Solid Polarized Target Primer for g_2^p Target Operators

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1   Overview of the Mechanism

Our Dynamically Nuclear Polarized (DNP) Target can provide greater than 90% proton polarization in an irradiated ammonia target sample, in a 5T magnetic field and at around 1K. The basic operating principle involves leveraging the spin-spin coupling of free electrons to the protons we wish to polarize. In a magnetic field, spin-spin coupling results in hyper-fine splitting, as seen in the figure 1.
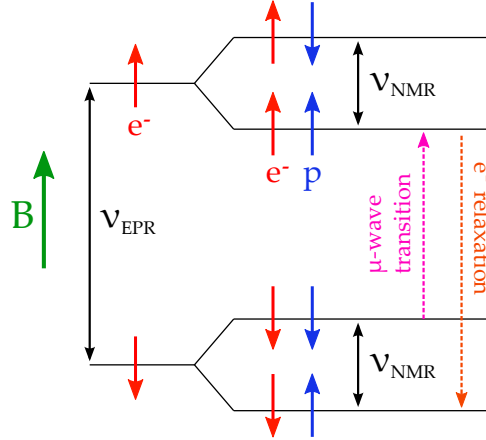

Figure 1: Energy diagram of DNP spin flip transitions.

Using microwaves of wavelengths corresponding to the energy gaps seen in the diagram, transitions can be induced to flip the spin of the proton along with the spin of the electron. As shown, the (down,down) state can be flipped to the (up,up) "aligned" state using microwaves, but by changing the microwave frequency it is also possible to flip the (down,up) state to the (up,down) state, *thereby allowing us to anti-align the proton without changing the magnetic field.* Thus both positive and negative polarizations are available using the same field.

Since the relaxation time of the electron at 1K is on the order of milliseconds, compared to the proton's tens of minutes, the same electron can be used to polarize many protons. The proton polarization is transferred away from the immediate vicinity of the free electrons via spin diffusion.

2 Thermal Equilibrium Polarization

The starting point for our technique is quite simple, but still crucial to the operation of the target. By placing our material in a high magnetic field B and at low temperature T and waiting for the material to reach thermal equilibrium, we can expect from Boltzmann statistics that our polarization should be:

$$P_{TE} = \frac{e^{\frac{\mu B}{kT}} - e^{\frac{-\mu B}{kT}}}{e^{\frac{\mu B}{kT}} + e^{\frac{-\mu B}{kT}}} = \tanh\left(\frac{\mu B}{kT}\right). \tag{1}$$

If we assume a 5T field and 1K temperature, comes out to a proton polarization of around 0.3%. We also note that the electrons, whose magnetic moment is 660 times that of the proton, have near 100% polarization.

While 0.3% proton polarization is obviously not practical for experiments, this starting point will provide a crucial point of calibration as we attempt to measure the polarization with NMR.

3 Dynamic Nuclear Polarization

DNP is the enhancement of polarization via the excitation of spin-spin transitions with microwaves. There are 5 basic ingredients which go into this technique: a high magnetic field, a low temperature, a microwave system, an NMR system, and a suitable target material. We will briefly walk through these 5 building blocks, and what systems are necessary to provide them in the experimental hall.

3.1 Magnetic Field

The magnetic field is provided by a superconducting split pair magnet capable of producing 5T at great (10^{-4}) uniformity in a $3 \times 3 \times 3$ cm^3 volume at the target cell. This magnet's open geometry allows for beam to pass at both parallel and perpendicular to the field. An Oxford Instruments power
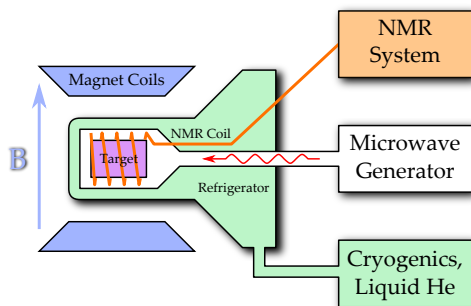
Figure 2: Overview of required systems for DNP.

supply provides the necessary current (around 80A at 5T), and controls the modes of operation. During g2p, the magnetic field will be set at 5T or 2.5T. A reservoir of liquid helium at 4K keeps the magnet superconducting.

The two operating modes are *connected* and *persistant*. A superconducting switch, which is simply a length of superconducting wire near a heater coil, changes these modes. In *connected*, the switch is "opened" by heating the superconducting wire until it is resistive; this "connects" the magnet coils to the leads of the power supply connected on either side of the switch. In *persistant* mode, the switch is allowed to cool; as the wire again becomes superconducting, the relatively high resistance of the power supply leads makes it "invisible" to the current in the superconducting coils.

Magnet energization and de-energization may only be performed by a target expert. The ramp up and down must be carefully controlled to avoid losses in superconductivity called *quenches*.

While "Magnet On" signs should make it clear, the large stray field of the magnet can be dangerous. Work in the hall should be carefully monitored, and the magnet may need to be ramped down. Safety first!

3.1.1   Shim Coils

The uniform field region can be tuned using a set of shim coils, but in practice this is not necessary. However, leaving the shim coils superconducting while the main coils are being energized will lead to trouble. Unless the shims are held at zero current by their power supply, the current induced from the main coil's energization can result in shim quenches in which too much shim

current causes a failure in superconductivity.

3.2 Low Temperature: Cryogenics

The target's temperature directly affects the efficiency of polarization. The temperature's effects are easily seen in a graph of polarization over time. As inevitable beam trips occur, the polarization rises as the heat load of the beam is removed. When the beam comes back, the polarization drops a few percent simply due to the heat load. An example diagram of the pumps and fridge which create and maintain the low temperature is shown in figure 3, although the precise pumps used in the hall are slightly different.

The fridge, magnet, and liquid nitrogen shield are hung in the *target can* which is held at vacuum by a diff pump. The liquid nitrogen shield, seen in the diagram in green, protects the liquid helium components from heat radiation from the room temperature outer vacuum can.
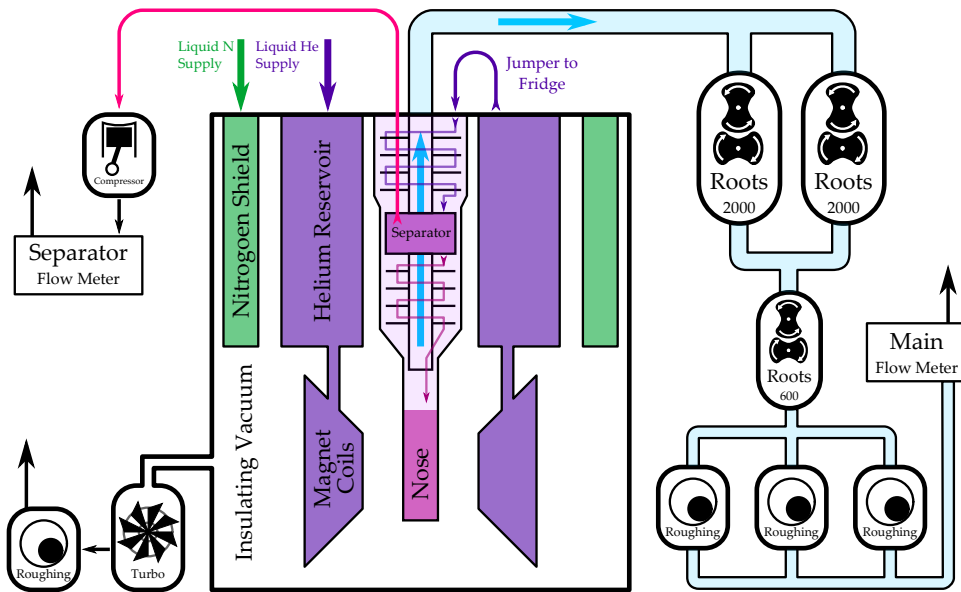

Figure 3: Example pump diagram, showing the flow of cryogens through the refrigerator and out the roots pumps.

You can follow the path of the cryogens in the figure 3. In the refrigerator, the *separator pump* pulls liquid helium from the magnet reservoir into the

separator. The separator can be thought of as a holding reservoir for colder liquid helium before it is transferred to the *nose.* The nose is bottom of the fridge, where the target material is held. As liquid helium is transferred from the separator to the nose, it passes through heat exchangers which are cooled by evaporating helium being pumped out of the fridge. To maintain the low temperatures despite as much as 1W microwave and beam power dumped into the target, huge roots blower pumps work to maintain a low pressure in the refrigerator. These pumps pull the evaporated helium up, past the heat exchangers and baffles, and out of the fridge.

Maintaining the level of liquid helium in the nose is critical to the operation of the target, and monitoring this level is one of a target operator's tasks. The level of liquid helium must remain above that of the target cups or the polarization will be lost and the material may be melted by the beam. The liquid level should be adjusted automatically by a PID loop, but it's crucial to watch this level to ensure the loop is working.

These are the key indicators of the refrigerator to watch, and the two valves we use to control them during normal running:

- "Main Flow": This indicates the flow of gas being pulled out of the nose of the fridge by the big pumps. This flow will indicate the heat load on the target in the form of boil off gas flow.

- "Run Valve": This controls the flow of liquid helium from the separator into the nose, and therefore is used to maintain the level of liquid helium over the target cups. Generally controlled automatically by PID loop.

- "Nose Level": This indicates the level of liquid helium in the nose.

- "Separator Flow": This is the flow of gas being pulled out of the separator, which acts as a buffer of cold helium to send to the nose. This flow is what pulls liquid helium from the magnet into the fridge.

- "Separator Valve": Controls the separator flow, used to keep liquid helium in the separator. In general, we need a balance between the main flow and the separator flow to ensure the fridge isn't being emptied by more being pumped out (main flow) than is being pumped in (separator flow). Generally controlled automatically by PID loop.

- "Separator Level": This indicated the level of liquid helium in the separator.

3.3 Microwaves

The microwaves induce the spin flip transitions and must be tuned very carefully to the frequency of the energy gap to maximize polarization. Unfortunately, as the target material accumulates radiation damage from the beam, this optimal frequency changes. **The most important task of the target operators is to constantly monitor and tweak the microwave frequency to maximize polarization.**

The microwaves are provided by the EIO tube, which allows the frequency of microwaves to be changed within limits by adjusting a bellows on the oscillation cavity. Wave guides carry the microwaves from the tube to a horn which shine on the target cups. The picture in figure 4 shows the gold horn above (to the right of, here) the two target cups on the new target inserts.

In g2p, two magnetic field strengths (5T and 2.5T) will be used and thus two nominal microwave frequencies will be used (140 and 70 GHz). This requires two different EIO tubes, but the operation will be similar.

Figure 4: The ladder of the new target inserts, showing both target cups and microwave horn.

3.4 Target Material

Choosing a target material is a compromise between our desire for a pure proton target and the practical necessities of materials which perform well under DNP and heavy radiation damage. Doped ammonia (^{14}NH$_3$) has emerged

as the most attractive materials for our uses. When doped with paramagnetic centers to provide free electrons for the spin-spin coupling, ammonia can achieve greater than 90% proton polarization.
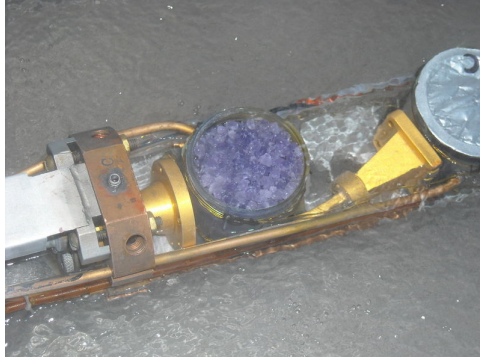

Figure 5: Material after removal from the beam during SANE.

To dope the ammonia with free electrons, it is irradiated in a smaller accelerator before it comes to JLab. This irradiation produces radicals such as NH_2 from the NH_3 in what is called a *warm dose*. In the beam at JLab, temperatures are much lower, and different radicals, such as atomic H, are produced under this *cold dose*.

Each target insert holds two cups with ammonia material samples. The cups are cylindrical, and roughly 1 inch in diameter and length. For g2p, the same microwave guide will be used for both top and bottom cups; the photo in figure 5 shows the old insert cups from a previous experiment.

3.4.1 Anneals

The number of paramagnetic radicals in the material must be carefully balanced to achieve the greatest polarization. Although the free electrons from these paramagnetic centers are necessary to polarize via DNP, they also allow polarization decay short-cuts for the aligned protons. As radiation dose from the beam produces more radicals than needed, the DNP process becomes less efficient and the polarization will fall. However, by heating the ammonia, we can allow some of these radicals to recombine. This heating is called an *anneal*, and for an experiment that runs at 80nA beam current, will

likely be necessary every day. After an anneal, the polarization can again reach maximal levels.

There is a limit to the lifetime of the ammonia however. As successive anneals are performed on a material sample, the decay rate of the polarization will increase, requiring more anneals per day. This is due to the buildup of radicals which cannot be recombined in an anneal. Eventually, the polarization decay rate will be so fast that it is no longer practical to use the material, so a new ammonia sample will be used. This replacement of material will occur once a week.

3.5   NMR

The NMR system is used to measure the proton polarization in the sample, and operates by observing spin flips of the proton at its Larmor frequency. By embedding the inductor of an LCR circuit in the target material, we can detect energy lost or gained in the circuit as a function of the circuit's frequency. A loss of energy in the circuit near the proton's Larmor frequency would indicate the *absorption* of energy as its spin is flipped to be anti-aligned with the magnetic field. Likewise, a gain of energy in the circuit would come from a proton *giving up* energy as it becomes aligned. This gain or loss is visible as a dip or peak in the NMR signal versus frequency. The area under this dip or peak is a proportional measure of the proton polarization in the material.

3.5.1   Baselines

To accurately measure the area of the NMR signal's dip or peak which is due to the polarization of the proton, we must carefully exclude any systematic changes in the NMR signal which are not due to polarization. To do this, we take a *baseline* measurement of the circuit's response **without** the polarization signal. This can be achieved by shifting the Larmor frequency of the proton out of range of our signal by changing the magnetic field. The baseline NMR signal is very sensitive to minute changes in the NMR circuit, and it it important to make frequent baseline measurements to ensure an accurate polarization measurement. A baseline should be taken at least every day after an anneal.

In addition to subtracting the baseline signal, a polynomial fit is performed to *wings* the NMR signal. This polynomial fit subtraction should
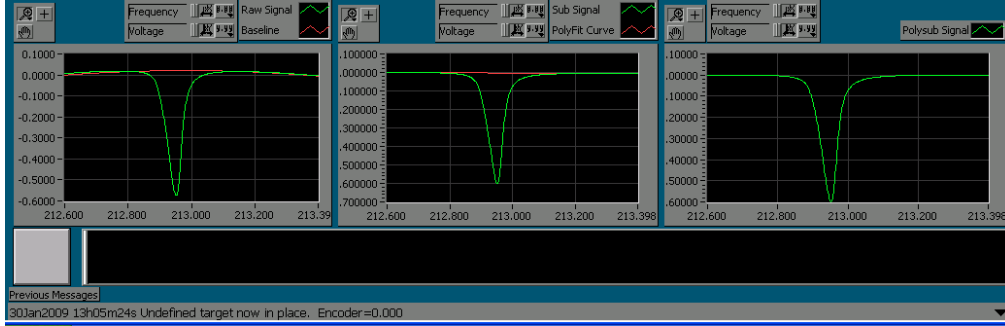
Figure 6: NMR signal integration panel of the NMR control program. Here the polarization is positive and enhanced greatly. The first panel shows the raw signal in green and baseline in red. The second shows the baseline subtracted and polynomial fit, and the third panel shows the final NMR signal to be integrated.

remove any residual baseline signal and leave only the signal due to the target polarization.

3.5.2 Thermal Equilibrium Measurements

To calibrate our polarization, we must discover the proportionality factor, or *calibration constant*, which relates area under the NMR dip or peak to the proper polarization. To do this, we take advantage of the known polarization when the sample is at thermal equilibium. After forming the calibration constant using this static, known polarization and the measured NMR area, we can apply this constant when the target is being dynamically polarized with microwaves.

A thermal equilibrium measurement (or TE) requires removing the beam and the microwaves, setting the pressure and temperature in the nose to be as constant as possible, and waiting for the NMR area to stabilize. The relaxation time of the polarization depends on the temperature, so the temperature is raised above 1K to decrease the time spent waiting. Even so, this will likely take as much as an hour per cup. The number and quality of the thermal equilibrium measurements directly affects the error on the target polarization measurement, so the TE should not be rushed! In experimental circumstances, the pressure to hurry and get back to taking beam

9

can result in sloppy TEs which adversely affect the experiment's systematic error. Take time to be accurate; time has been budgeted to allow for these TE measurements.