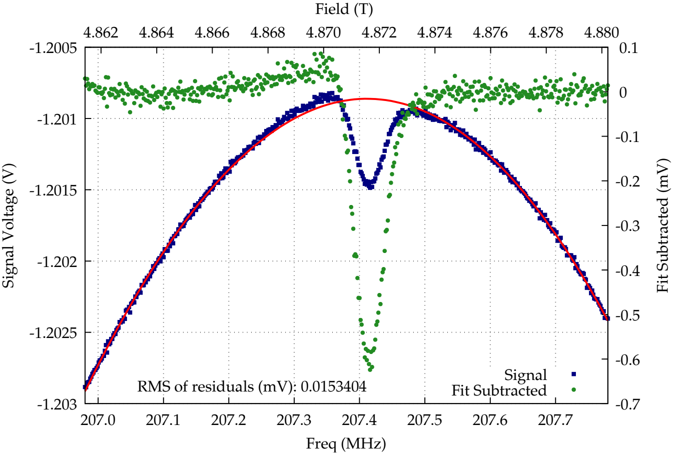


New Varactor Cold Board with Phase Shifter

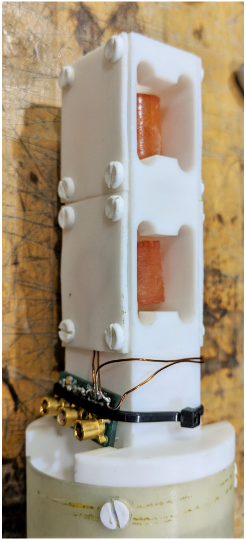


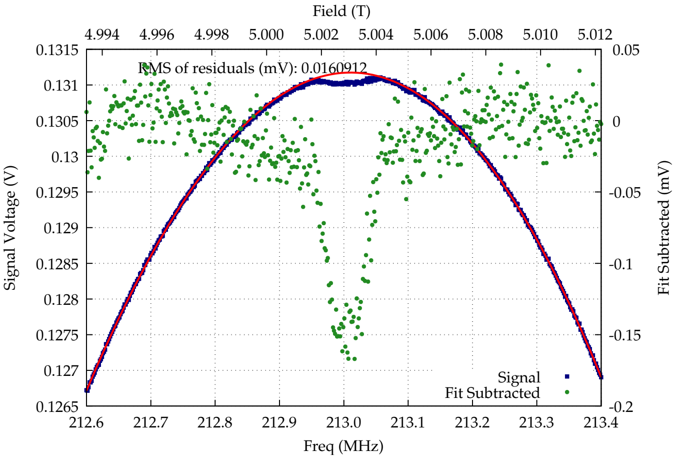


New Varactor Cold Board, Enhanced

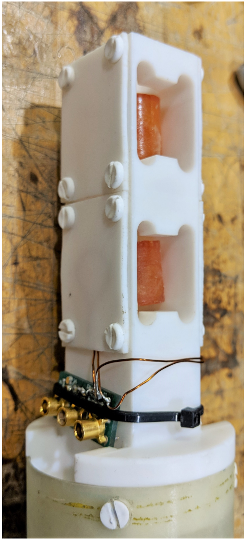


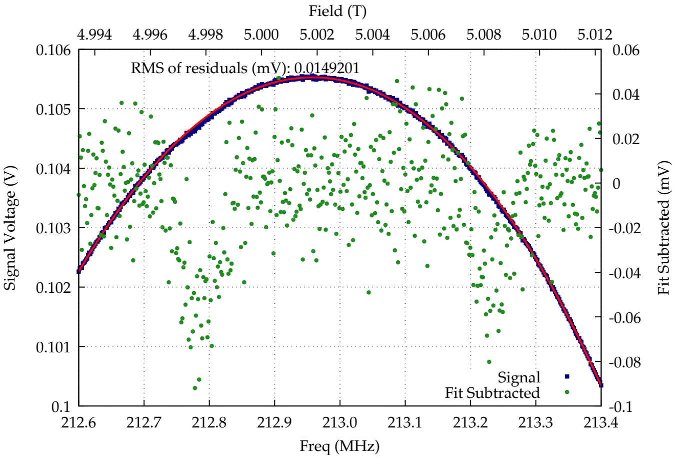
External Double Coil, Shims Off



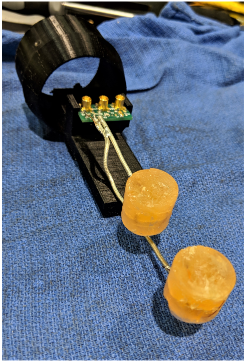


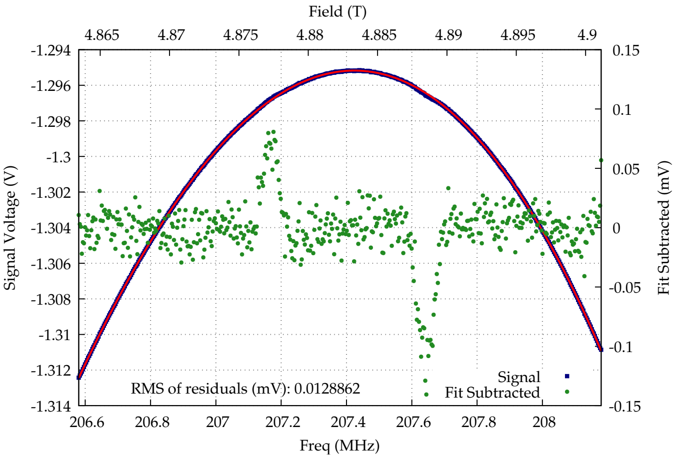
External Double Cell, Shims On





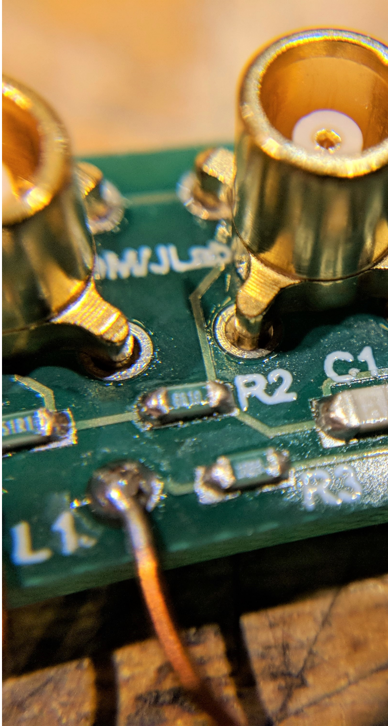
Double Cell, Opposing Polarization





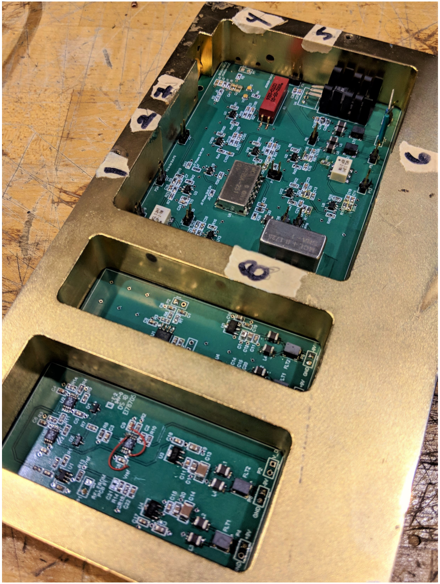
Outline

1. NMR and JLab Solid Targets
    - Introduction
    - New Challenges
2. Progress
    - Cold Tank Circuit NMR
    - 77 K NMR Test Bed
3. Looking Forward
    - Improving Hardware
    - Next Steps



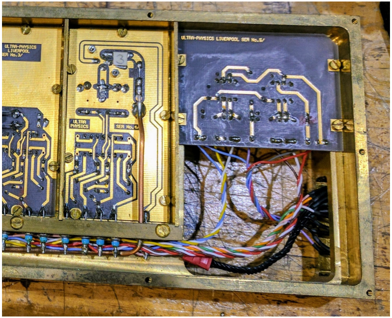
A New Liverpool Q-meter

- Qmeters now limited commodity
- Components that break are often not easy to fix
- Bochum group already building new Q-meters in the Liverpool style with new components
- We are following their lead, developing expertise to make improvements
- Small improvements: internal phase shifter, jumper to enable cold board
- Future: quadrature measurement with phase shift and two mixers



Other Ways to Reduce Noise

- Digitize inside Q-meter
  - Internal ADC board under testing
  - Interfaced by Zedboard FPGA
  - Proof of concept implementation is slow
- Cold Amplifer
  - Amplify signal in cryostat, before long transmission cable
  - Initial tests show commercial amplifiers work at 77 K, no improvement in signal to noise seen yet
  - High B field a problem



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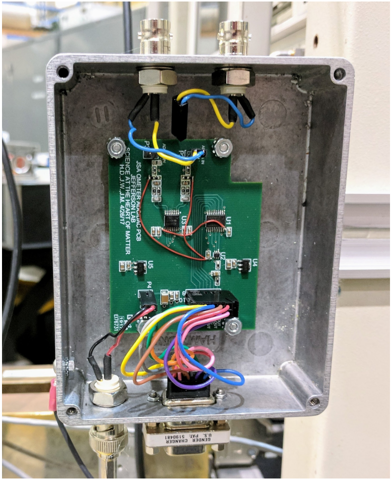

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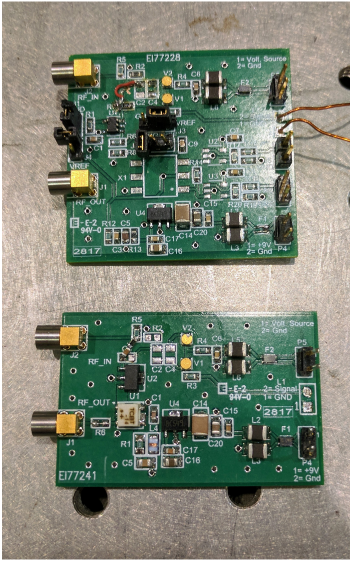
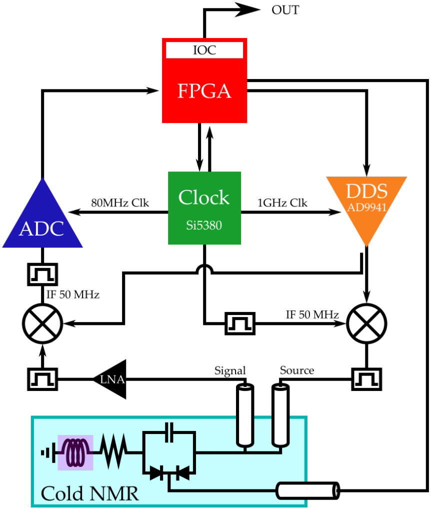


What about something other than a Q-meter?

- Can we do 213 MHz NMR with new, fast ADCs?
  - 1 GHz ADCs readily available
  - Down mix with 50 MHz
- Or be really clever with signal synthesis to get 213 MHz without downmixing
  - Work of JLab electronics group's H. Dong
- Off-the-shelf Lock-in Amp?
  - Zurich Instruments 600 MHz LI
  - Works, but not as fast as Q-meter



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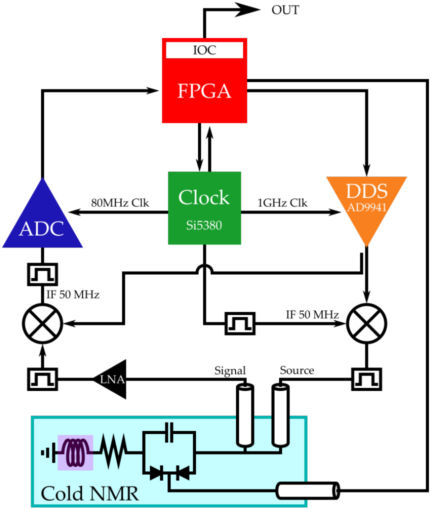

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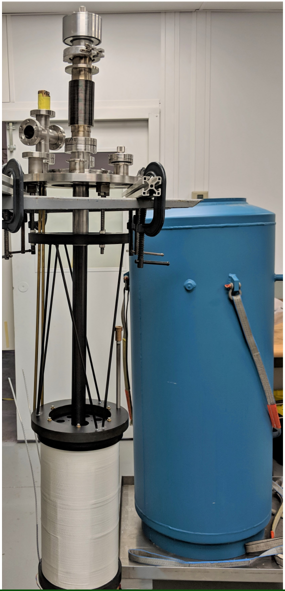
Next Steps: 4 K NMR

- Signals are too small for some external coils, other interesting measurements (N)
- Increase the signal $\Rightarrow$ 4 K
- Existing 7 T magnet, dewar (J.Pierce)
- High-T_C, vapor-cooled leads for magnet and shims
- Superconducting shims
- New, miniaturized NMR boards
- Teflon insert ladder
- Drop in an evaporation fridge to get a full 1.2 K DNP system



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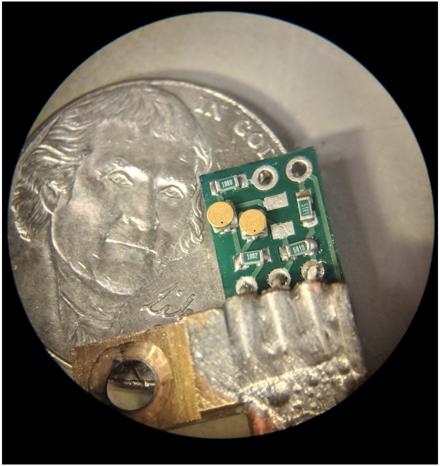

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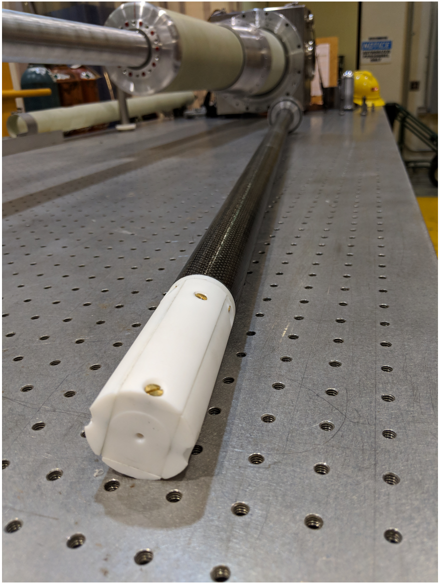

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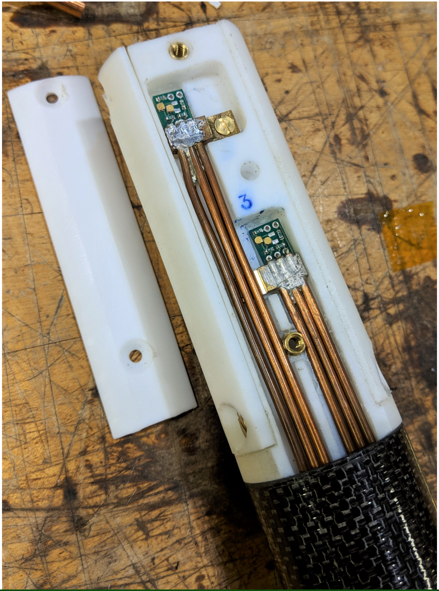

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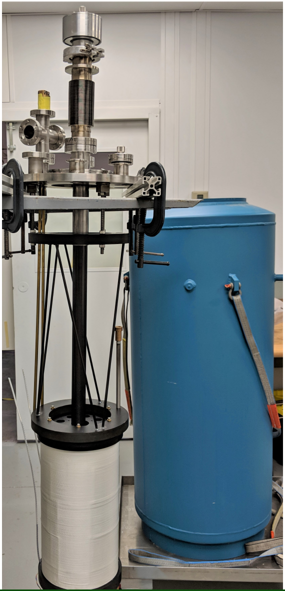


Summary

- Continuous-wave NMR tests proceed as CLAS12 Polarized Target is built
- Demonstrated:
  - 77 K, thermal equilibrium, cw-NMR methods
  - Cold NMR noise reduction confirmed
  - Varactor diode tuning of cold NMR board
  - Electronic phase shifting to allow remote tune
  - Shim simulation and optimization in holding field
  - TE signals in 2 separated cells with a single coil
  - Opposing polarization of 2 cells with a single microwave excitation frequency via shimmed local fields
- Tests will continue at 4 K
- Initial cool-downs of CLAS12 fridge will include applications of these results

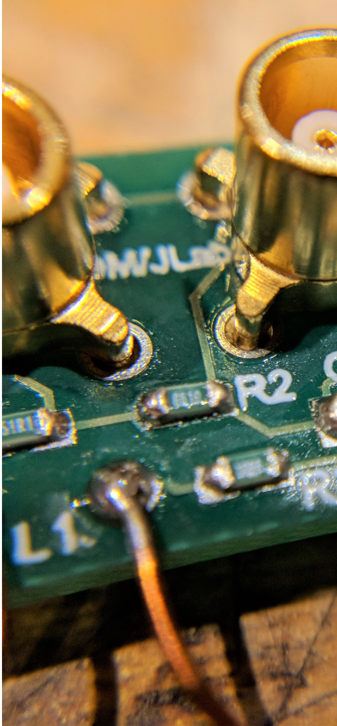
Jefferson Lab Polarized Target Group:

- C. Keith, J. Maxwell, D. Meekins
- J. Brock, C. Carlin, D. Griffith, M. Hoegerl, P. Hood

Special thanks:

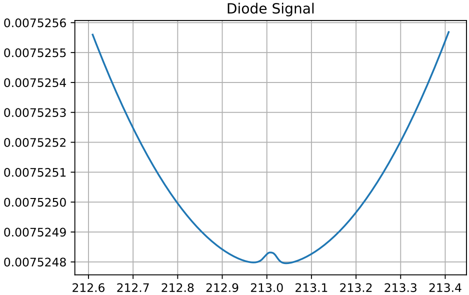
- H. Dong, J. Wilson (JLab Electronics Group)
- J. Pierce (ORNL)
- Crabb, Day, Keller (UVa)
- V. Lagerquist (ODU)

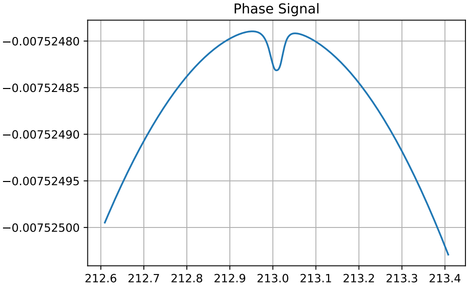
Thank you for your attention!



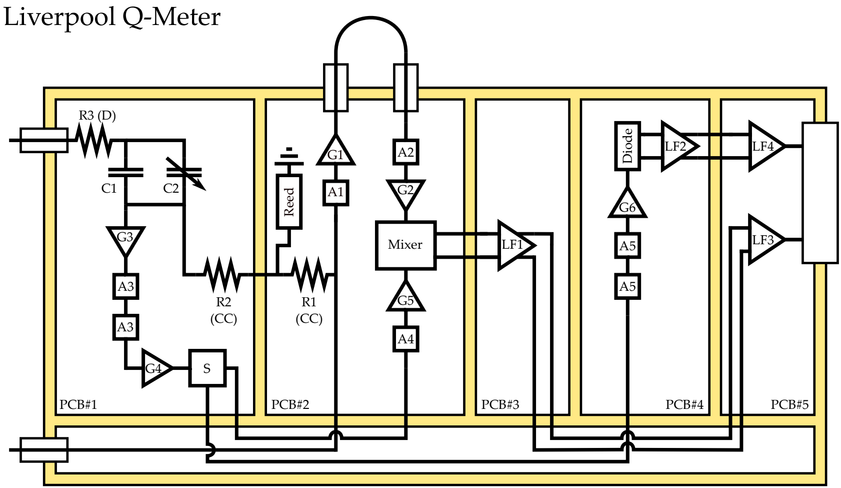
Q-meter Python Simulation Progress

- Started as translation to Python from MathCAD (Houlden)
- Can tune phase and diode, add stray capacitance
- Adding magnetic susceptibility for realistic signal, mixer

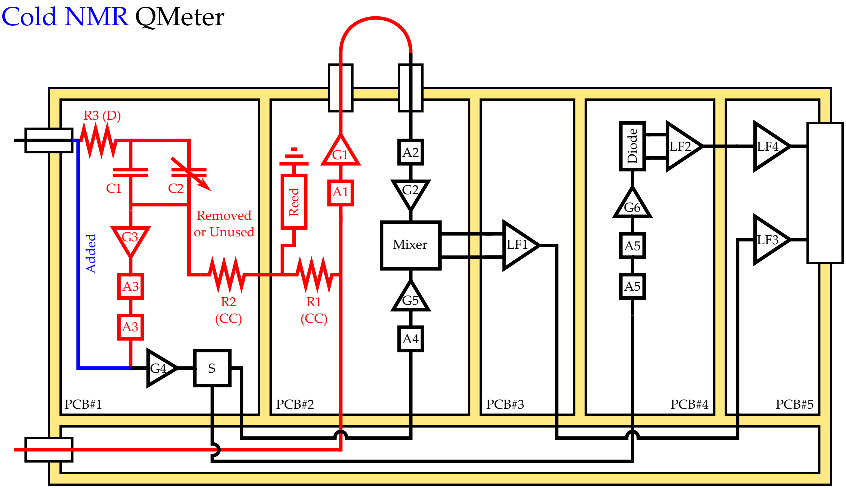




Liverpool Q-meter Layout



Cold Circuit NMR Q-meter Layout



New Varactor Cold Board at Room Temperature