JLab's Polarized NH_3 and ND_3 Targets

Performance for Run Group C

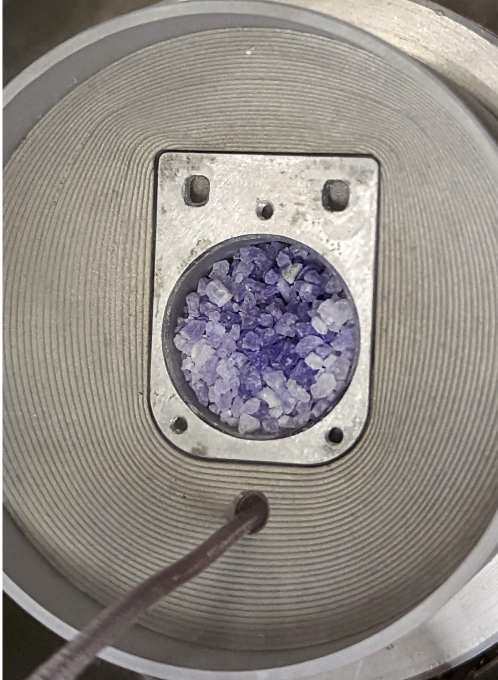
J. Maxwell

With slides contributed by J.Brock, C.Keith

Jefferson Lab

TARGET Group

Tensor Spin Observables Workshop
Trento, Italy
July 12th, 2023



Outline

1. Polarized Solid Targets
   - Dynamic Nuclear Polarization
   - Performance in Beam
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   - Requirements
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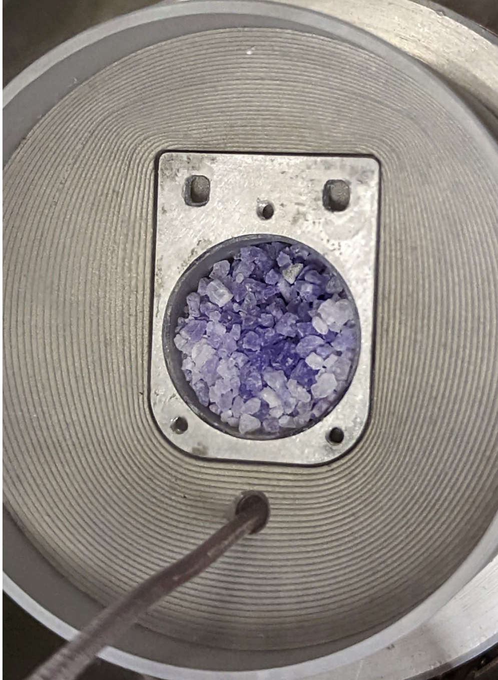
   - Preliminary Results



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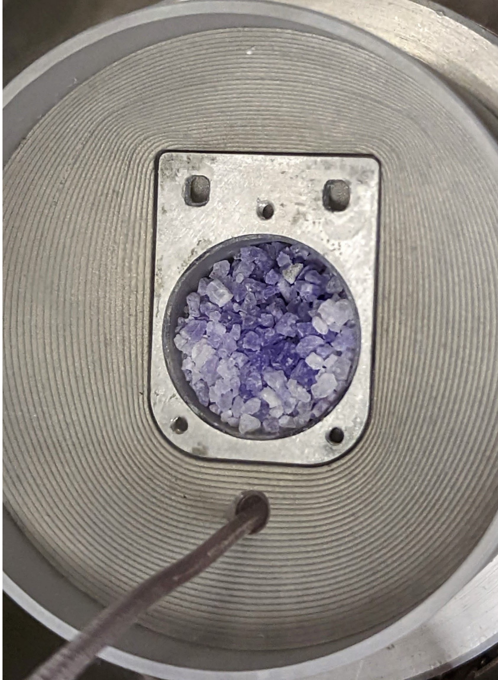


A Starting Point for a Polarized Target

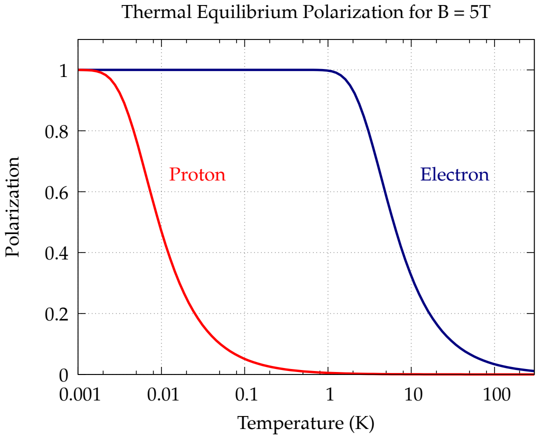
- At equilibrium, populations follow Boltzmann distribution: $N_\downarrow/N_\uparrow = e^{-2\mu B/kT}$

- "Brute force," spin 1/2:

$$P_{\mathrm{TE}} = \frac{e^{\frac{\mu B}{kT}} - e^{\frac{-\mu B}{kT}}}{e^{\frac{\mu B}{kT}} + e^{\frac{-\mu B}{kT}}} = \tanh\left(\frac{\mu B}{kT}\right)$$

$$P(t) = P_{\mathrm{TE}}(1 - e^{-t/t_1})$$

- Spin-lattice relaxation t_1 related to T!
- At 1 K, 5 T, P_e ~100%
- Can we use high e polarization to polarize p?



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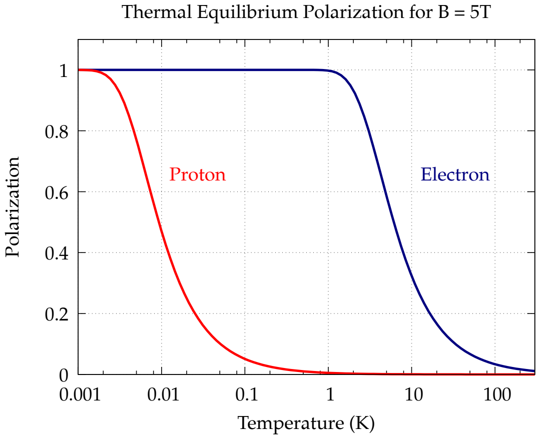
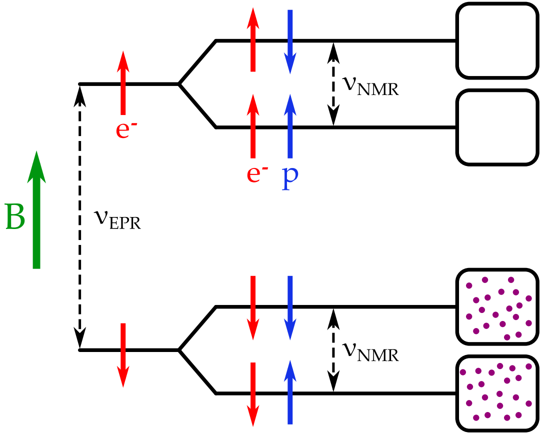


Polarizing with the Solid State Effect

- Use hyperfine $e-p$ spin coupling
- Induce flip-flop transitions: μ-waves
  - $\nu_{\mu+} = \nu_{\text{EPR}} - \nu_{\text{NMR}}$
- Relaxation times are the key
  - $e \approx$ milliseconds
  - $p \approx$ 10s of minutes
- Continue until new equilibrium
- At 5 T & 1 K: $P_p \sim 95\%$, $P_d \sim 50\%$
- Choose polarity without changing magnetic field
  - $\nu_{\mu-} = \nu_{\text{EPR}} + \nu_{\text{NMR}}$



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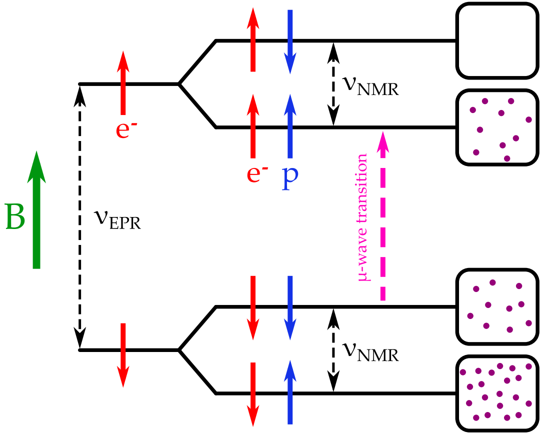

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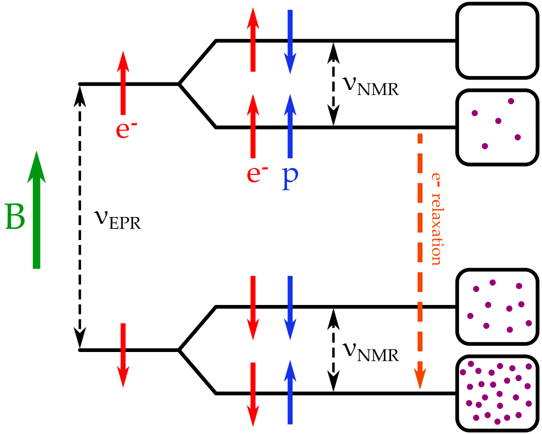

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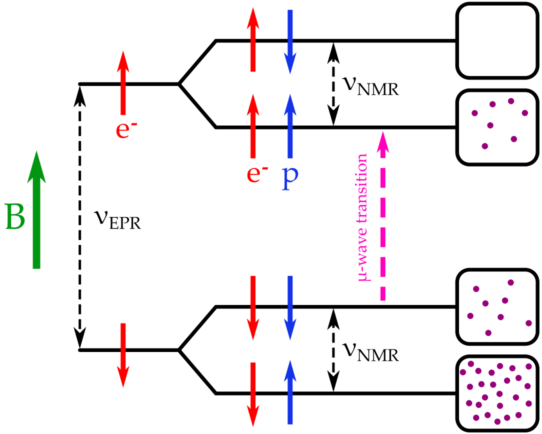

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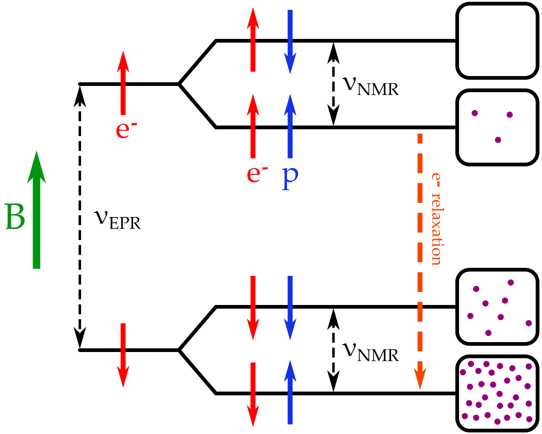

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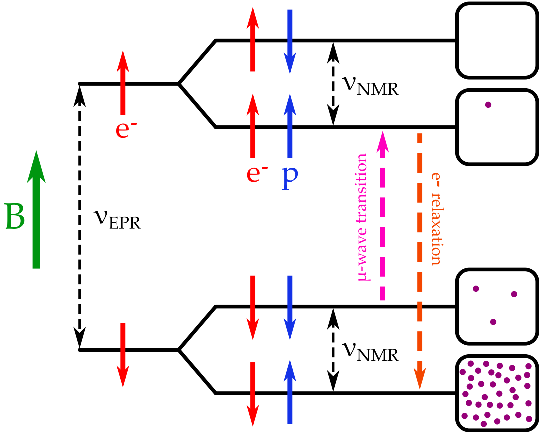

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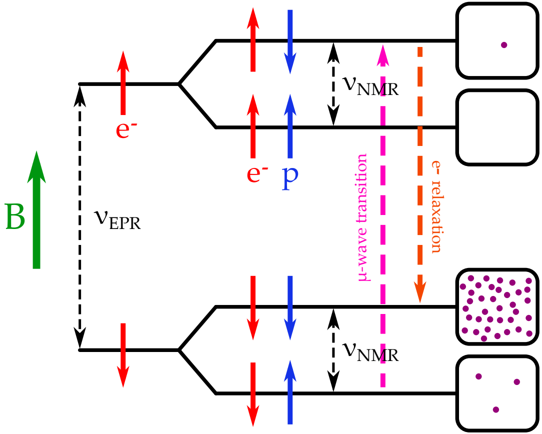
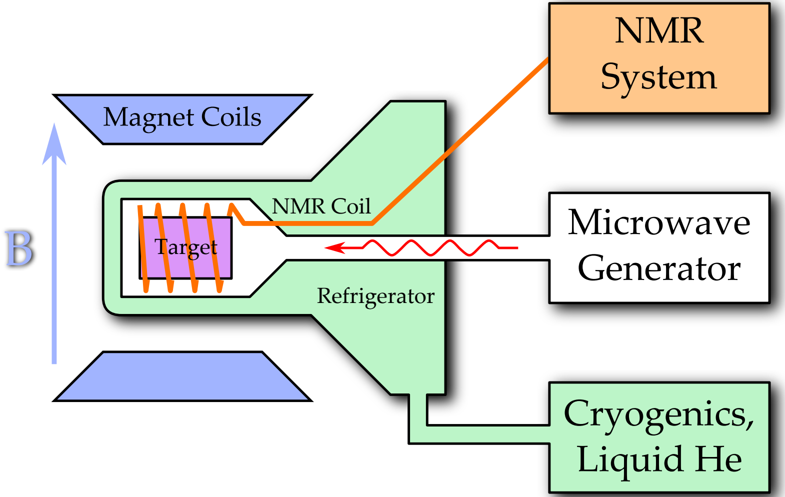


A Dynamic Nuclear Polarization System



DNP System Components

- 5 T superconducting magnet
  - Field uniformity can limit polarization
- Liquid ^{4}He evaporation refrigerator to give sufficient of cooling power at 1 K
  - Superfluid conducts heat out of material beads very well at these temperatures
- Extended Interaction Oscillator tube makes 20 W microwaves at 140 GHz for 5 T
- Target material must be doped with free electrons for flip-flops!
  - Paramagnetic centers from free radicals: chemical doping or ionization from irradiation

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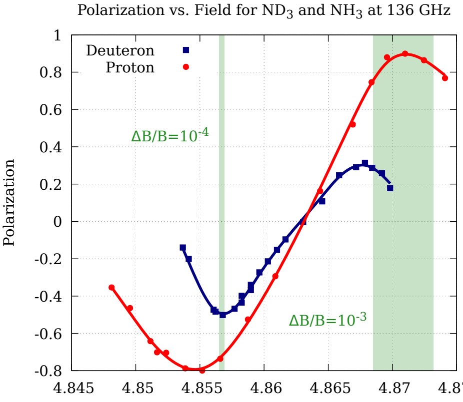

M. Seely, Yale 1982

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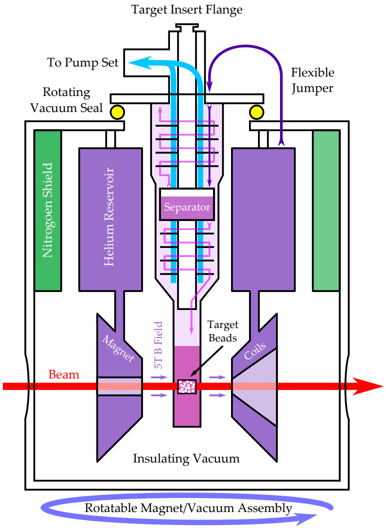

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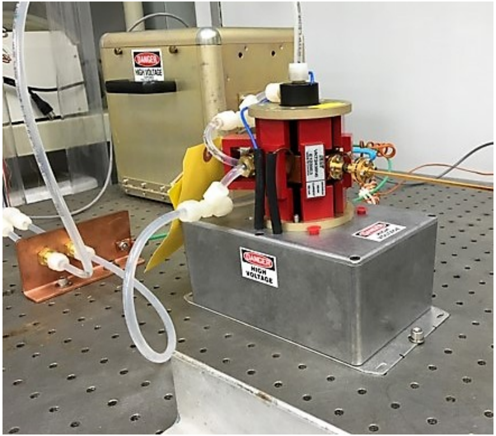

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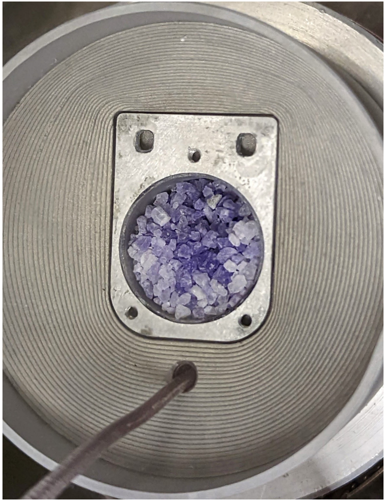
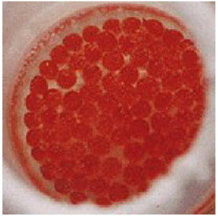
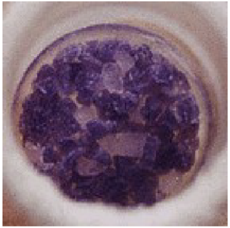


Material	Type	Dopant	Dilution	Polarization	Rad. Res.
Butanol	C_4H_9OH	TEMPO	13.5%	90-95%	Moderate
D-Butanol	C_4D_9OD	TEMPO	23.8%	40%	Moderate
Ammonia	$^{14(15)}NH_3$	Irrad.	17.6%	90-95%	High
D-Ammonia	ND_3	Irrad.	30.0%	50%	High
Lithium-H	^7LiH	Irrad.	25.0%	90%	Very High
Lithium-D	^6LiD	Irrad.	50.0%	55%	Very High


NMR Measurements

- In field B_0 apply RF field to material at Larmor frequency ω_0
- Coil of L_0 perpendicular to B_0 to induce spin flip
- LCR circuit so that $\omega_0 = 1/\sqrt{LC}$, observe change in impedance with frequency

- As frequency changes: circuit response Q-curve and polarization signal
- Sweep frequency around ω_0 to integrate in ω
- Must calibrate signal area against known polarization:

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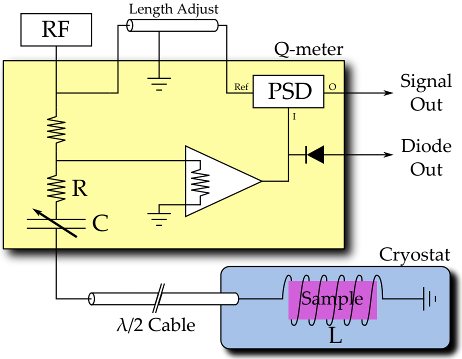

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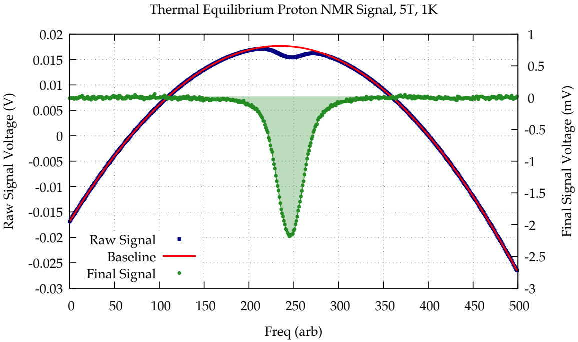
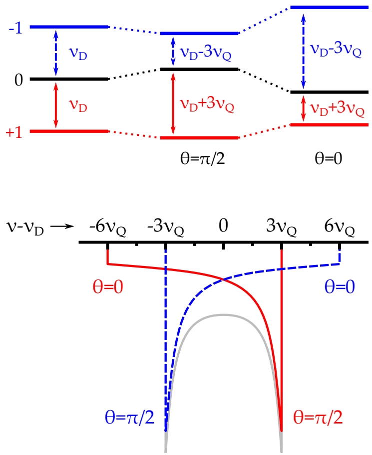


Deuteron Polarization and NMR Lineshape

- If d were a dipole, levels would be degenerate: single line NMR peak
- Quadrupole moment of d interacts with electric field gradients in the lattice
- Energy level changes with angle between field and molecule θ
- Observed NMR signal is sum of $-1 \rightarrow 0$ and $0 \rightarrow 1$ transitions
- Separation allows manipulation with RF
- Measuring relative size of peaks gives polarization, allowing calibration!



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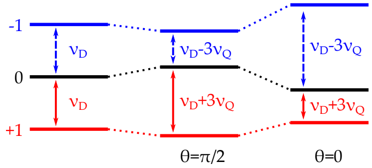
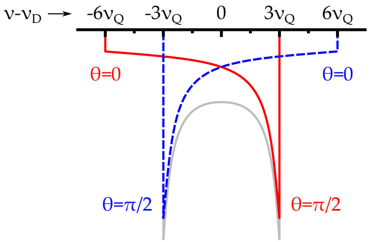
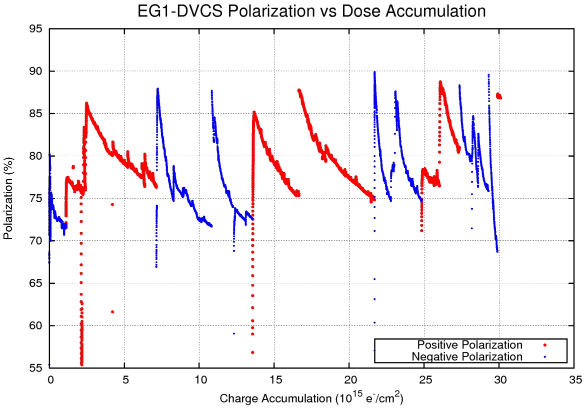




NH_3 Performance in Experimental Beam

- Initial dose $\sim 10^{17} e^{-}/\text{cm}^2$ ($NH_2^{\bullet}$) in liquid argon before experiment
- Polarize above 90% after this irradiation
- "Cold" dose at 1 K will produce more radicals ($H^{\bullet}$). Too many hurt polarization!
- Regain polarization via anneals: heating recombines radicals (∼90 K for ∼30 mins)
- With more beam, stable radicals are produced which can't be removed ($N_2H_4^{\bullet}$)
- Polarization decay rate increases until running untenable, material replaced.



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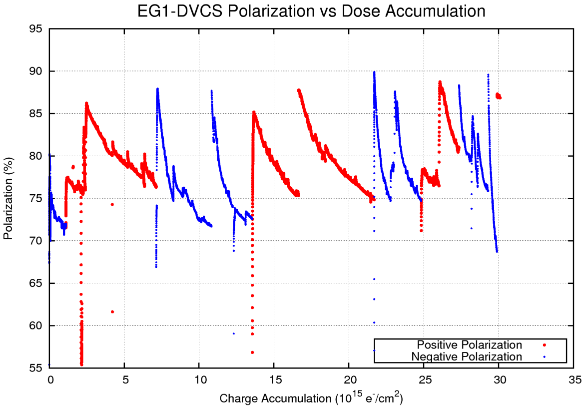

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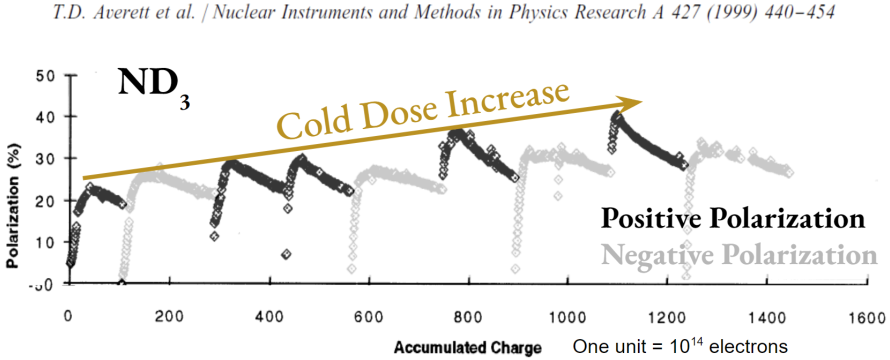
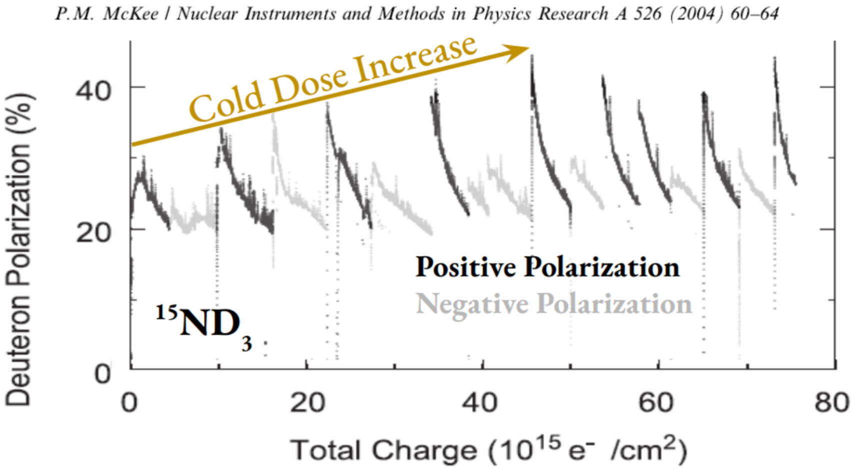


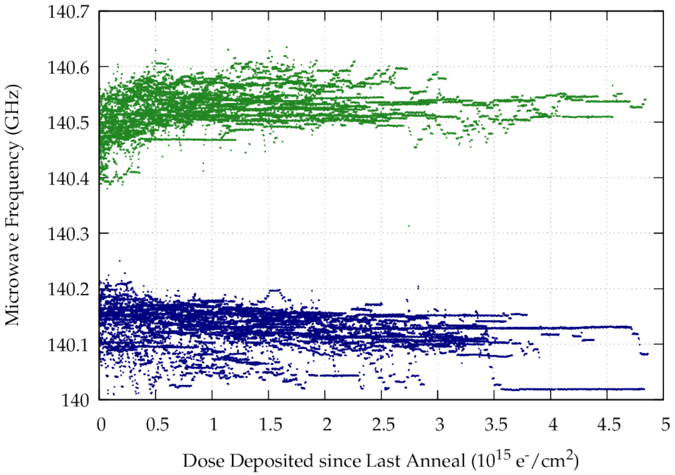
- 'Cold' dose is crucial to increasing polarization with successive anneals
- High-current irradiation runs at 1 K needed for Hall B

$^{15}ND_3$ Performance in Experimental Beam



Optimal Microwave Frequency

- Accumulation of paramagnetic centers also causes a shift in the optimal microwave frequency
- Operator must adjust the frequency
- Development of automation at UVa and a newly funded ML effort at JLab



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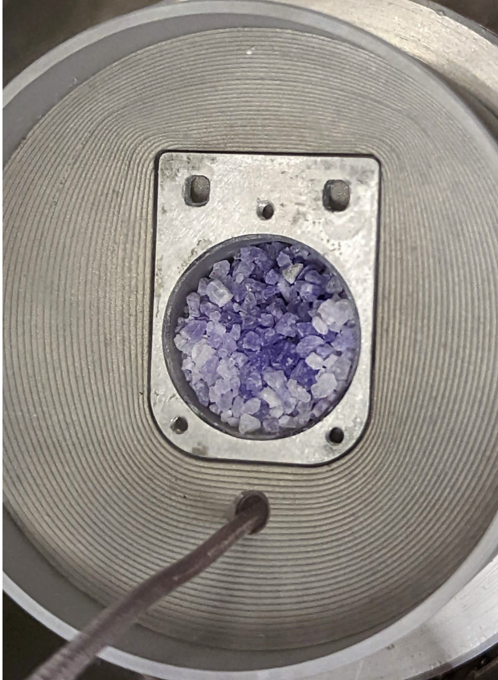

Requirements


D
A
B
C

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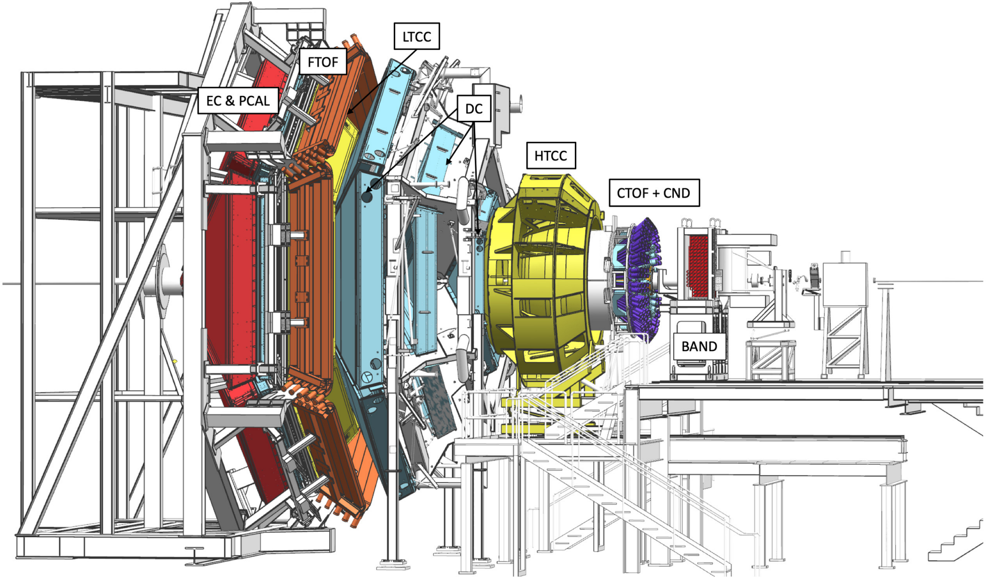

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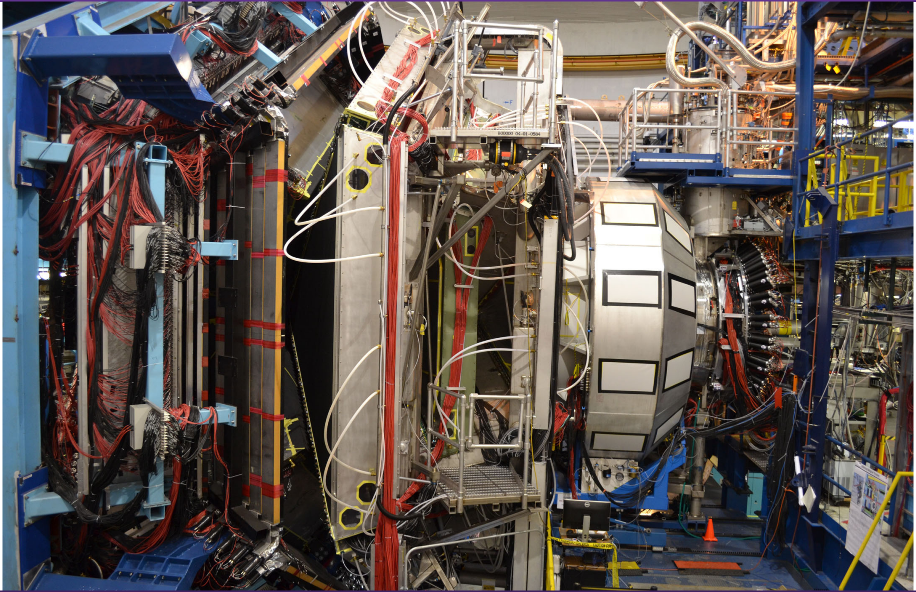

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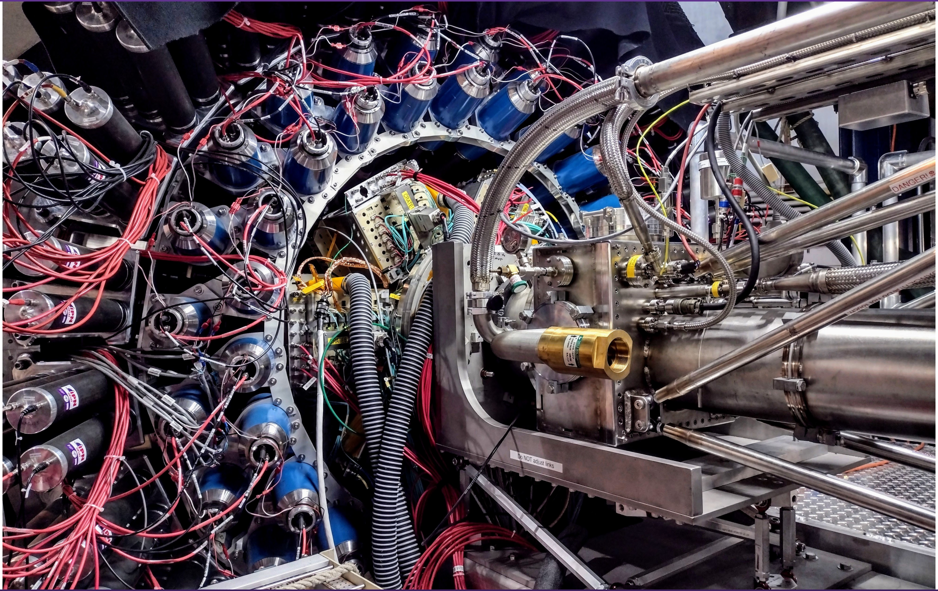

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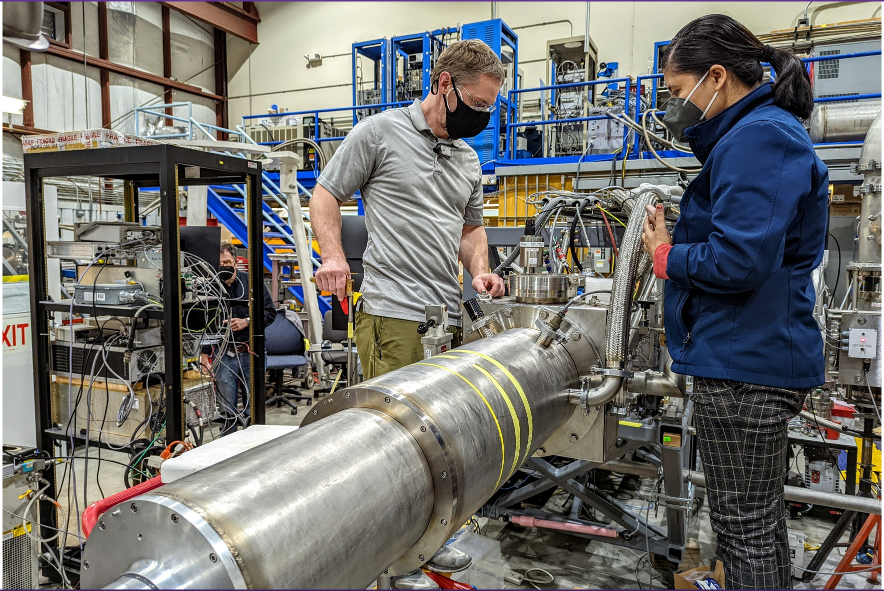
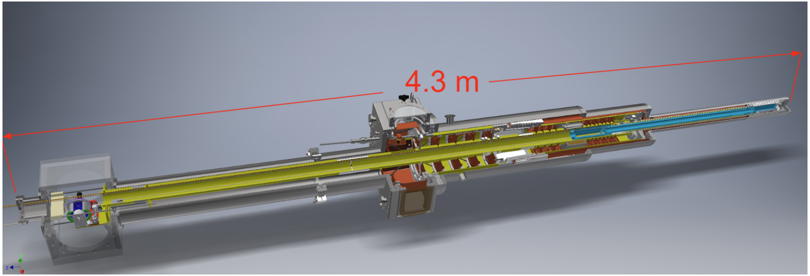


Requirements

- Fit in CLAS12, which does not allow vertical access to interaction region
- Accommodate cells of 5 cm length, varied diameters
  - 15 mm for FT On (Moller shield)
  - 20 mm for FT Off
- NMR coils outside cell to avoid scattering background
- Include magnetic shims to improve field uniformity for deuteron polarization
- Accommodate anneals and material changes with minimal overhead

Target designed by J. Brock, C. Keith

Fridge Design



- Very long, horizontal cryostat to fit in CLAS12, 10 cm bore. 5 mm clearance.
- New ^4He evaporation fridge, provides 1 W at 1.07 K
- Beamline blocks removal, would require disassembly with every change
- "Trolley" retracts bath for quick material changes (J Brock)

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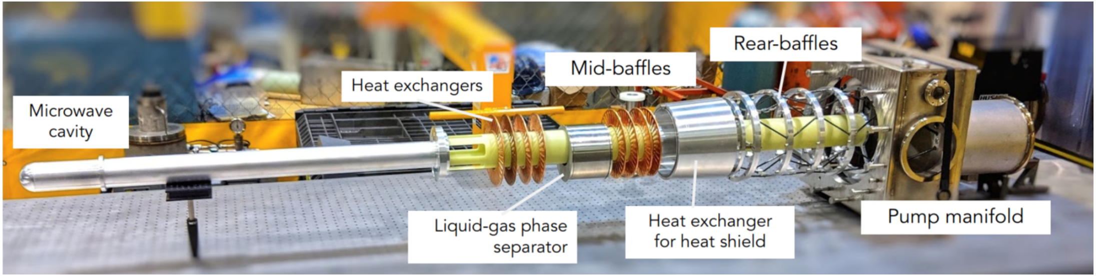

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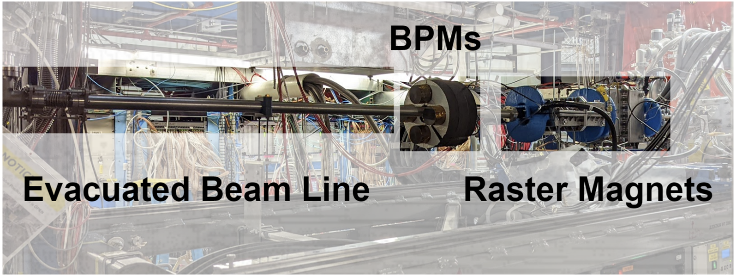

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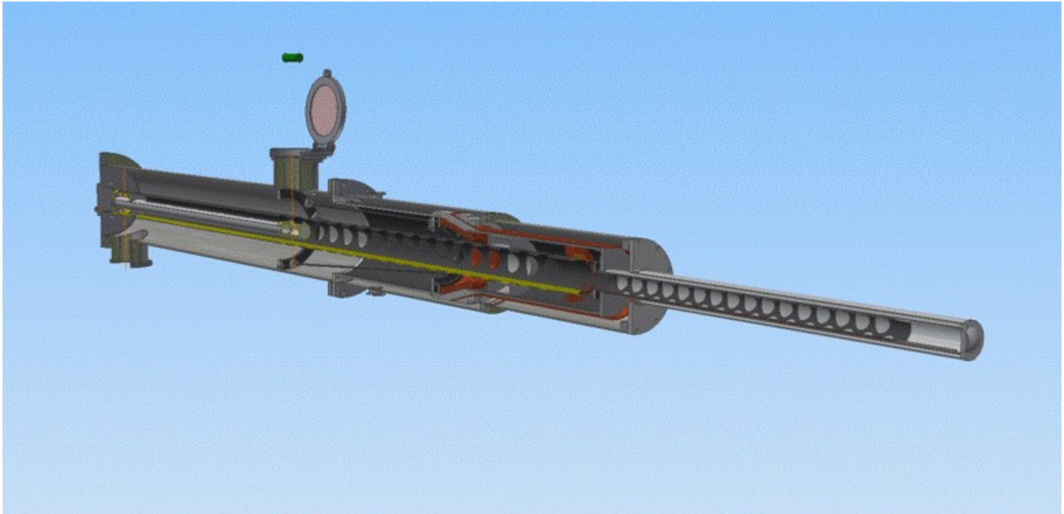
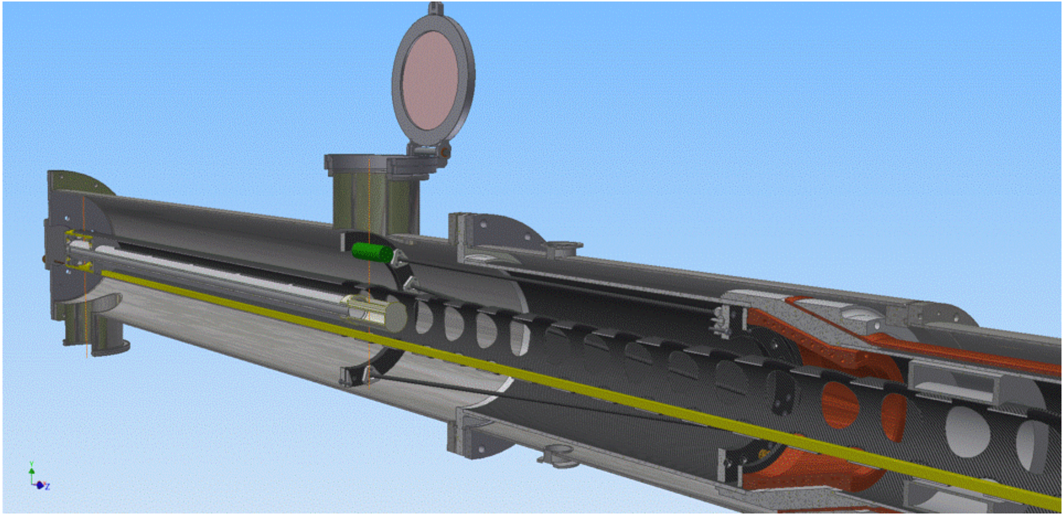
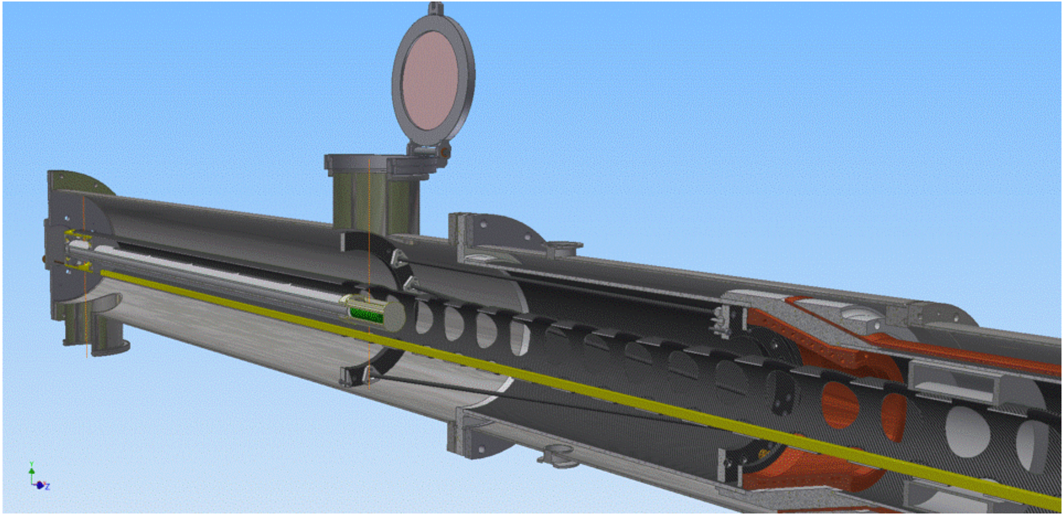
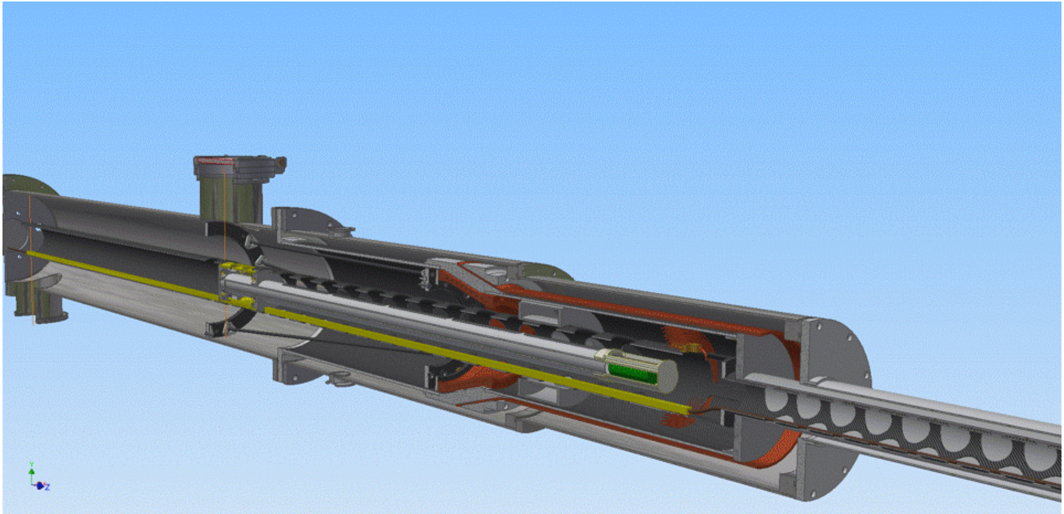
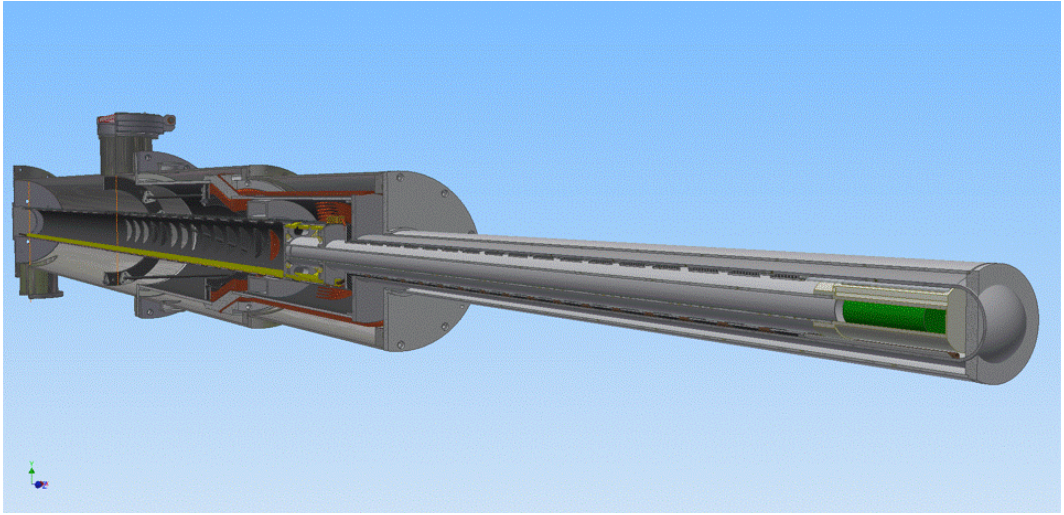




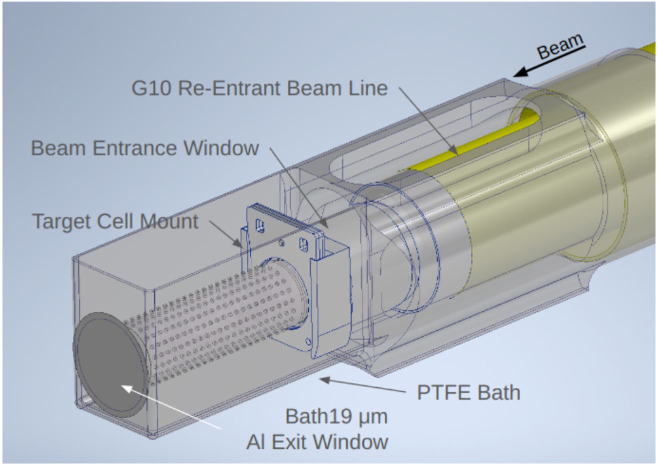






Liquid Helium Bath

- Beam travels through evacuated center of trolley
- Liquid He fills bath and extends upstream in annular space that runs length of the trolley
- Cell is held securely in mounting bracket, and inserted from above with special tool
- Beam passes through AL windows on bath (2), cell (2), pump tube and OVC



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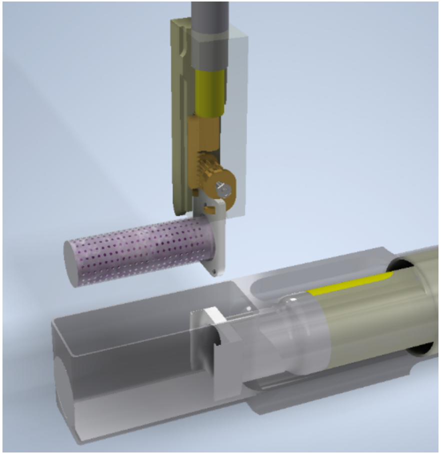

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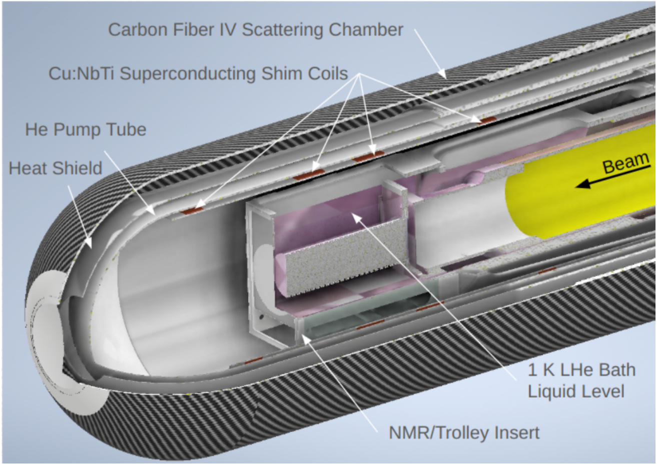
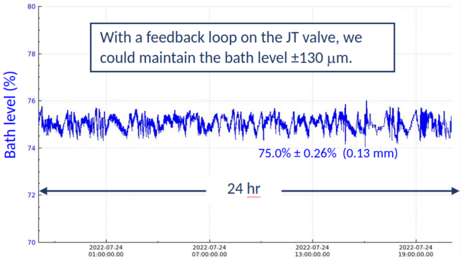
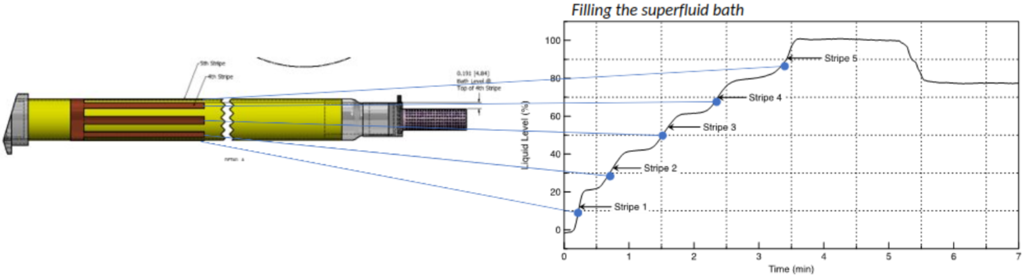


- Bath is only 40 mm deep: only 10 mm from top of cell to top of bath
- Capacitance-based level probe developed with sensitivity of $\sim$20 μm
- Stripes aid level determination
- Maintained level within 130 μm





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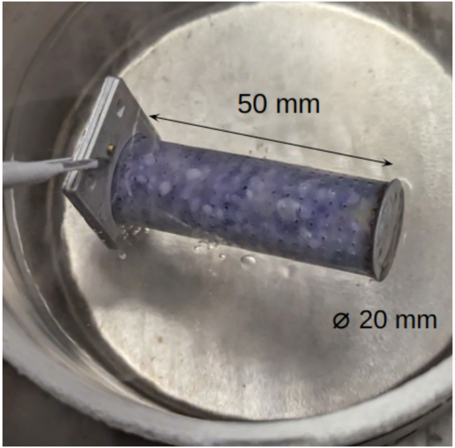
- Perforated PCTFE (Kel-F) cylider with 20 μm Al windows
- Supported upstream by Al frame
- 15 or 20 mm diameter for $NH(D)_3$
- Also C, CH_2 and optics cells
- Loading cells has been a painful, imprecise operation in the past
- Another C.Keith/J.Brock design, the gas-cooled "chimney"
- Material loaded before the experiment, staged in "ladder"



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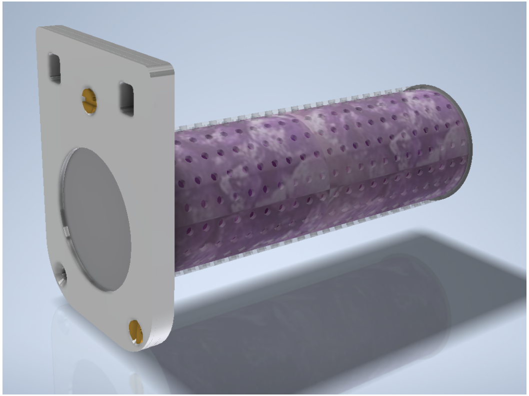

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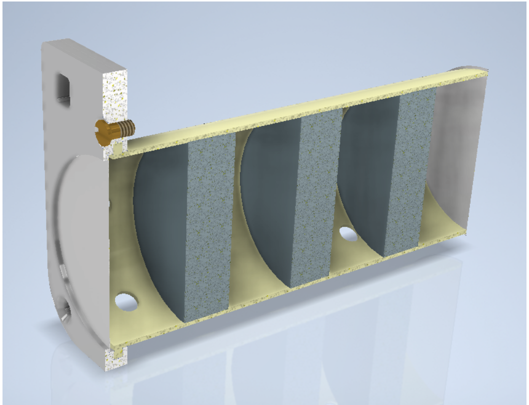

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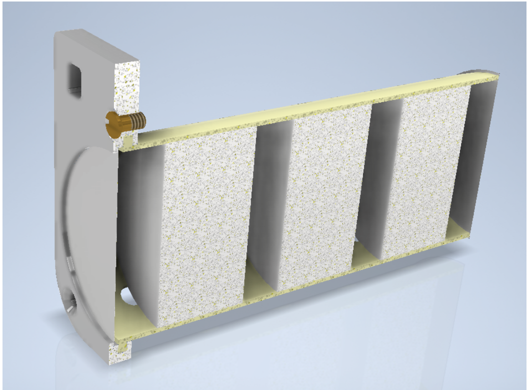

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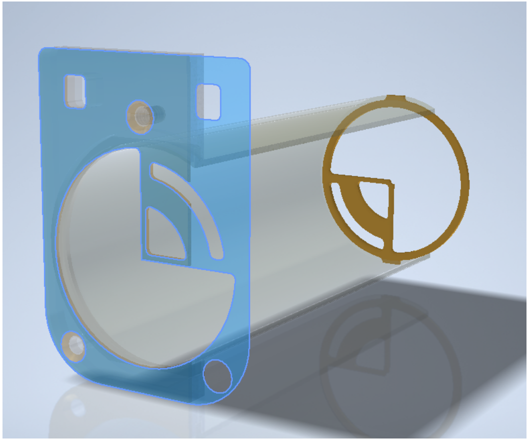

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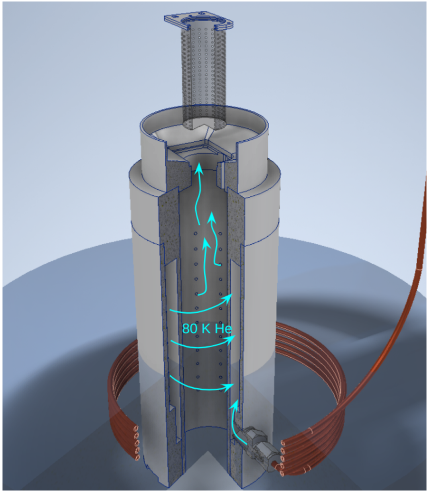

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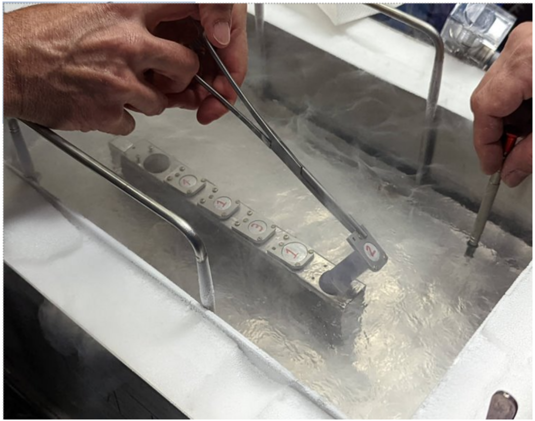
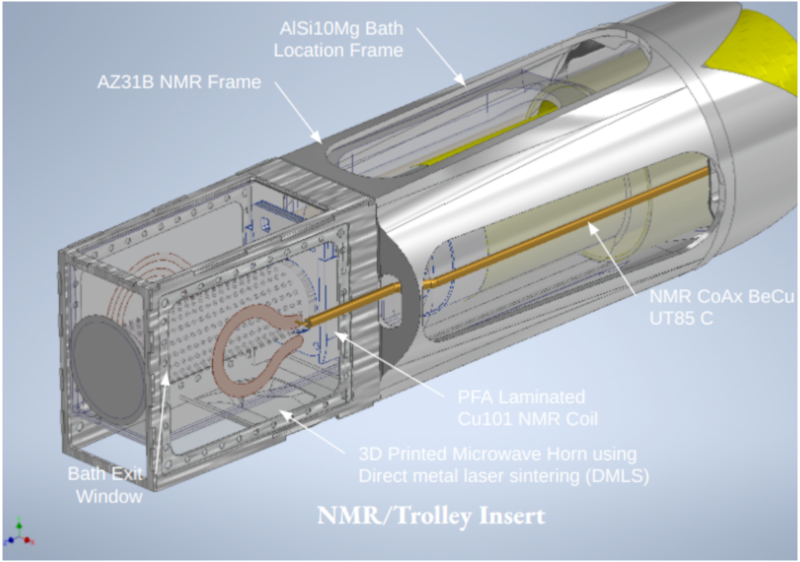
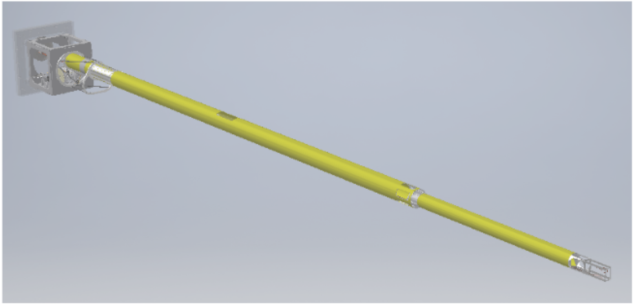
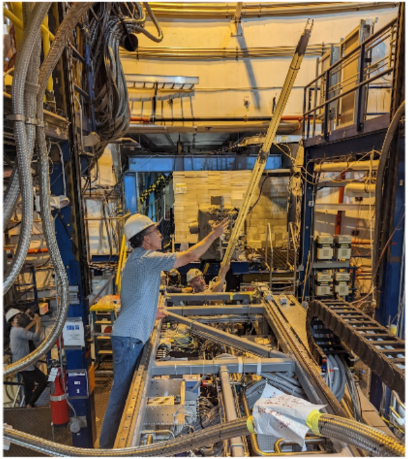


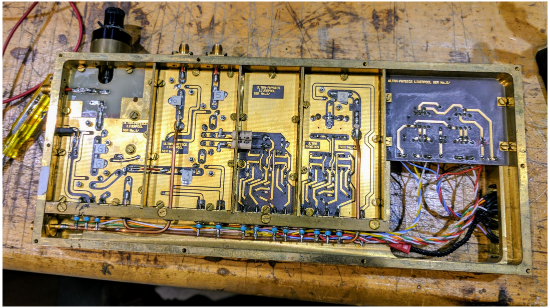

AlSi10Mg Bath
Location Frame
AZ31B NMR Frame
NMR CoAx BeCu
UT85 C
PFA Laminated
Cu101 NMR Coil
3D Printed Microwave Horn using
Direct metal laser sintering (DMLS)
Bath Exit
Window
NMR/Trolley Insert





New NMR System

- Venerable Liverpool system aging
- New system hews closely to Liverpool design, with all new components, significant ease-of-use improvements
- Electronic tuning of tank circuit capacitance and phase shift
- Tank circuit within cryostat for Deuteron
- Modular design with off-the-shelf components
- Fits in 3U chassis, only needs ± 5 V



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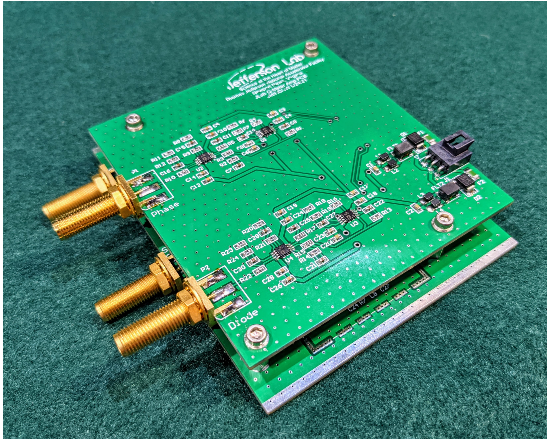

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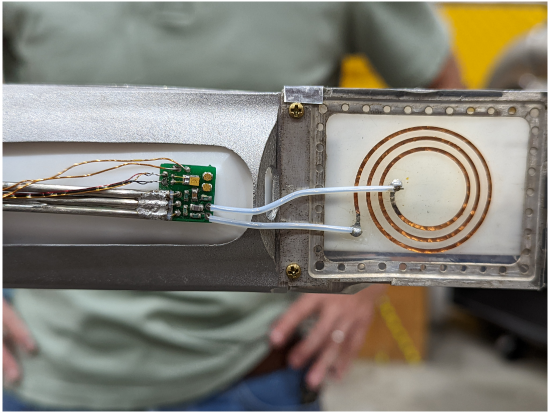

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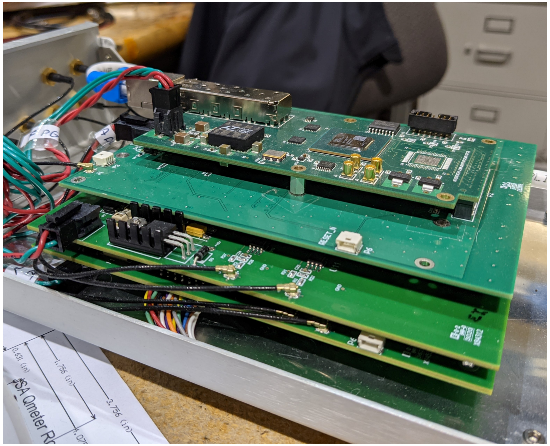
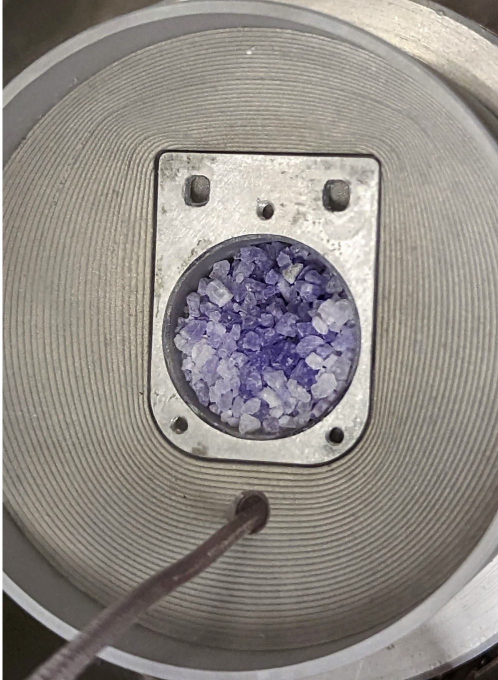
New NMR DAQ System, Software

- New FPGA-based data acquisition
  - Xilinx FPGA with frequency sweep algorithm, UDP/TCP communication
  - 2 ADC channels, 24-bit, 256 ksps
  - 2 DAC channels for tuning
- Interface directly to synthesizer of Rohde & Schwarz signal generator, reducing settle time by more than half
- New software package in Python
  - PyQt5, pyqtgraph, SciPy
  - 3-stage, modular curve analysis, including deuteron peak fits
  - Task-based tabs for tuning, baselines, thermal equilibrium measurements

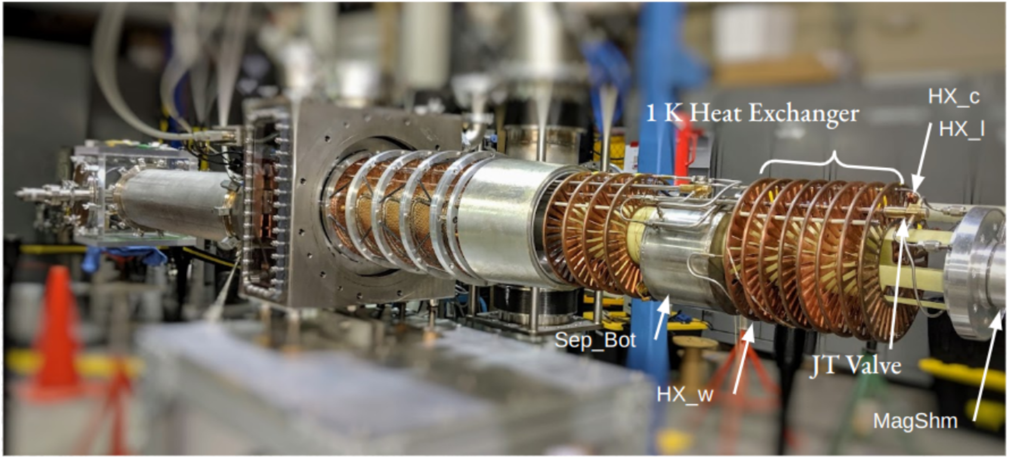


Outline

1. Polarized Solid Targets
   - Dynamic Nuclear Polarization
   - Performance in Beam
2. Run Group C Target
   - Requirements
   - Design
3. Target Performance
   - Operation
   - Challenges
   - Preliminary Results



Fridge Performance: Design by Brock, Keith



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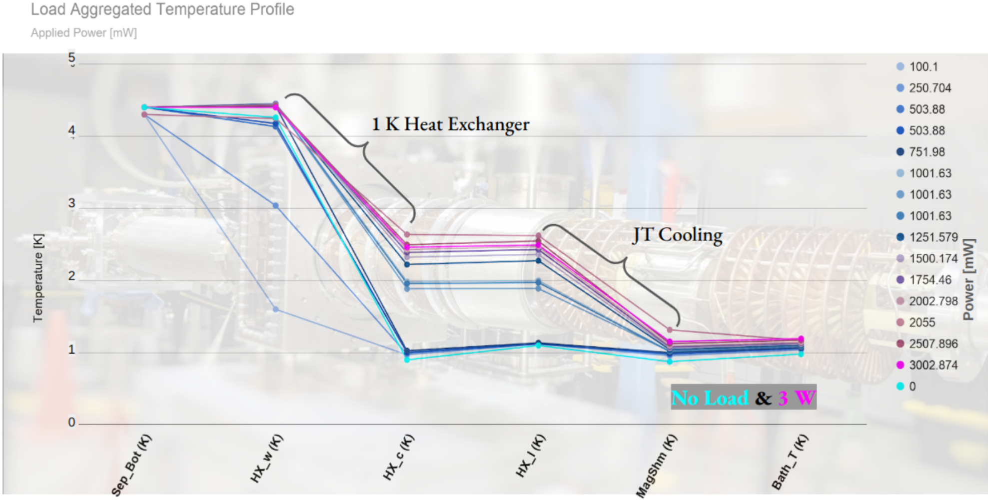
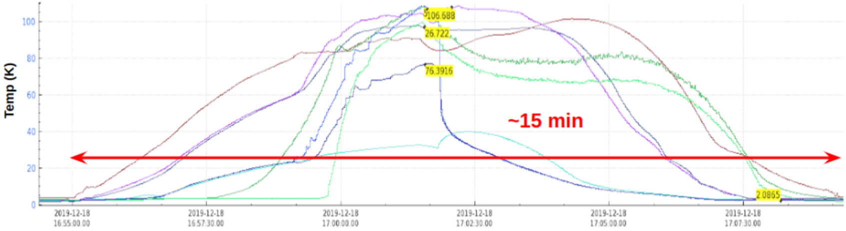
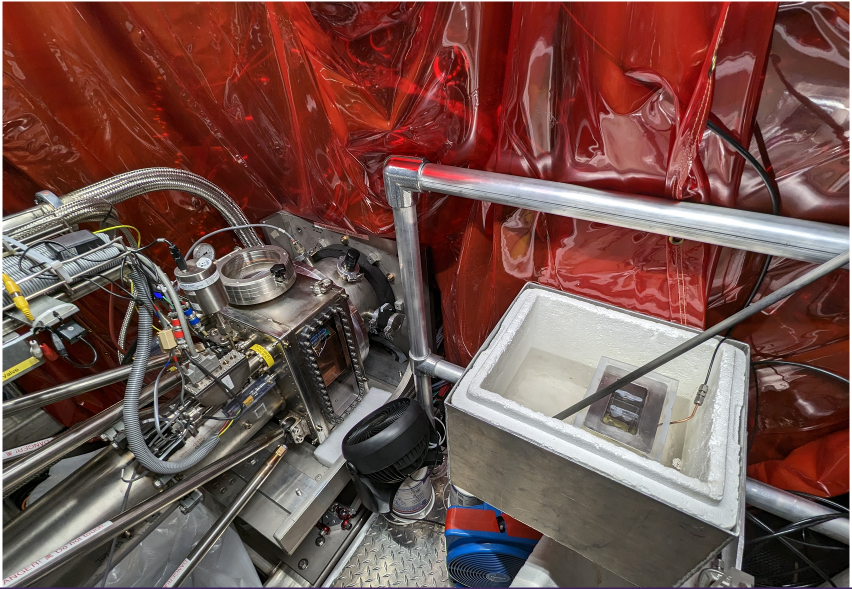


Material Changes

- 80 material swaps over the experiment
- Trolley system saved roughly 19 days of downtime overhead
- Accomplished by 2 people, one to load, one to work the fridge and pulley
- Anneals were swaps, with $\sim$ 30 minutes in LAr before putting back in

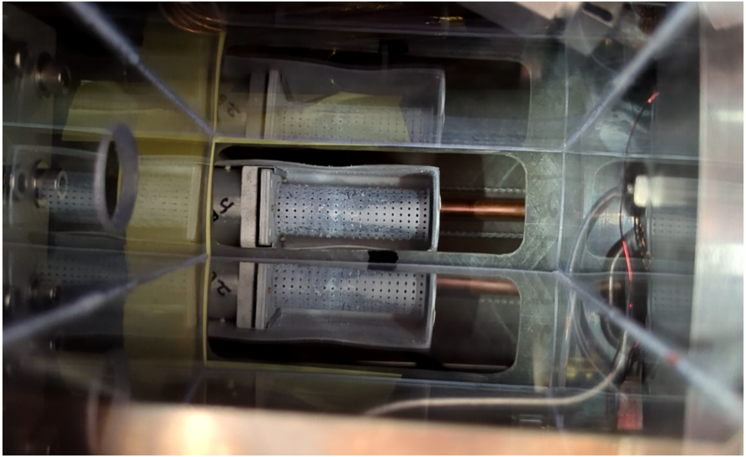


Operation



Operation

Material Florescence Video

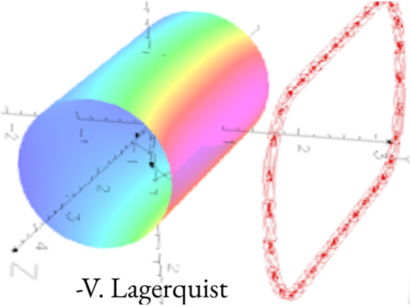


Experimental Challenges

- Overall, RGC was the smoothest polarized target experiment in recent memory
- Remarkable uptime: 132 days of target operation, 3.7 days down due to target
- Few small issues caused minimal impact
  - Failure of NMR cable (tuning) after initial cool-down
  - Heater to maintain NMR cold tank fried
  - Icing of run valve
- Failure of NMR cable (tuning) after initial cool-down
- Heater to maintain NMR cold tank
- Largest experimental delay came from 5 T solenoid power supply failure (firmware issue), caused a down from November to late January

Exterior NMR Cells

- Requirement for external coils was clear:
  - P_bP_t for final analysis to be determined from scattering
- A simple coil should be considered more as near-field detectors
- Condensation near coil caused small but important signal
- For Deuteron line-shape polarization determination, this can be very misleading!
- NMR measurements are a crucial guide to maximizing polarization. External coils not recommended!



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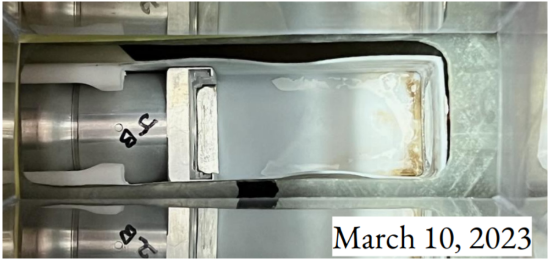

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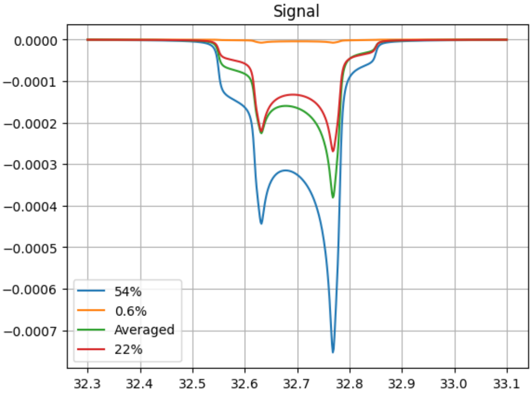
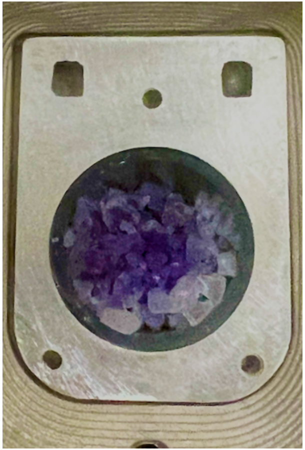


Raster Size and NMR Measurements

- Beam is rastered to spread evenly over cell
- 15 mm and 20 mm diameter cells needed for different detector configurations
- Raster must be limited to avoid beam collisions with cell walls
- For 15 mm, a 12 mm spot was used
- Exacerbated NMR inaccuracy due to external measurement coil
- Challenging to choose μwave frequency
- More sensitive to the portion NOT in beam!
- Better alignment $\rightarrow$ larger raster



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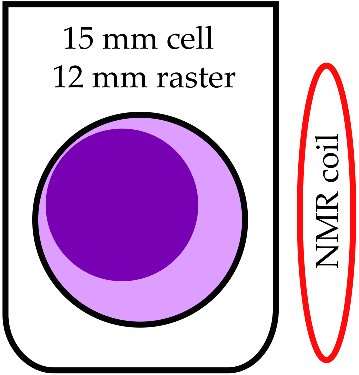

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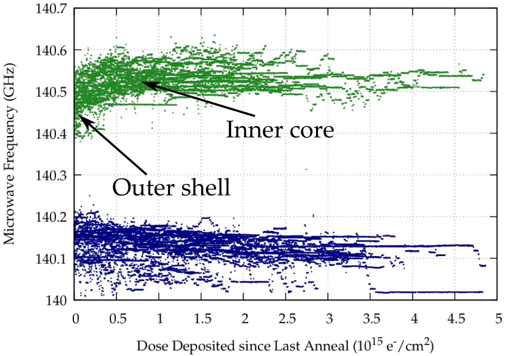

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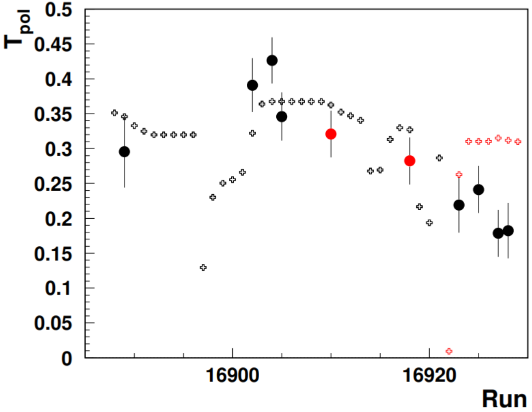
Deuteron NMR (open) vs. Scattering (filled)



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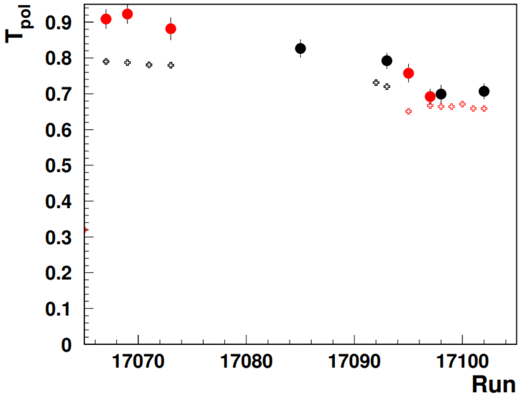
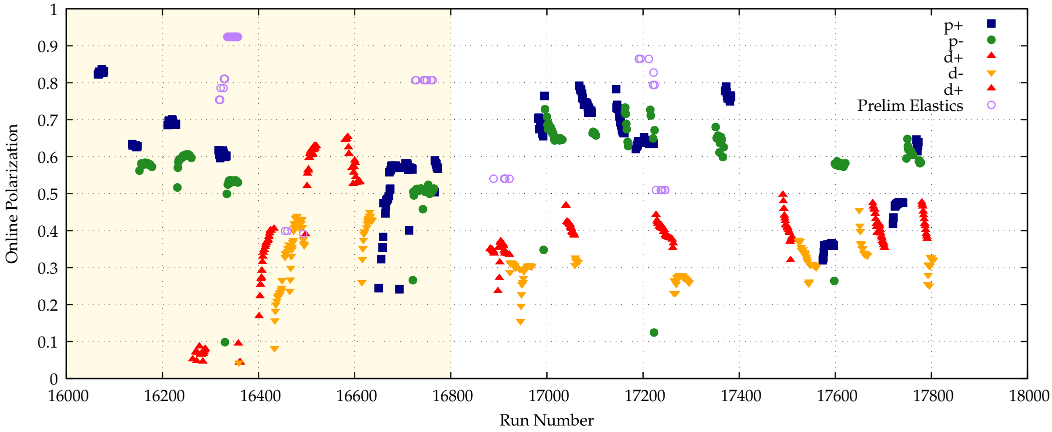
Proton NMR (open) vs. Scattering (filled)

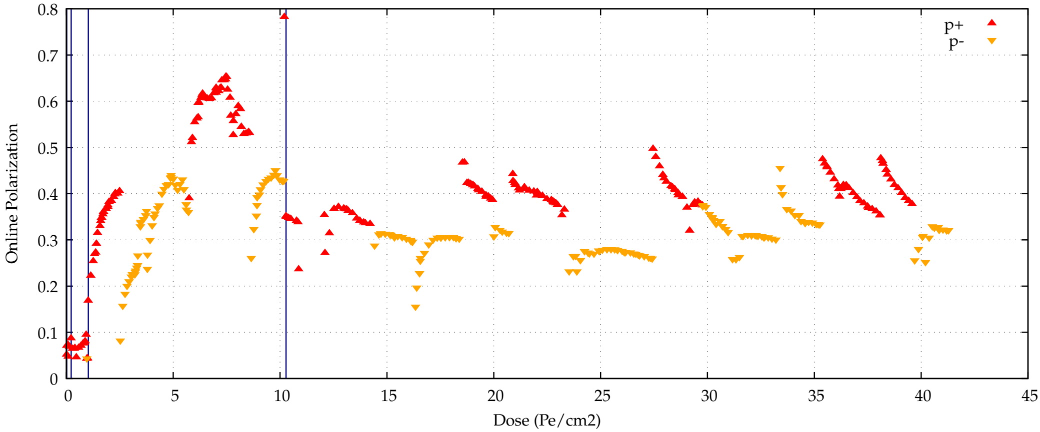


Charge-averaged Online Polarization Performance by Run



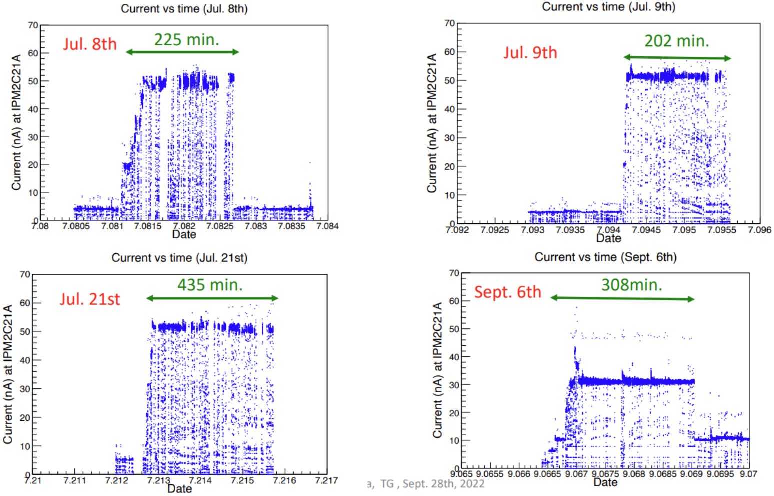
Shaded region: poor NMR due to smaller cell size, misalignment, or cold board failure. Prelim points from N. Pilleux.

Deuteron Polarization Performance Vs. Dose



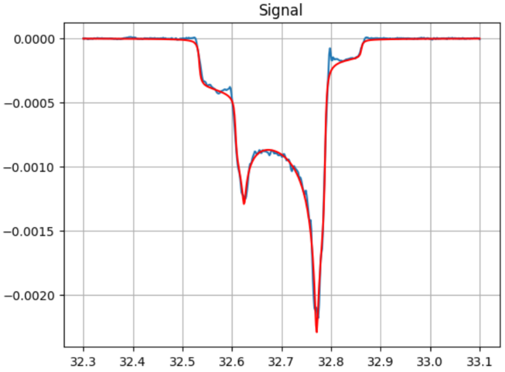
Polarization analysis ongoing. JM, D. Seay, I. Fernando, P. Pandey.

Currents during Irradiations (from IPM2C21A, BPM)

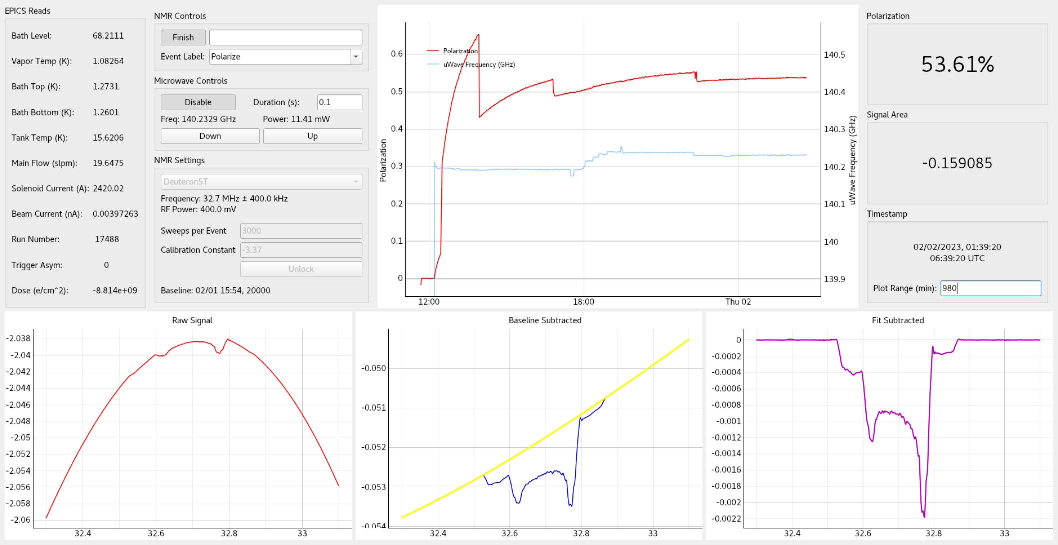


Record In-Beam Deuteron Polarization

- After running 1 ND_3 sample from June to November (20 mm cell), magnet power supply failed
- Roughly 22 Pe/cm^2 accumulated
- Sample was stored in LAr from November to January
- Moved to a 15 mm cell
- Started polarizing during a beam down, so no rush to start data taking
- 6 hours later, polarization $\sim$ 54%

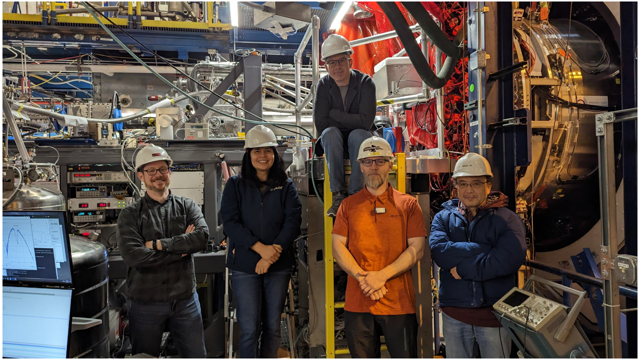


Preliminary Results



Performance Summary

- Overall, a great deal of hard work paid off.
- Excellent deuteron, OK proton polarization. Lots of good data!



RGC was a huge effort by a large group.
Thanks to all!

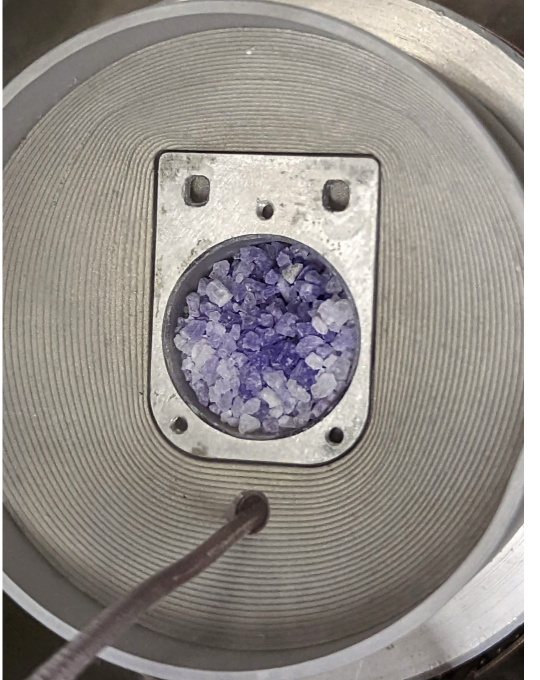
JLab Staff:

- **Target Group**: J. Brock, C. Carlin, C. Keith, J. Maxwell, D. Meekins, T. Kageya, P. Hood, M. Hoeger, D. Griffith
- **Fast Electronics, Survey, Radcon, Hall B**

RGC Collaboration:

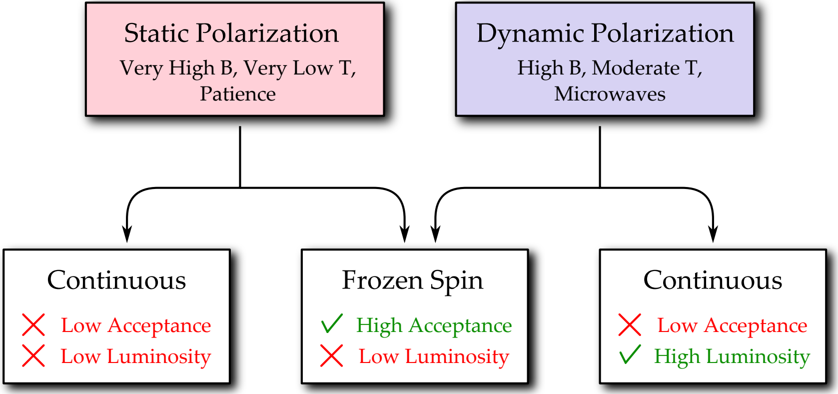
- **Spokepersons and Users**
- **Especially Graduate Students**: V. Lagerquist, P. Pandey, D. Holmberg

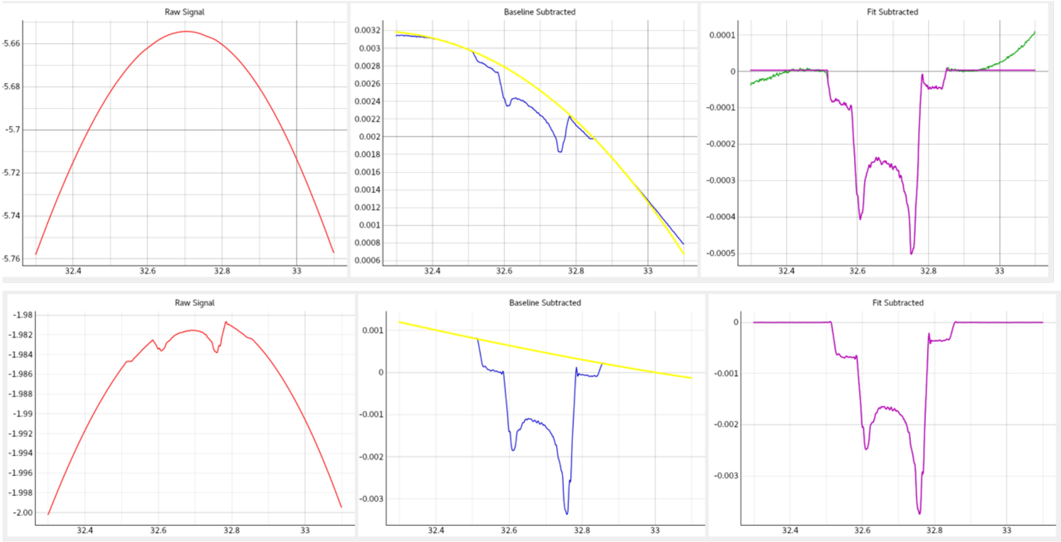
Thank you for your attention!

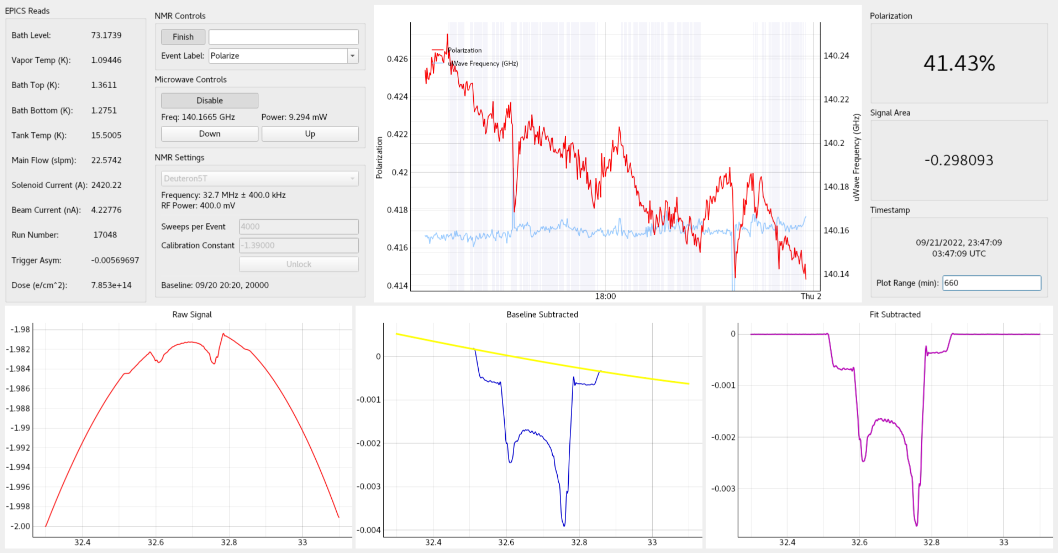


Solid Polarized Target Options for Experimentalists

- High B field → large magnet; access can be occluded
- Very Low T → large refrigerator; heat load is limited so beam current is limited




Raw Signal
-5.66
-5.68
-5.7
-5.72
-5.74
-5.76
32.4
32.6
32.8
33
Baseline Subtracted
0.0032
0.003
0.0028
0.0026
0.0024
0.0022
0.002
0.0018
0.0016
0.0014
0.0012
0.001
0.0008
0.0006
Fit Subtracted
0.0001
0
-0.0001
-0.0002
-0.0003
-0.0004
-0.0005
Raw Signal
-1.98
-1.982
-1.984
-1.986
-1.988
-1.99
-1.992
-1.994
-1.996
-1.998
-2.00
Baseline Subtracted
0.001
0
-0.001
-0.002
-0.003
Fit Subtracted
0
-0.001
-0.002
-0.003


EPICS Reads
Bath Level: 73.1739
Vapor Temp (K): 1.09446
Bath Top (K): 1.3611
Bath Bottom (K): 1.2751
Tank Temp (K): 15.5005
Main Flow (slpm): 22.5742
Solenoid Current (A): 2420.22
Beam Current (nA): 4.22776
Run Number: 17048
Trigger Asym: -0.00569697
Dose (e/cm^2): 7.853e+14
NMR Controls
Finish
Event Label: Polarize
Microwave Controls
Disable
Freq: 140.1665 GHz
Power: 9.294 mW
Down
Up
NMR Settings
Deuteron5T
Frequency: 32.7 MHz ± 400.0 kHz
RF Power: 400.0 mV
Sweeps per Event 4000
Calibration Constant -1.39000
Unlock
Baseline: 09/20 20:20, 20000
Polarization
uWave Frequency (GHz)
18:00
Thu 2
Polarization
41.43%
Signal Area
-0.298093
Timestamp
09/21/2022, 23:47:09
03:47:09 UTC
Plot Range (min): 660
Raw Signal
Baseline Subtracted
Fit Subtracted