Characterizing ^{3}He Nuclear Spin Relaxation in Vessels of Glass and Metal

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Abstract

Historically, high-pressure gaseous polarized ^{3}He targets at Jefferson Lab (JLab) have utilized "target cells" fabricated entirely out of glass. With the 12 GeV upgrade nearing completion, experiments requiring significantly higher luminosities are planned, and to meet the challenge, it is desirable to produce metal end windows through which the electrons will enter and exit the target cell. The polarization technique used in ^{3}He targets at Jefferson Lab utilizes spin-exchange collisions between ^{3}He atoms and alkali-metal atoms polarized through optical pumping. Unfortunately, relatively few studies have investigated the spin relaxation of nuclear-polarized noble gases on metal surfaces, particularly under the conditions in our targets. We have tested various cells incorporating both glass and metal and have found that acceptable spin-relaxation rates can be obtained by electroplating gold coatings on OFHC copper substrates. Initial tests using titanium substrates, an alternative material for end windows, have not yet been as successful as copper ones. Various types of glass (borosilicate glass, aluminosilicate glass, and uranium glass) were used in the construction of these cells. These studies have produced multiple cells that demonstrate the viability of ^{3}He targets incorporating metal windows into their design.

To you who comes next, may this thesis serve as a bridge to bigger things.

The Bridge Builder

By Miss Will Allen Dromgoole

An old man, going a lone highway,
Came at the evening cold and gray,
To a chasm, vast and deep and wide,
Through which was flowing a sullen tide.
The old man crossed in the twilight dim,
That sullen stream had no fears for him;
But he turned, when he reached the other side,
And built a bridge to span the tide.

"Old man," said a fellow pilgrim near,
"You are wasting strength in building here.
Your journey will end with the ending day;
You never again must pass this way.
You have crossed the chasm, deep and wide.
Why build you the bridge at the eventide?"

The builder lifted his old gray head.
"Good friend, in the path I have come," he said,
"There followeth after me today
A youth whose feet must pass this way.
This chasm that has been as naught to me
To that fair-haired youth may a pitfall be.
He, too, must cross in the twilight dim;
Good friend, I am building the bridge for him."

[1]

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First and foremost, I would like to thank my advisor, Dr. Gordon Cates. Not only have you taught me more about physics simply through our one-on-one discussions than I probably even realize, you refused to let me ever let myself down. Graduate school was a tremendously difficult time for me personally and no matter how many times I wanted to run away, quit grad school, change fields, transfer schools, or just simply fall off the face of the physics earth, I walked out of your office refreshed and ready to attack our next problem. I am so honored to have worked with you and like so many other people I have encountered, I cannot wait to share all the great stories I have accumulated from our time together.

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Since all these cells are made of glass, I would be amiss not to mention Mike Souza. Other than the fact that without him not a single one of these cells would have been constructed, his never-ending knowledge of glass composition, techniques, and nature helped steer us at every intersection of this journey.

Last I would like to take a moment to thank all those that helped in the production of our glass-to-metal seal assemblies and answering my numerous phone calls to pick their brains about their products. Larson Electronic Glass (specifically Larry Bitz) for sealing the glass to metal for us, Able Electropolishing (specifically Matt Van Acker) for polishing the seal assemblies, Epner Technologies Inc. (Rob Brueggemann and David Epner) for both gold coating the seal assemblies and discussing electroforming.

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1 Introduction

Helium-3 targets used in electron scattering experiments at Thomas Jefferson National Accelerator Facility (JLab) have historically been polarized via spin-exchange optical pumping (SEOP). The cells containing the ^{3}He gas have been composed entirely of aluminosilicate glass due to its exceptional nuclear spin relaxation properties. Most metals, on the other hand, cause a significant amount of nuclear spin relaxation. Due to the free neutron's half life of 10.2 min, ^{3}He is used as an effective neutron target. The ^{3}He nucleus is able to do this since its two protons are have their spins anti-parallel leaving the single neutron inside the nucleus with the remaining spin.

After the 12 GeV upgrade to JLab, experiments are planned that will run at higher beam energies as well as electron beam currents up to 60 μA. It is feared that the glass targets will be unable to survive long enough to collect sufficient amounts of data in such harsh environments. One solution to this problem is to change the target design to incorporate end windows of a more resilient material for the point at which the electron beam enters and exits the cell. Tests on OFHC copper electroplated with gold have presented a viable option due to its structural integrity, nuclear relaxation properties, and its familiarity with manufacturers.

In the work described here, the nuclear spin relaxation of 19 cells with various geometries and material composition have been characterized. These cells tested various different glasses, metals, and cell geometries. Ultimately, a lifetime (the time constant associated with the spin relaxation of the helium nuclei) of 15.6 h was obtained by using a Pyrex cell with a 5" long by 1" outer diameter copper tube oriented horizontally. The inner surface of

this tube was electroplated with gold. Since the fractional amount of metal used in a target cell will be significantly less than that used in these cells, targets would be predicted to have lifetimes on the order of 100 h, still sufficiently long for performing an experiment.

2 Theory

When a polarized helium nuclei approaches a surface, the nuclei can either interact with the surface or the inner bulk material. If the surface is porous, it allows polarized gas to penetrate, bounce around, and return to open space. Various types of glass fall into this first category, interactions with metal on the other hand, are of a different type. Metals can be expected to be more impenetrable to the polarized gases, and so the relaxation can be expected to occur at the surface of the metal. This difference makes the cells in this study quite distinct in that they provide insight into materials whose spin-relaxation properties are not well known.

2.1 Relaxation from Glasses

Since the helium can penetrate the surface of many glasses, it is able to enter the bulk material, and while inside, may encounter various paramagnetic sites, dangling bonds, or impurities that cause relaxation. The origin of these relaxation sites in glass and their associated mechanisms still remain poorly understood due to glass' complexity. Fitzsimmons et al. [2] showed that by switching to glass melts that were less porus to helium than Pyrex, such as aluminosilicate glass, relaxation was reduced. The nuclear relaxation is then presumably largely limited to surface relaxation rather than permeating into the bulk of the aluminosilicate glass. Timsit and Daniels developed two distinct relaxation mechanisms: absorption that happens in the bulk material and adsoption that occurs at a surface of a material [3]. They derived the expected lifetime due to absorption, T_{absorb}, for a spherical cell of diameter d containing N free helium nuclei. To do this, one must first calculate, τ, the

time for a helium nucleus to jump between successive layers of glass that have a thickness of δr using the equations below:

$$T_{absorb} = \frac{6\tau}{\beta N_{impurity}} \frac{N}{n_g} \tag{1}$$

$$\tau = \tau_0 \exp(E_d/kT) \tag{2}$$

$$n_g = \frac{6NkT\delta rS}{d} \tag{3}$$

Where $N_{impurity}$ is the impurity fraction, n_g the number of absorbed helium nuclei, β is the number of atoms inside the glass that surround the dissolved helium nuclei, and S is the helium solubility of the given surface material.

Cells that are polarized using spin-exchange optical pumping will also benefit from the fact that as the rubidium in the cell is heated, a film of alkali metal will deposit itself on the walls of the glass. Relaxation due to this adsorption process on metal should rise proportionally to $T^{3/2}$ according to Denninger et. al. [4]. This coating was utilized by Heil et al. [5] to create an increase in cell lifetimes which they concluded was from both the substrate filling microscopic pores in the glass, thus blocking diffusion, and having a low adsorption energy, decreasing helium sticking times at the walls. This sticking time, τ_s, follows Frenkel's Law:

$$\tau_s = \tau_{s,0} \exp(E_{ad}/kT) \tag{4}$$

The associated lifetime for this adsorptive relaxation also can be found in Timsit, Daniels, and May's paper [3]:

$$T_{adsorb} = \frac{NT_i'}{N_{impurity} n_g'} \tag{5}$$

$$n_g' = \frac{3N\bar{v}\tau_s}{2d} \tag{6}$$

Here T_i' is the intrinsic relaxation time of a nuclei near a paramagnetic site on the surface, $\bar{v}$ is the mean velocity of the nuclei, and n_g' is the number of adsorbed helium nuclei.

Yet another way to limit this absorbtive relaxation of permeable glasses is by coating the inner surface with sol-gel [6]. Sol-gel is a mixture of aluminum nitrate nonahydrate $[Al(NO_3)_3 9H_2O]$, ethanol, and deionized water. After applying a coating to a glass and baking it to solidify the sol-gel, the sol-gel limits helium diffusion into the bulk glass when properly prepared. No cells in these studies were coated with sol-gel.

2.2 Relaxation from Metalic Surfaces

Adsorption relaxation is relaxation that occurs purely at the surface of a material. Relaxation from metals will be dominated by adsorption relaxation rather than absorptive relaxation. Two additional types of relaxation distinctive to metals also exist, relaxation from Eddy currents and relaxation due to Korringa scattering. The relaxation from Eddy currents and Korringa scattering have been derived in many places but what follows below is from the thesis of Ian Nelson from the University of Wisconsin [7] [8] [9]. The relaxation from Eddy currents are the result of an induced current on the surface of the metals and can be minimized with a stable magnetic field.

Korringa scattering is a much more exotic type of relaxation that is a result of the electronic nature of metals. A polarized nucleus can become depolarized by exchanging its spin with a conduction band electron through a hyperfine interaction similar to that used to polarize helium in SEOP. In order to conserve angular momentum, the interacting electron from the metal's Fermi sea must undertake a spin flip. This event is dictated by statistical

mechanics and thus an electron can undergo the hyperfine interaction should it be within kT of E_F. The rate of Korringa relaxation then can be found to be [8]:

$$\frac{1}{T_{Korr}} = \frac{(4\pi)^6}{9h^7}(g_s\mu_B\frac{\mu_K}{K})^2\eta^4m^3\epsilon_F kT \tag{7}$$

Calculations done by Nelson estimate cells limited by Korringa scattering would display lifetimes on the order of thousands of years for helium but for xenon polarization, the lifetimes would be on the order of hours to tens of hours depending on the metal. It is rather unlikely that the limiting relaxation mechanism for these cells falls to Korringa scattering. A useful way to compare absolute surface relaxation rates is through the "relaxivity," ϱ, a fundamental property of the surface. This quantity enables a comparison of various materials as well as a way to extrapolate from one sized cell to another:

$$\frac{1}{T_{Surface}} = \varrho\frac{A}{V} \tag{8}$$

For relative comparison, Nelson reports predicted relaxivities for gold (1.07×10^{-4} cm/s), silver (3.76×10^{-5} cm/s), and indium ($1.13 \times 10^{-}5$ cm/s) and measured values of 8.45×10^{-5} cm/s, 4.55×10^{-5} cm/s and 6.63×10^{-5} cm/s respectively.

Work done by Denninger et al. [4], characterized various surfaces that were not the usual alkali metals found in polarized gaseous cells. The metals coated the inner surface of various glasses. Gold was found to display a 20 h lifetime, comparatively long to other metals in the study: Al (6 h), Ag (5 h), titanium nitride (2 h), and Al_2O_3 (4 h) among others. Their result suggests a gold-coated target window, possessing a relatively small surface compared to the entire cell's, would display small enough relaxation to be incorporated in future targets.

3 Apparatus

The apparatus used in this experiment has been described in great detail in previous publications [10, 11, 12]. Pulse NMR was chosen for these studies because it is a much more localized type of NMR, whereas adiabatic fast passage, AFP, flips the entire amount of nuclear spins in the cell. With a more localized technique, the segments of the cell that contained metal were avoided, minimizing Eddy currents or other magnetic feedback. The pNMR technique allowed the polarization of the cells to be measured in segments where no metal was present because of this localization.

In general, the cells used in these experiments were composed of glass and metal as can be seen in Figure 8. The spherical top segment of the cell, the "pumping chamber," was placed inside an oven and was heated in order to increase the number density of rubidium vapor in the cell. This oven had multiple windows through which the laser light passed to polarize the vapor. A cylindrical segment of resized glass, known as a "transfer tube," was used to permit polarized gas to diffuse from the pumping chamber to lower segments of the cell. A target cell would have a "target chamber," where the electron beam passes through, at the other end of this transfer tube. These test cells had a glass-to-metal-to-glass seal in place of the target chamber.

3.1 Coils and Magnetic Field Production

Three different types of coils were used in this experiment: a pNMR solenoid coil wrapped uniquely for each cell, the Helmholz coils for the main holding field, and three Maxwell coils to produce field gradients.

3.1.1 pNMR Coils

The pNMR was a solenoid style coil, hand-wrapped, and soldered to a BNC connector. Typically, only one such coil was wrapped on the transfer tube. This coil was placed approximately 2 inches below the bottom of the oven. The majority of cells used here had the pNMR coil wrapped on the vertical section of transfer tube with a typical diameter of 2.84 cm. For these studies, the coils typically had r = 1.42 cm, L = 2.54 cm, R = 0.3 Ω, L= 0.008 mH, and 40-50 turns of 20 gauge copper wire.

3.1.2 Main Holding Field

The main holding field was produced via a set of large Helmholtz coils. The coils have an approximately radius of 0.66 m, 240 turns of guage 6 square magnet wire, and a separation of 0.66 m. They were driven by a Walker power supply to produce around 19 G at the center of the oven. A shunt resister with 5 mV/A output was connected in series with the coils in order to measure the current through the coils. This value was typically 5-7 A for these studies.

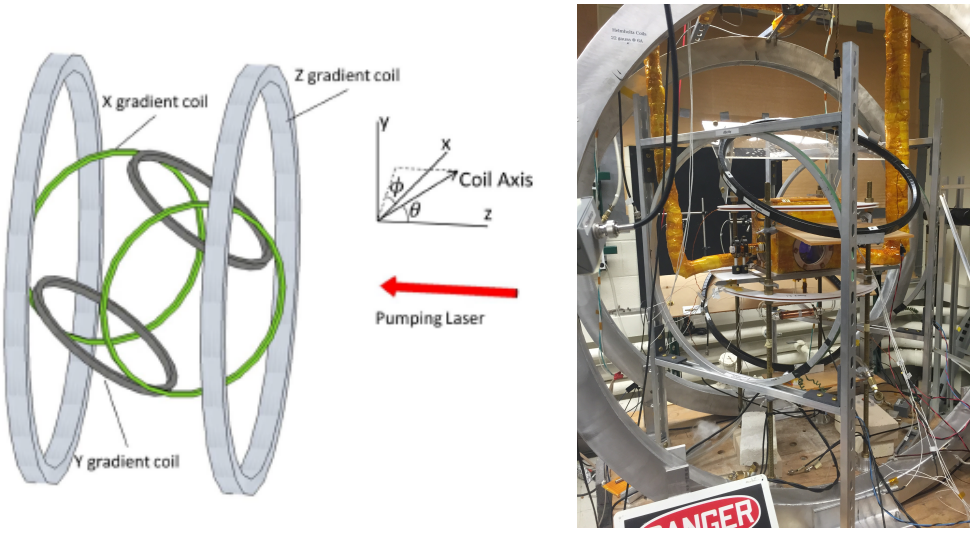

Figure 1: A diagram [10] and photo of the coil setup.

3.1.3 Gradient Coils

Three gradient coils were used in these studies to eliminate inhomogeneity in the proximity of the pNMR coil. All three sets of gradient coils were Maxwell coils that have been wound in opposite directions as to create inhomogeneity without altering the field magnitude [10].

The "Z-Gradient Coils" were wound directly on top of the Helmholz coils to produce gradients in the z-direction, which in our lab is in the horizontal direction along the path of the laser beam. This gradient coil had a r= 66 cm. It was powered by a power supply in voltage control mode with approximately 1.5 V output.

The $\frac{\delta B_z}{\delta x}$ coils, also known as the black coils, were used to create inhomogeneity in the x-direction. The coils themselves were oriented 54.7° from the laser light axis and had a 33 cm radius. The $\frac{\delta B_z}{\delta y}$ coils, also known as the green coils, were used to create inhomogeneity in the y-direction and were oriented 54.7° relative to the laser light axis but the opposite direction of the black coils and had a 28 cm radius.

3.2 Electronics Overview

A polarization measurement was initiated with a trigger pulse from an Agilent function generator that could be manually or remotely activated via LabView program. This trigger pulse caused another Agilent function generator to send a pulse length of 400 microseconds at 56.6 kHz to the aforementioned hand-wrapped pulse NMR coil located on the cell transfer tube. The pulses were typically about 1 V peak-to-peak. A matchbox, containing passive components such as capacitors, was used in order to impedance match to the Agilent's 50 Ohm output.

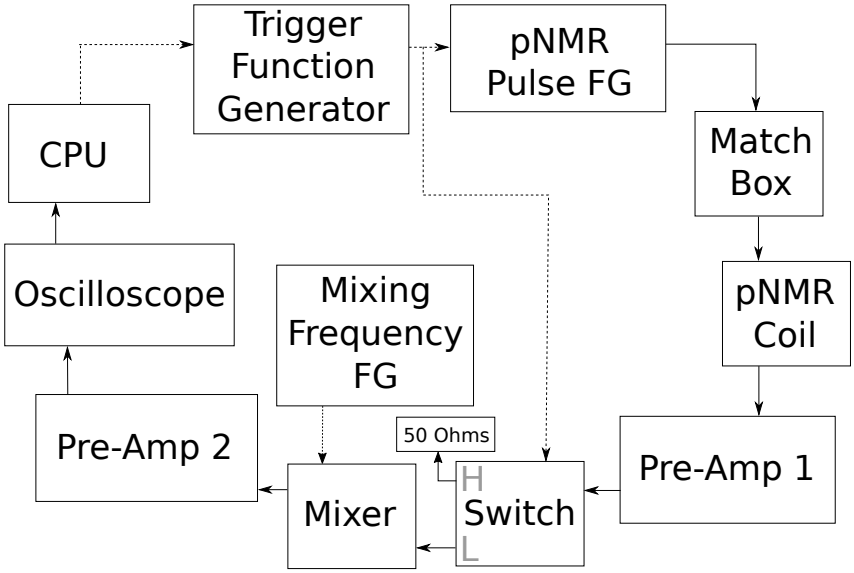

Figure 2: A diagram of the electronics used in these studies. Solid lines define the path of the pNMR pulse and data path while dotted lines are other electronic outputs.

The same coil used to excite the nuclear spins of the gas was also used as the receive coil in this experiment. The signal detected by the recieve coil was then fed into the first SRS pre-amplifier with a band pass filter range of 30 kHz -100 kHz and a gain of 200. The

signal next passed through an electronic switch that was gated to only collect data once the excitation pulse had decayed away. An electronic mixer then was used to mix the signal with a sinusoidal output from an Agilent function generator that was approximately 70 Hz higher than the helium nuclei's resonant frequency of 56.6 kHz at our holding field strength. The mixed-down signal then was sent through one final SRS pre-amplifier with a filter of 30 Hz - 100 Hz and gain of 50 to select the difference of the two mixed frequencies at around 70 Hz, and the output of the second preamp was then sent to a Tektronix Oscilloscope.

A computer was used to record the free induction decay (FID) from the oscilloscope in order to store and analyze the data. This procedure was then performed however frequently was desired over an extended period of time. These FIDs were fit to an exponentially decaying sinusoidal curve using a LabView program. A plot of the fitted FID amplitude versus time was then used to extrapolate the time constant of either the polarization or depolarization curves to be discussed in the next paragraph. A typical FID is shown below.

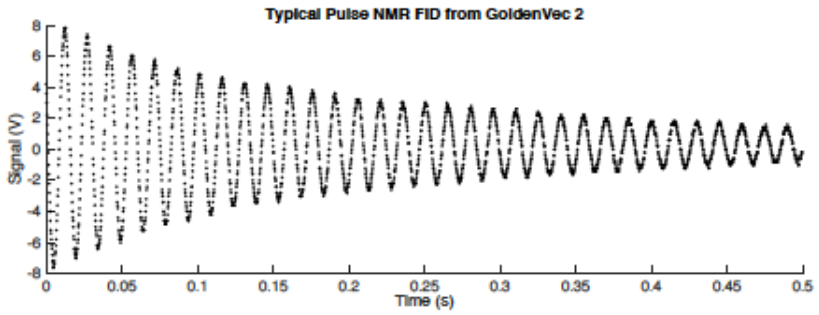

Figure 3: A typical FID signal taken during GoldenVec2's tests with T_2 of about 150 ms.

There are two main types of data collection for these studies, a "spin up" and a "spin down." During a spin up the polarization of the helium nuclei in the cell increases, while during a spin down the nuclei are allowed to depolarize. The curves plotting the polarization

signal as a function of time describe or characterize the polarization of the cell. During a spin up, the oven in which the cell's pumping chamber was placed was heated up to 170°C to increase the amount of rubidium vapor present. Lasers were turned on to polarize the alkali vapor. These polarized rubidium atoms then exchanged their electronic spin via hyperfine interactions to the helium nuclei. Data are periodically collected and fit a curve of form: $P(t) = P_0 * e^{-t/\tau} + P_\infty(1 - e^{-t/\tau})$. To perform a spin down, the oven was cooled to room temperature, the lasers were shut off, and data are then collected. The spin down data fit a curve of form $P(t) = P_o * e^{-t/T_1}$. Example plots of these two types of spins are shown in Figure 10 on page 29.

3.3 Laser Overview

The high power, diode-laser arrays used to polarized the cells were from two different companies, Newport and Raytum. These lasers produced spectrally narrowed infrared light at 794.8 nm to pump the D1 optical transition of the rubidium vapor. Typical laser powers were in the 15 - 20 W range for any given period of pumping with a full-width half-max linewidth of about 0.2 nm. The laser light passed through SMA-coupled optical fibers and into a coupler that could be used with a chiller to combine multiple laser output together. Earlier studies used the multiple lasers with a combiner, while later studies used a Raytum laser that had a higher total power output.

The light next passed through a set of optics, typically 2 inches in diameter seen in Figure 4. It first went through a convex lens (L1), bounced off a set of periscopic mirrors, and one last convex mirror (L2). These optical elements were utilized for directing the laser spot

onto the cell as well as controlling the beam-spot size.

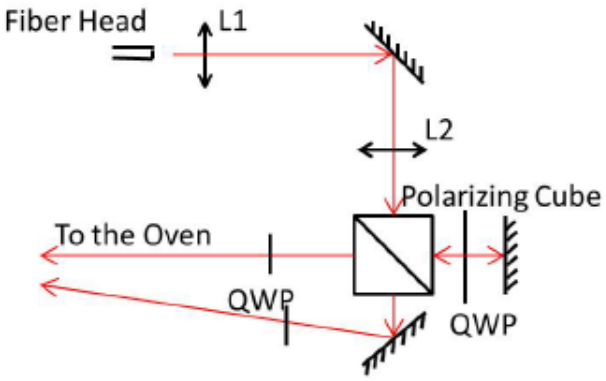

Figure 4: A diagram of our 2" optics setup with main and skew beam paths drawn.

Circularly polarized light was required to drive the correct optical transition for the spin-exchange optical pumping, SEOP, process. The light exiting the second lens (L2) passed through a polarizer cube that separated the two linear polarization states of the laser light, effectively creating two beams called the main and skew beams. The main beam was created by the light reflected at the surface inside of the cube passed through a quarter wave plate, flat mirror, and then once more through the same quarter wave plate (QWP) before re-entering the polarizing cube. Passing the light through this QWP twice gives it linear polarization parallel to the inner plane of the cube. The beam then passed through the cube and yet another QWP to create a circular polarization before traveling approximately 2 m to the oven.

The skew beam was created from the portion of laser light that was not reflected inside of the cube bounced off an angled flat mirror before passing through a QWP and traveling the same two meters to the oven. This beam is called the "skew beam" due to the small skewed angle, relative to the main beam, that it had upon reaching the oven. Polarizations

for both beams have been measured to be in excess of 99% circular polarization.

4 Metal Cell Fabriction

Creating the metal cells used in this study required the collaboration of five different groups. Mike Souza at Princeton University was our glassblower, Larson Electronic Glass produced the glass-to-metal seals used, the University of Virginia's machine shop mechanically polished the metal tubes, Able Electropolishing electropolished the metal tubes, and Epner Technology Inc. electroplated the metal tubes with the desired finished surface. We note that the glass used in these cells were always "resized" from stock material of a different size. This created a smoother surface for the purpose of spin-relaxation. The decrease of nuclear spin relaxation by resizing glass was noted and described in a SLAC publication by Cates [13].

This section will cover the steps taken to produce the glass and metal cells used in these studies. The cells began with a cylinder of the desired metal tube and the desired type of glass attached on either side of the tube to create the glass-to-metal-to-glass seals of the sort shown in Figure 5. The inner surface of each seal assembly was polished in preparation for the electroplating of gold. The seals were then attached to a glass spherical pumping chamber via other segments of tubing called transfer tubes. The constructed cell then was filled with helium-3 gas, nitrogen gas, and rubidium metal before being sealed.

4.1 Glass-to-Metal Seal Assemblies

Glass does not seal hermetically with all metals; different metals require glass types with appropriate coefficients of expansion. Larson Electronic Glass made the glass-to-metal seals for these experiments, and while they are normally used in vacuum applications, we incor-

porated them into our high pressure cells. The glass-to-metal seals themselves are of a type known as Houskeeper seals [14]. The seal assemblies used in the production of our cells are 5" in length and 1" outer diameter with 3" of glass attached at both ends via a Houskeeper seal (see Figure 5). Each cell had a metallic surface area of 101.3 cm^2. Two varieties of metal were used in these studies: Oxygen-free-high-conductivity copper (OFHC copper) and titanium. For the copper seals, there were two main varieties of glass used as well: Pyrex and GE180. GE180 glass does not have a coefficient of expansion that is compatible for sealing to copper and thus required a segment of transition glass called Corning 7052 Kovar sealing glass. This was possible because 7052 Kovar sealing glass has an appropriate coefficient of expansion to bond with both the copper and GE180. In order to create a robust seal, the glass and metal must have similar coefficients of expansions. This is why 7052 was used as a transition glass for GE180, while the Pyrex was able to bead directly to the metal tubes itself [15]. All the titanium cells used Pyrex.

Mechanically, the Houskeeper seal was created by machining down the outer diameter of the metal tube by 0.01" for 0.8" lengthwise at either end of the metal tube where the glass was to be sealed. The feathered down edge was then beaded with 0.2" of glass, either Pyrex or Corning 7052 depending on the desired glass seal [16]. Finally, the resized glass from Mike Souza was spliced to the bead already wetted to metal, resulting in a 1" OD metal tube with 1" OD glass sections on both ends. Once the glass to metal seals are finished, they are returned to the University of Virginia and each seal is leak checked down to the 10^{-10} mbar*l/s level.

Figure 5: OFHC copper glass-to-metal seal from Larson.

4.2 Polishing Process

Once the seals proved to be leak-tight, they were given to the UVa Machine Shop to be mechanically polished. A wire brush was attached to a lathe and spun while inside the tube to polish the inner surface of each glass-to-metal seal assembly. This process gives the metal a more uniform surface to make the electropolishing process more uniform.

The mechanically polished tubes are then sent off to Able Electropolishing. The electropolishing process produces a much smoother surface for the electroplating to be laid onto while also reducing the microscopic surface area that the helium nuclei can encounter. Able Electropolishing removes around 20 μm, which significantly smoothed the mechanical surface of the metal tubes [17].

4.3 Electroplating

Due to gold's more favorable spin relaxation properties, it was desirable to plate the inner surface of the OFHC copper and titanium tubes with gold [4]. This process was completed by Epner Technology Inc. No magnetic materials were used in the electroplating process since they could potentially depolarize the gas in the cell and interfere with the NMR measurements. This requirement made the electroplating more difficult because nickel and chromium (both ferromagnetic) are commonly used as undercoats. To make the 5 μm gold coating more durable, a copper strike (a type of undercoating process) was applied to the inner surface of the copper tubing as an undercoat instead [18].

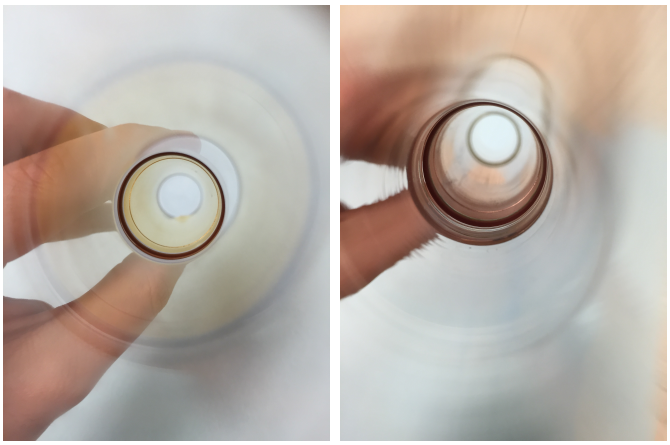

Figure 6: A comparison of a bare and gold electroplated OFHC copper tube.

4.4 Glassblowing

When the gold-coated glass-to-metal seal assemblies were returned to UVa, they were leak checked one last time to insure that the seals had survived all the preparation and shipping between companies. Any impurities that may have been picked up along the way were

then removed from the seals' inner and outer surfaces using an ultrasonic cleaner filled with ethanol, deionized water, and methanol for thirty minutes per chemical.

At this point, the glass-to-metal seal assemblies themselves were ready to be incorporated into a test cell by Mike Souza. At Princeton, all of the glass used was resized to the correct size from smaller, 16 mm, stock tubing. It has been observed that resizing the glass decreases the overall amount of relaxation measured in target cells [13]. This process is believed to reduce the occurrence of micro-fissures that exist in the walls of the glass thus decreasing spin relaxation due to wall collisions. These resized segments were then spliced together to create the string, pumping chamber, and transfer tubes.

A typical cell, made entirely of glass, would be completed at this point and the finished product would be annealed by placing it into an oven to bake the glass. A GE180 based cell underwent a 5 minute ramping period from room temperature up to 780°C. The cell sat at 780°C for 5 minutes, then was left to cool to room temperature for at least 5 hours. This procedure was identical for Pyrex except that the max baking temperature was 565°C [15]. Being exposed to heat for extended periods of time will cause the gold finish to migrate into the metal tubing and also weaken the Houskeeper seals by destroying the oxide layer under the glass. As a result, seal assemblies were attached to the other segments of the cell after this baking period was finished. The complete product was then shipped back to UVa.

4.5 Typical Cell Contents and Fill Procedure

With the exception of one cell, all cells used in these studies contained approximately 700 Torr of helium-3 gas, 70 Torr of nitrogen gas, and less than 1 gram of twice distilled, natural

isotopic rubidium. Only one cell in this study was a "hybrid cell" that contained a 5:1 alkali mixture of potassium to rubidium yet the same gas composition as seen below in the table.

4.5.1 Cell Fill Preparation

The gas system must be prepared 10-15 days before filling a cell. The first step in this task was to get the gas system roughed out and pumped down to a pressure that was safe for the diffusion pump to run. This was done with a mechanical pump, also called the "rough pump" or roughing pump. Once the mechanical pump had been turned on and allowed to pump on itself for 10-15 minutes, the ballast valve on the back was opened, allowing the pump 10-15 more minutes to have its exhaust open to the atmosphere. This allowed gas trapped in the oil chamber to escape, increasing effective pumping rates. After closing the ballast valve, the mechanical pump was used to rough out the entirety of the gas system down to below the 10^{-3} Torr level, including both the backing (foreline) of the diffusion pump and the top of the diffusion pump momentarily. Note that 10^{-3} Torr was chosen because that was the lowest readable pressure by our thermocouple gauges ("TC3" and "TC4" as shown in Figure 7). TC4 was located above the input to the mechanical pump, while TC3 was located in the chamber called the "column" directly above the diffusion pump's gate valve.

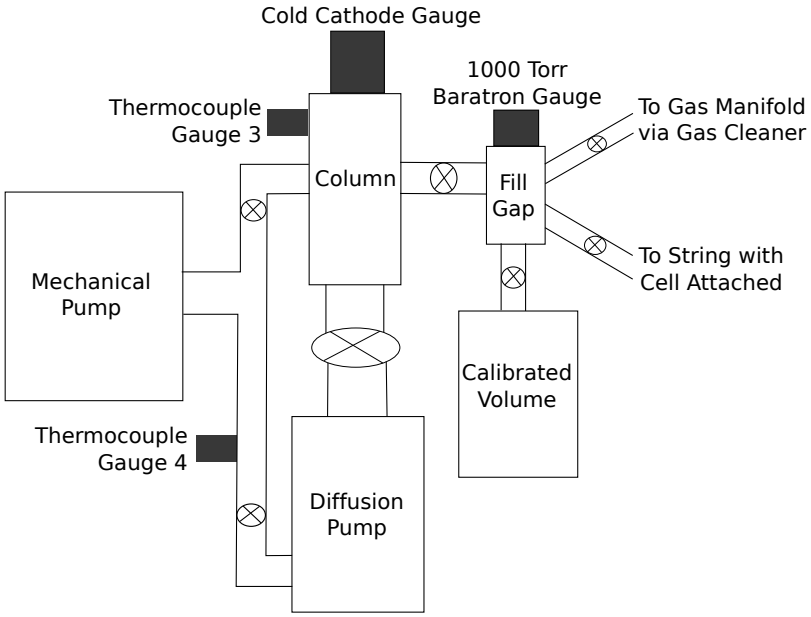

Figure 7: Diagram of the vacuum system used for filling a cell.

With the gas system at a safe pressure for the diffusion pump, the mechanical pump was closed from the system and opened to the foreline of the diffusion pump by opening the backing valve. The mechanical pump will thus pump away anything that the diffusion pump has removed from the gas system. The diffusion pump heater was turned on by plugging in the cord and allowing the oil to warm up for an hour. Once the oil was hot, the cold trap above the diffusion pump was filled with liquid nitrogen and was kept filled every day thereafter. The cold trap was filled after turning the heater on in order to allow the oil to remove any impurities that have accumulated at the top of the pump. Cooling it down first would simply freeze the impurities to the wall of the trap, possibly diffusing into the clean system at a later time.

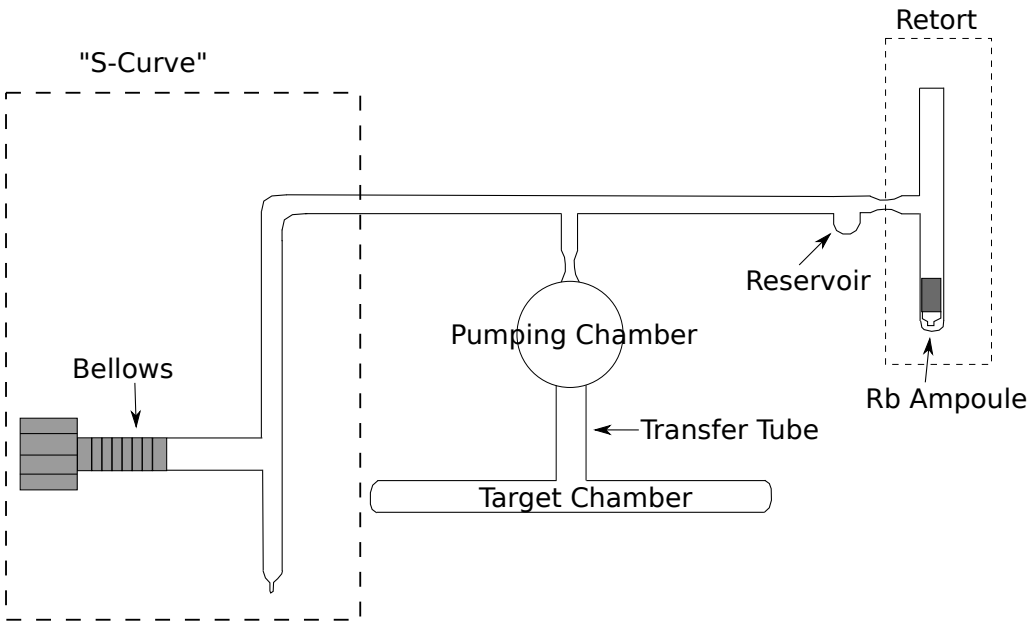

Figure 8: A diagram of a Pyrex string used for a GE180 cell. The uranium transition traditionally sits on the vertical segment just above the pumping chamber.

Concurrent to this process, the cell and accompanying string of glass were attached to the gas system via a bellows. The bellows was attached to an "S-curve" of glass with a glass blowing lip. This curve was spliced onto a flat segment that had the cell already attached from the glassblower. The final segment of the string to be attached was a T-shaped segment called the retort that had the alkali metal placed in at a later time and a reservoir into which the alkali could be distilled. Once the retort had been spliced onto the end of the string, it was roughed out with the mechanical pump and backfilled with dry nitrogen gas. To add the rubidium to the string, a pre-scored ampoule of alkali metal was cracked open while the sealed end of the retort was being opened. It was then quickly dropped into the retort,

open end downwards, and the retort opening was once again sealed up. The string was then purged by filling it with dry nitrogen and roughing it out three times.

4.5.2 Cell Pumping

Once the diffusion pump was ready to go, the string had been spliced together, and rubidium added to the string, it was time to begin the week-long process of diffusion pumping on the cell and string. To begin, every chamber of the gas system as well as the string and cell were roughed out by the mechanical pump to the 10^{-3} Torr level. The diffusion pump's gate valve was opened to the first chamber (the column), given 10 minutes to pump it down, and then the cold cathode gauge was turned on. This was done to prevent turning the cold cathode gauge on at a pressure high enough to break it. Once each segment of the gas system had dropped to the 10^{-7} Torr level, the next segment was opened until the entire gas system and string were on the diffusion pump. The diffusion pump was left to pump on the string and cell.

While the diffusion pump was pumping on the string, "flamebakes" were performed on the glass surfaces of the string and cell in order to drive impurities off of the glass. A flamebake consisted of using a blue flame from a methane-oxygen torch to lightly brush the surface of the glass starting at the point furthest away from the diffusion pump and moving closer to the diffusion pump. A single flamebake was defined as doing this process twice. On the second or third flamebake, the alkali metal was heated to the point that it melted and fell out of the ampoule that was added to the string, leaving some impurities behind such as any rubidium that had reacted with air when cracked open.

The last remaining step of the pumping stage was to "chase" the alkali metal into the

pumping chamber sphere on the cell to be filled. While still pumping on the string with the diffusion pump to increase the mean free path, a hot, blue flame was used to move the alkali metal from the retort into the reservoir (see Figure 8), which was a small nipple on the string closer to the cell. This chasing process was done by evaporating the alkali metal from one place and condensing it to another place further up stream. Once distilled into the reservoir, the T-segment of the string was removed and the alkali metal was left for at least 24 hours while the diffusion pump removed impurities left in the string. On the day of the fill, the alkali metal was distilled one final time from the reservoir to the pumping chamber.

4.5.3 Cell Fill Procedue

On the day of a cell fill, those portions of the gas system not already under vacuum were roughed out with the mechanical pump up to the nitrogen and helium gas bottle valves. Once all segments were roughed out, the sections up to the gas system valve for the helium manifold and the N_2 valve for the nitrogen manifold were filled with their respective gases to help minimize outgassing. The diffusion pump was then left to pump on all segments of the gas system, not including the string, for 4-5 hours. It was desirable to have the gas system at 10^{-8} Torr level while having the string and cell are at 10^{-9} Torr level before any gas handling began.

We utilized two main methods for cleaning the gas used to fill our cells. One method employed with a nobel gas purifier, and the second utilized a cryogenic trap, and both required some advanced preparation. For the nobel gas purifier, while the diffusion pump was pumping on the various gas chambers, it was heated up to 400°F and left to sit at that temperature for 3 hours. In order to cause the "getters" inside the purifier to be refreshed

by forced outgassing. The normal operating temperature of the nobel gas purifier for a fill was 320°F.

The homemade "cryotrap" (shown in Figure 9) took less time to prepare but was more complicated to operate. It consisted of a long segment of copper tubing that was connected to the gas system with an input and output valve. The tubing was bent in the shape of a rectangle that sat along the bottom of a box-shaped Dewar with an open top. A styrofoam lid and grease were used to thermally seal the box when it was filled with liquid nitrogen or liquid helium, depending on which gas was being cleaned. Two silicon diodes were placed on the bottom and on the wall just above the top of the tubing to measure the temperature as well as determine whether or not the tubing was submerged in the liquid cryogen. As the gas flowed through the submerged tubing, the liquid nitrogen would condense impurities out of the nitrogen gas and the liquid helium did the same for the helium-3 gas.

Figure 9: The Dewar cryotrap filled with liquid nitrogen while cleaning the gas.

With the cryotrap cold (77 K) from the liquid nitrogen, the volume of the string and cell needed to be measured in order to determine how much gas was put into each cell. This was done by first putting 300 Torr, as measured on a 100 Torr/V Baratron diaphragm pressure gauge, into a calibrated volume (CV) of 992.9 cc. The gas used for the volume measurement was also passed through the cryotrap or nobel gas purifier. The pressure in the CV was defined as the reading on the baratron with the gas in both the fill gap (FG), since this was where the Baratron was connected, plus the CV with the calibrated volume's valve closed. The gas in the FG was then roughed out by the mechanical pump and the diffusion pump was allowed to pump on the FG after it has been roughed out. The volume of the string and

cell could then be determined by knowing the pressure in the CV and using the ideal gas law while observing the pressure decreases as the CV and string valves were opened. The volume measurement process was completed before filling the cell as well as after the cell had been pulled off via glassblowing in order to calculate the amount of gas that actually ended up in the cell. The gas from the volume measurement was subsequently roughed out and the string pumped on with the diffusion pump one last time to make certain no residual gas remained in the string when the fill took place.

To fill the cell with dry nitrogen gas, the gas was throttled with two valves through the liquid nitrogen cryotrap and into the fill gap plus string and cell with a pressure of around 70 Torr for these cells. Precise values for each cell can be found in Table 1. Once at pressure, the string valve was closed and the residual nitrogen roughed out of the gas system to prepare for the helium fill. While the gas system was being roughed out, the liquid nitrogen was removed from the cryotrap Dewar and replaced with liquid helium. Once the temperature of the cryotrap had dropped to 4 K, the helium gas was throttled through the cryotrap and into the fill gap while the string valve was still closed. Once the pressure of the fill gap had risen well above the 70 Torr placed in the string already, say 200 Torr, the string valve was slowly opened and the target total pressure of 760 Torr was added to the string plus cell. The gases were left to mix overnight with the string valve closed until the next day when the cell was pulled off. The string pressure was recorded in absence of the cell, the string roughed out, and one final volume measurement was completed.

Cell Name	Fill Type	Flamebakes	Pressure (Torr)	N2 pressure	Fill Date
Coated Sphere Cell (Tyrion)	NGP	n/a	n/a	n/a	6/18/09
Spool Piece 1 (Gold Maiden)	NGP	3	611	76.10	6/18/10
Spool Piece 2 (Gold Maiden 2)	NGP	6	688	76.50	8/14/10
Spool Piece 3 (Gold Maiden 3)	NGP	3	697	75.	11/11/10
Goldfinger	NGP	8	699	75.1	4/28/13
Cupid	NGP	10	703	75.8	6/15/13
Goldeneye	NGP	8	709	75.2	10/2/13
GoldRush	NGP	9	699	75.1	11/8/13
Pyrah	NGP	13	698	75.1	2/1/14
GoldenVec	NGP	9	696	75.5	10/18/14
TitanVec	NGP	9	699	77	12/15/14
GoldenVec2	Cryogenic	11	700	75.8	2/4/15
Titan	NGP	9	699	75	3/11/15
GoldenVec180	Cryogenic	13	704	76	6/17/15
GoldenVec360	Cryogenic	15	701	75	7/11/15
Tweety	Cryogenic	12	700.	75	9/22/15
Sylvester	Cryogenic	15	700.	76.3	11/20/15
Kappa1	Cryogenic	11	701	75.2	2/6/16
Goldfinger180	Cryogenic	12	710.	75.1	5/19/16

Table 1: A table of fill parameters for each cell in this study.

5 Relaxation Measurement Results

As indicated earlier, over 19 distinct samples were studied in this work. These cells collectively represent a development of techniques for future use in target design as well as medical studies using gases that are polarized through spin-exchange optical pumping. The geometry used evolved over time to fulfill the next step to understanding these complex cells. Each different stage of cell production represented our discovery of new crucial considerations ranging from the used materials and magnetic field gradients to gas cleaning and glassblowing techniques. Table 2 compares the physical characteristics of each cell used in these studies in the chronological order with which they were produced. Figure 10 is included below to provide an example for a typical spin up and spin down curve. The associated time constant of a spin down curve equates to a cell's lifetime, or the inverse of total relaxation experienced by the helium nuclei.

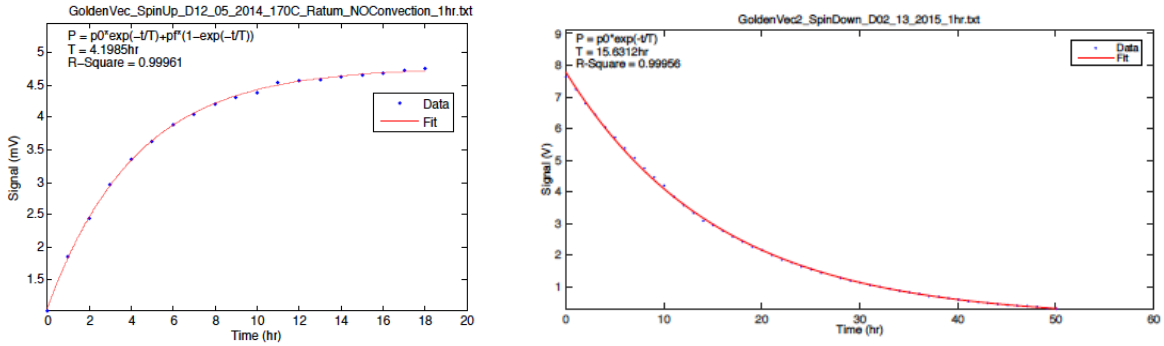

Figure 10: An example of a typical Spin Up and Spin Down.

Cell Name	Geometry	Glass Type	Tube Type	Coating
Coated Sphere Cell (Tyrion)	Sphere w/ Valve	GE180	None	Gold
Spool Piece 1 (Gold Maiden)	Vaccum Flanges	Pyrex	Spool Piece	Gold
Spool Piece 2 (Gold Maiden 2)	Vaccum Flanges	Pyrex	Spool Piece	Gold
Spool Piece 3 (Gold Maiden 3)	Vaccum Flanges	Pyrex	Spool Piece	Gold
Goldfinger	Vertical	Pyrex	OFHC Copper	Gold
Cupid	Vertical	Pyrex	OFHC Copper	None
Goldeneye	Vertical Valve	Pyrex	OFHC Copper	Gold
GoldRush	Vertical	Pyrex	OFHC Copper	Gold
Pyrah	Vertical	Pyrex	None	None
GoldenVec	Horizontal	Pyrex	OFHC Copper	Gold
TitanVec	Horizontal	Pyrex	Titanium	Gold
GoldenVec2	Horizontal	Pyrex	OFHC Copper	Gold
Titan	Vertical	Pyrex	Titanium	None
GoldenVec180	Horizontal	GE180	OFHC Copper	Gold
GoldenVec360	Horizontal	GE180	OFHC Copper	Gold
Tweety	Vertical	Pyrex	Canary Glass	None
Sylvester	Horizontal	GE180	Canary Glass	None
Kappa1	Sphere	GE180	None	None
Goldfinger180	Vertical	GE180	OFHC Copper	Gold

Table 2: A display of cell design for each cell used in these studies. Each cell is presented in chronological order with which they were produced and tested.

5.1 Gold Coated Sphere

Historically, our first attempt at a cell that contained both glass and metal was named Tyrion. Tyrion was a spherical GE180 cell that had the interior surface coated with gold. A lifetime of 1.21 h was measured for the cell. This result has been attributed to a significant amount of impurities in the cell. Other coating techniques were investigated, but they all were particularly difficult. It was thus decided to investigate gold coated pieces of metal that could be incorporated into a cell's design. These tests occurred prior to my arrival at the University of Virginia and have been included as a complete account of our groups work with cells incorporating metal.

5.2 Gold Coated Spool Pieces

Our first studies of spin-relaxation on metal surfaces involved a custom OFHC spool piece whose interior surface was plated with gold using the aforementioned procedure. One end of the spool piece was capped off and the other end was attached, using a custom fitting, to a glass flange. This glass flange then had a segment of tubing, a transfer tube, that lead to a spherical pumping chamber. The seals in the assembly were elastomer o-rings. The entire assembly itself was known as "Gold Maiden" and was used for all three series of measurements. This series of measurements began production in 2010 (also prior to my arrival) and are referred to universally as "the spool piece."

Before displaying rubidium discoloration, Gold Maiden 1 (the first series of measurements) was measured to have a lifetime of up to 2.14 h after AFP losses had been accounted for. This discoloration thus led us to believe the cell had either leaked or experienced signif-

icant outgassing from the o-ring. The pumping chamber of this cell was separated from the metal using glassblowing and in the absence of the tube and flanges, a lifetime of 109.2 h was obtained from the GE180 pumping chamber alone post AFP loss corrections. The second series of measurements was never able to be attempted due to yet another leak. Using pulse NMR techniques, Gold Maiden 3 was recorded to have a 6.49 hour lifetime despite having o-rings between the flanges. Small modifications were made to the assembly between each series of measurements. These tests gave hope that a cell incorporating gold coated copper could have acceptable relaxation rates should the cell design not have these o-rings.

5.3 Gold Coated OFHC Copper Seal Assembly Cells

To get away from the flange and o-ring design used for Gold Maiden, it was desired to bond the glass directly to the metal. Glass-to-metal seal assemblies as described in the previous chapter were prepared and the first wave of cells were filled in 2013. Five different Pyrex cells of this vertical configuration were produced: Cupid, Goldfinger, Goldrush, Goldeneye, and Pyrah. Figure 11 below shows the drawing used to create these cells. Cupid was a bare, OFHC copper tube cell. Goldfinger and Goldrush were intended as identical copies of one another, with a vertical gold coated tube. However, Goldfinger was found to have a leak at both seal points during its fill process and was sealed with Celvaseal Leak Sealant vacuum glue. Goldeneye was designed with a valve to separate the rubidium vapor in the pumping chamber from the gold coated seal segment. Pyrah was an all Pyrex control cell. By design, cells with this geometry were estimated to have a volume of 354.6 cm^3 and a surface area of 400.9 cm^2.

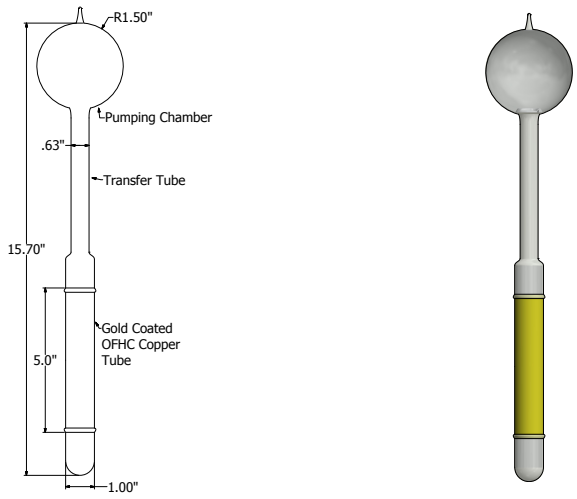

Figure 11: Drawing of a cell with vertical geometry.

As seen in Table 3 below, Goldfinger and Cupid showed relatively similar max lifetimes of 3.59 h and 3.13 h respectively. Subsequent measurements displayed different behaviors between the two cells. Both cells unfortunately showed a decrease in observed lifetimes to 2.36 h and 0.27 h respectively. Goldfinger degraded, but not as drastically or quickly as Cupid's degradation as seen in Figure 12. This degradation was also attributed to a reaction occurring between the copper or gold with the rubidium vapor that is present while the cell was being polarized at 170°C for every pump up performed. It is known that rubidium forms an amalgam with the metal but we are uncertain on the specific details of this interaction. It is also important to acknowledge that the leak discovered during Goldfinger's fill process may have also contributed to this long-term behavior.

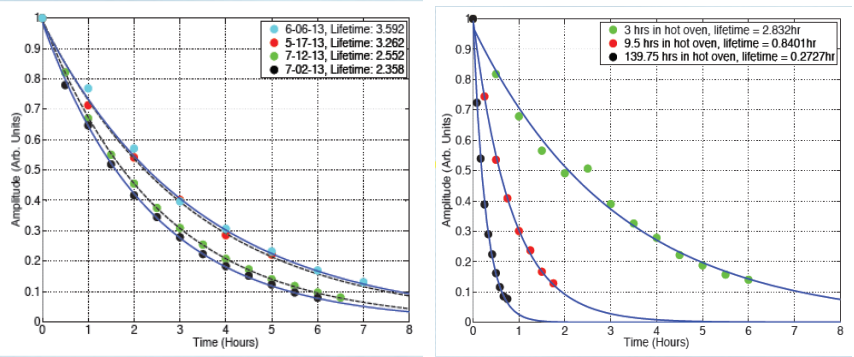

Figure 12: Comparing the evolution of Goldfinger's lifetime (left) and Cupid's lifetime (right).

Our experience with Goldfinger's and Cupid's lifetime degradation caused concern that this rubidium interaction may hinder future cell production and so there was a desire to isolate the rubidium vapor from the metal tube. A cell named Goldeneye was designed to do this by incorporating a valve into the transfer tube (between the pumping chamber and metal tube) as seen in Figure 13. While performing a spin down to measure the lifetime, the cell was kept at room temperature and so Goldeneye was designed with a valve that could be closed to keep the rubidium vapor away from the metal tube. Extreme care was taken to only polarize the cell with the valve closed, then when at room temperature, the valve was opened and gas allowed to diffuse before the spin down data was collected. It was found that with the valve closed, Goldeneye displayed a lifetimes of 13.94 h and while open the cell lifetime was 4.09 h. A significant degradation of the lifetime from spin down to spin down was not observed. This large disparity in lifetime suggested that an enormous amount of relaxation was being introduced to the cell in the bottom segment of the cell.

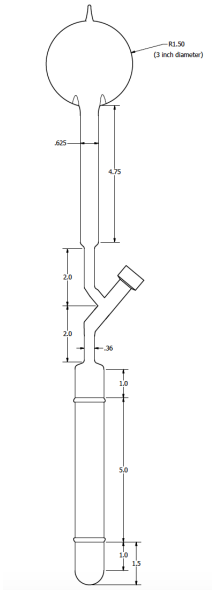

Figure 13: A drawing of GoldenEye, the valved cell.

By adding a valve to the cell design, the overall cell length of Goldeneye was quite long compared to the size of our holding field's sweet spot. Its length therefore guaranteed that the bottom of the cell was now so far away from that sweet spot that it experienced quite large inhomogeneity. While the measured lifetime of 13.94 h was long compared to Cupid and Goldfinger, the inhomogeneity still was providing additional relaxation. A calculation of the magnetic field gradient is included in Figure 14.

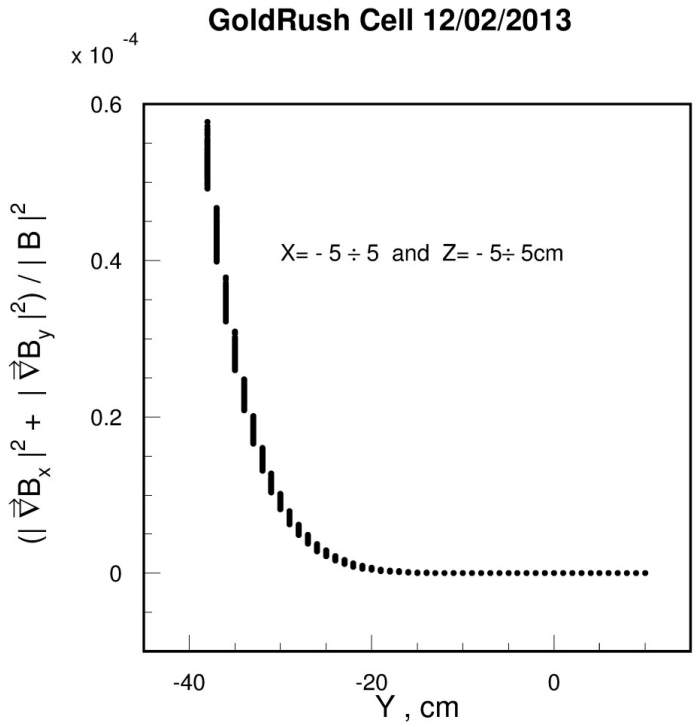

Figure 14: Magnetic field graident map along the vertical direction at our cell's location.

Goldrush, Goldfinger's sister cell, was used to test this observation about the cell's overall length being too long. Once polarized, the cell was raised by approximately 10 cm since the original mounting position was optimized to focus the laser light on the pumping chamber. By doing this, Goldrush's lifetime was increased from 12.1 h to 14.81 h, at that time a group record for cells incorporating metal. Even the unelevated lifetime of 12.1 h was drastically longer than Goldfinger's lifetime. Though not entirely clear, this difference can be attributed to the leak. Some of the rubidium may have reacted during the leak, producing impurities in the cell.

A Pyrex control cell with identical dimensions to Goldfinger and Cupid, Goldrush, was produced. This cell was named Pyrah. Upon testing, Pyrah and had a lifetime of 19.71 h. Having an identical geometry, this cell also could be elevated by 10cm once polarized. Upon

elevating it, the lifetime increased to 26.52 h, the longest observed lifetime from a Pyrex cell in this study.

5.4 Horizontal Style Cells

The impact of field inhomogeneity on cell lifetime motivated the next change in design. The metal tube was turned horizontally, centered, and aligned symmetric on the central axis of the pumping chamber, which shortened the overall vertical length of the cell. These horizontal tube cells now had two transfer tubes, each running from the bottom of the pumping chamber to either side of the glass-to-metal seal. This change in geometry reduced the overall cell length (top of pumping chamber to lowest point of cell) from 15.75" for Goldrush to 10" for a horizontal style cell. By design, these cells have an estimated volume of 312.9 cm^3 and a surface area of 540.4 cm^2.

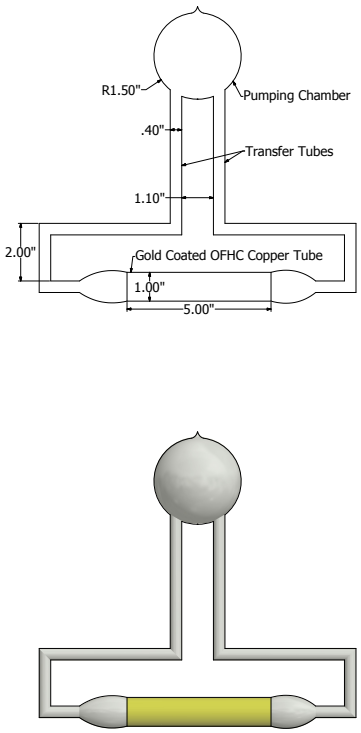

Figure 15: Drawing of a cell with horizontal geometry.

GoldenVec was the first cell to have this horizontal geometry and was found to have a lifetime of 10.6 hrs, on the order of Goldrush prior to elevation but somewhat lower than hoped for. At that time, a separate work with a prototype target cell (ProtoVec2) found that by cleaning impurities from the gas with a cryotrap rather than a nobel gas purifier, a greater lifetime could be obtained. A sister cell for GoldenVec, GoldenVec2, was created with this cryogenic cleaning method and was found to have an even longer lifetime of 15.6 hrs. GoldenVec2 holds the record in this study for longest lifetime from a cell incorporating any metal component.

5.5 GE180 Cells

It is experimentally known that aluminosilicate glass displays less relaxation than borosilicate glasses from prior experience [2]. GE180 glass, a type of aluminosilicate glass, is alkali resistant as well as less porous to the helium. In order to create a target cell with metal windows, it was necessary then to switch from Pyrex, a borosilicate glass, to GE180, an aluminosilicate glass. GoldenVec180 and GoldenVec360, both with a horizontal tube geometry to match the horizontal Pyrex cells, were made with GE180 pumping chamber, transfer tubes, and a tiny portion of Corning 7052 to seal the GE180 to the OFHC copper tube.

Both GoldenVec180 and GoldenVec360 were made with an identical design to GoldenVec2 and were also filled with a cryotrap to clean the fill gases. The cells displayed lifetimes of 4.43 h and 3.01 h respectively. This result was certainly not anticipated since GE180 is commonly preferred in spin-exchange optical pumping of helium experiments. One interpretation of the poor result from both GoldenVec180 and GoldenVec 360 was that the Corning 7052, which had never been tested in our group, was causing the significant relaxation seen in these GE180-based cells.

This belief that the transition glass was inhibitive of a better lifetime led us to investigate an alternative transition glass. Uranium glass, also known as canary glass, is commonly used in our group as a transition between Pyrex and GE180 in our glass manifolds. Its ability to fuse with both glasses made it an exceptional candidate to be used in a comparison between a cell made of Pyrex to one of GE180.

To build cells to test the feasibility of using the uranium glass, a 5" by 1" segment of resized uranium glass was used to replace that identical length of copper used in prior cells

such as Goldfinger and GoldenVec. Two such cells were produced, one with a Pyrex pumping chamber and the vertical geometry of Figure 11 and a second with a GE180 pumping chamber and the horizontal geometry of Figure 15.

Tweety was the vertical cell with a Pyrex pumping chamber and transfer tubes. To great surprise, a lifetime of 22.7 h was measured. Tweety's lifetime was on the order of the Pyrex control cell's lifetime of 26.52 h. This surprised us because preconceptions among those working with polarized gases expect a cell with uranium glass to display significant relaxation because of uranium's paramagnetic nature [19] [20]. This result in itself was valuable since it shows that uranium glass does not cause drastic nuclear spin relaxation and could be used for cell production in future work.

The second uranium glass cell, named Sylvester, was a horizontal cell made with a GE180 pumping chamber and transfer tubes. This result proved to be more interesting than Tweety. Even without metal or transition glass, Sylvester had a mere lifetime of 6.39 h. This value sits higher than GoldenVec180 but significantly lower than the other two entirely glass cells (22.7 h for Tweety, 26.52 h for Pyrah). Great care was taken so that the glassblower would blow and anneal the glass for this cell identically to the way he did for a cell incorporating metal. This cell thus had no difference from GoldenVec and GoldenVec 2 other than uranium glass where the copper tube and the transition glass had been. This shorter lifetime from Sylvester suggests that the problem thus lies with the GE180 glass itself.

The cause of significant relaxation in GE180 cells was either that the cells used a melt of glass that had a lot of impurities in it or significant micro-fissures existed in the glass due to inadequately annealing the glass. A spherical GE180 cell was made from the same melt of glass used for every other cell in these studies. That cell, Kappa1, was a 1 atm cell with a

hybrid alkali mixture of potassium and rubidium. Using adiabatic fast passage rather than pNMR, which was made possible due to the absence of a metal tube, Kappa1 ruled out an intrinsic problem with the glass melt itself when it was found to have a lifetime of 72 h.

The difference between the GE180 used in Kappa1 and GoldenVec360 was the way it was prepared. Traditionally, a glass cell was baked in an oven at Princeton once all glass components have been spliced together in order to help relax (anneal) the glass as described above in section 4.4: Glassblowing. In order for the seals on the glass-to-metal seals to remain hermetic, they must not be introduced to temperatures over 200°C. Therefore, all segments of the cell except the glass portion directly attached to the copper tube were placed in the oven. The glass-to-metal seal assembly was then spliced onto the cell once it had cooled. Micro-fissures that are normally eliminated by this baking process remained in the glass used for the GE180 cells in these studies. Pyrex is normally much thinner, anneals at a lower temperature, and has a more accessible specific heat for working with and so the effects of skipping this baking process were less evident in data from the Pyrex cells than data from the GE180 cells.

One final cell, Goldfinger180 was produced after Kappa1 eliminated the possibility that our melt of GE180 was bad. This cell was a GE180 cell with a vertical geometry. For this cell, the glassblower was instructed to minimize the amount of glass that did not get baked in his oven after being assembled. Less of the glass that was attached to either side of the glass-to-metal seal assembly seen in Figure 5 was used in the cell. After assembling the string and connecting it to the vacuum system, it was discovered that the string segment was unable to be pumped down to the 10^{-3} Torr level. The Houskeeper seal closest to the pumping chamber was not holding vacuum up to the standard of our other cells. To remedy

this, two coats of an epoxy type sealant called Stycast 1266 (different from the type used for Goldfinger) were applied to the seal. After curing, the string was then able to be pumped down to pressures normally experienced for our gas system. Upon testing, the cell was measured to have a 10.4 h lifetime, a lab record for GE180 based cells that incorporate metal. The cell was elevated in the same fashion as Goldrush and Pyrah (the Pyrex analog of Goldfinger180 and the control cell) and was found to have a 12.4 h lifetime in the elevated position. Goldfinger180's results thus confirmed that both the segments of GE180 that were not annealed acceptably and magnetic field gradients introduced additional relaxation.

5.6 Titanium Glass-to-Metal Seal Cells

Significant time and energy have been invested in gold plated copper due to its good relaxation properties and ease to machine. However, a metal with a higher tensile strength and lower atomic number than gold's 29 would be preferred. Titanium, atomic number 22, is one alternative that fits both the aforementioned criteria.

Two Pyrex based cells were created to measure relaxation on titanium. One cell was a bare titanium glass-to-metal seal assembly in a vertical configuration and the other was a gold coated titanium seal in a horizontal configuration. Titan, the bare titanium cell, had such a short lifetime that no spin down was ever able to be obtained. TitanVec, the horizontal configuration cell, was observed to have a lifetime of 0.52 hrs. During the ultrasonic cleaning process, gold flakes peeled off of TitanVec's tube. This poor lifetime was attributed to the helium being exposed to the bare titanium or electroplated undercoat instead of the favorable gold surface.

Cell Name	Max Lifetime (h)
Coated Sphere Cell (Tyrion)	1.21
Spool Piece 1 (Gold Maiden)	2.14
Spool Piece 2 (Gold Maiden 2)	Cell leaked
Spool Piece 3 (Gold Maiden 3)	6.49
Goldfinger	3.59
Cupid	3.13
Goldeneye-Closed	13.94
Goldeneye-Open	4.09
GoldRush	14.81*
Pyrah	26.52*
GoldenVec	10.6
TitanVec	0.52
GoldenVec2	15.6
Titan	Very short
GoldenVec180	4.43
GoldenVec360	3.01
Tweety	22.7
Sylvester	6.39
Kappa1	72.17
Goldfinger180	12.4*

Table 3: A table of max measured lifetimes for each cell. All numbers listed with an astericks were recorded while elevated. Note: Kappa1 was a hybrid cell and only a sphere.

6 Convection Study Results

In electron scattering experiments, depolarization of the helium gas occurs as the electron beam passes through the target. For experiments that require the target be polarized, the depolarized gas must be replaced with polarized gas from the pumping chamber. Historically, target cells have had a single transfer tube connecting the pumping chamber to a horizontal, cylindrical target chamber (where the electron beam passes through). In this scenario, the polarized gas replenishing rate is determined by the rate at which the gas can diffuse through the transfer tube. If a target was to be made with two transfer tubes, a geometry similar to the horizontal cells used here, then making one transfer tube hotter than the other would cause the gas to circulate. The temperature difference between the two tubes could be increased so that the target chamber's depolarized gas is replaced faster than diffusion would allow.

Since the cells in these experiments with a horizontal geometry have two transfer tubes, the cell creates a loop around which the gas can travel. Thus, convection of the gas can be driven to increase the circulation of gas within the cell. To do this, two Kapton heater tapes were wrapped around a horizontal segment of the transfer tube that had a pNMR coil wrapped on it as seen in Figure 16. A variac was used to power the heater tapes while a RTD was inserted under the tape heater to monitor the heated transfer tube's temperature. The temperature gradient between the two transfer tubes determined convection speed and so the heater tape was wrapped in insulation and Kapton tape to minimize heating the other transfer tube. The other transfer tube, which did not have a heater wrapped on it, had a cold air jet focused on it for cooling.

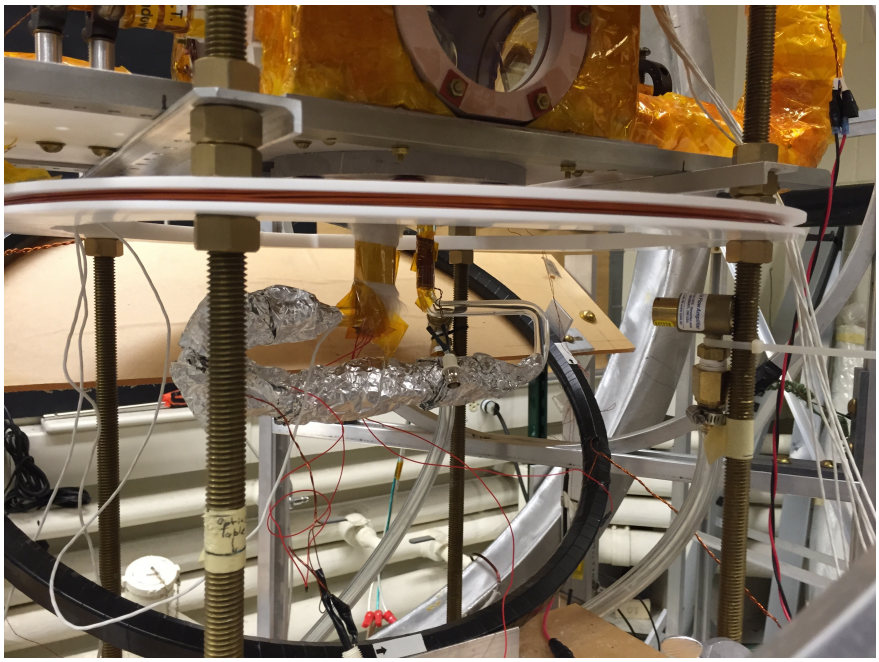

Figure 16: Photo of the convection test aparatus.

In order to measure the convection speed, a portion of gas was depolarized using a pNMR coil and then data were collected every 20 seconds in two other coils wrapped at different points on the transfer tubes. When the portion of depolarized gas approximately the size of the "zapper coil" passed through the two pickup coils, a decrease in the polarization was observed. As this portion of gas continued beyond the pickup coil, the polarization rose again to create a dip in a plot of polarization versus time.

Knowing the path length from the zapper coil to the pickup coil combined with the time it took the slug of gas to arrive, the speed it was traveling was produced. The dip in polarization was so small that the pickup coil on the far side of the OFHC copper tube was unable to resolve the drop. The near side pickup coil was able to produce Plot 17. With the variac at 13 V, the RTD reported T= 60°C, corresponding to a convection speed of 30 cm/min. This speed was 6 times greater than the 5 cm/s that is required to replenish the

beam depolarization during an electron scattering experiment. It was found that even by turning off the heater tape and leaving the insulation on the tube, the cool air vent was able to make a temperature gradient large enough for convection to be driven.

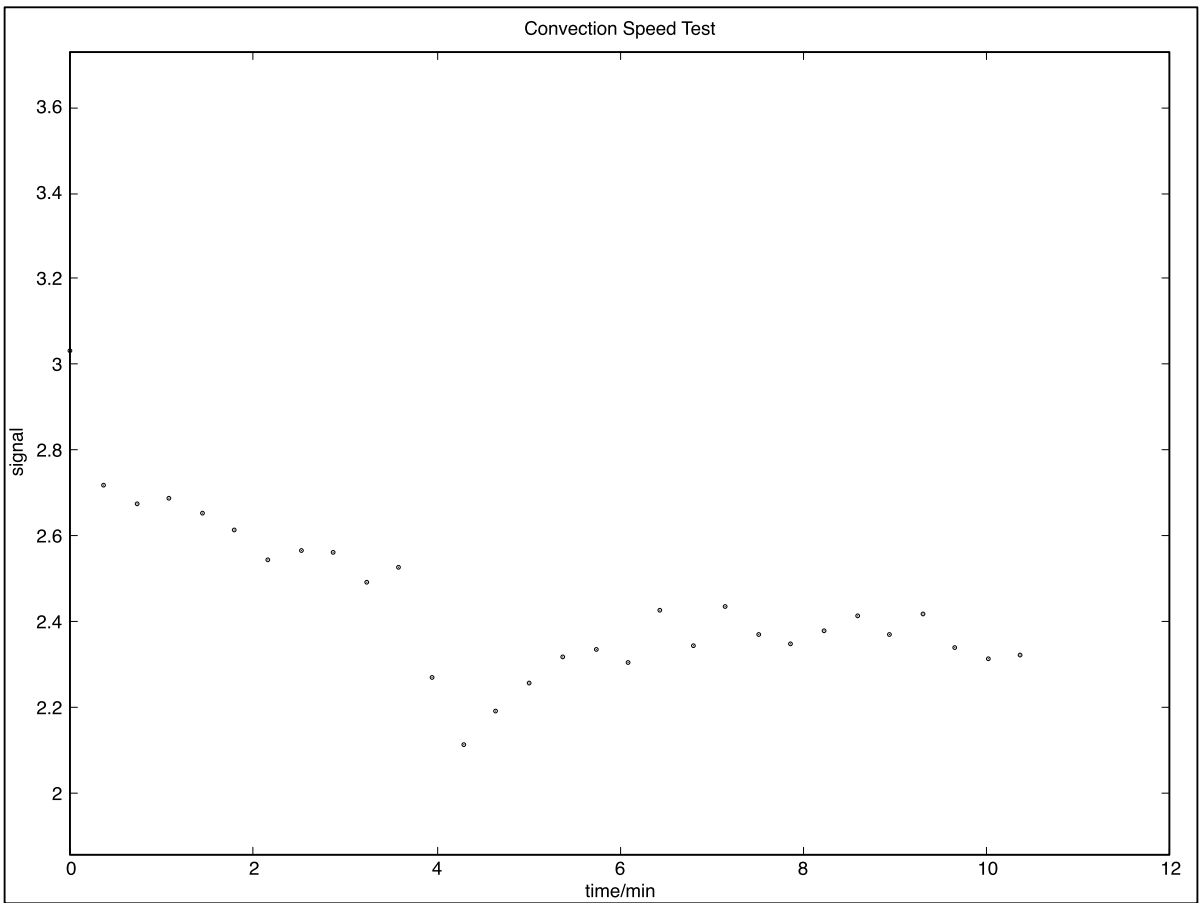

Figure 17: Plot of polarization versus time for the pickup coil that was on the nearest to the zapper coil. Data was collected every 20 seconds and the zap occured just before the dip in polarization.

This convection speed enabled a study of the long term evolution of a cell's lifetime to be performed. Since the convection was running 6 times faster than the expected experimental conditions of an electron scattering experiment, the helium, nitrogen, and rubidium vapor were able to interact with the glass-to-metal seal assembly more frequently than they're

expected to during an experiment. Both GoldenVec and GoldenVec2 were left with the oven hot and convection running at 30 cm/min for 310 h and 262 h respectively. They also spent 341 h and 251 h in a hot oven while polarizing the cell. Even after all that time for the gold to interact with the gas, GoldenVec's lifetime dropped from 10.6 h to 6.06 h while GoldenVec2's lifetime only dropped from 15.6 h to 13.4 h. While being noticeable drops, if these cells were only driven at the 5 cm/min rate they would have to be hot for 78 and 65 total days respectively to match the same amount of interaction our cells were exposed to. These periods of times would be sufficiently long enough for a typical electron scattering experiment to be ran at JLab.

7 Conclusion

7.1 Summary

The nuclear spin relaxation of 19 cells with various geometries and material composition have been characterized in this work. The longest lifetime obtained from a cell containing a gold coated copper tube was 15.6 h from GoldenVec2, which was Pyrex and had a horizontal metal tube. When using the even smaller amount of metal required for an end window, the relaxation introduced by the metal should be sufficiently small. Even after long periods of exposure to rubidium vapor, the lifetime of the cells remained sufficiently long.

These studies found that when switching from borosilicate to aluminosilicate glass, the cell lifetimes decreased. This additional relaxation was predicted to be a result of how the aluminosilicate, GE 180, was prepared. Care must then be taken in the production of a cell to minimize the amount of glass that is not baked after being blown together. It is still desirable to use aluminosilicate glass because of its positive experiences in past experiments as well as its alkali metal resistance and lower porosity to helium.

Finally, two cells incorporating titanium, both bare and gold coated, displayed enormous levels of relaxation. The relaxation was so large that spin downs to measure lifetimes were unobtainable or had to be done with the oven still hot.

7.2 Future Directions

The cells incorporating gold-coated OFHC copper tubes in this study are an important milestone towards building a metal end window for target cells at Jefferson Lab. The appropriate thickness for minimizing unwanted scattering events while maintaining enough tensile

strength to survive an electron scattering experiment must still be determined. Initial calculations have found that 2 mils or approximately 50 μm should be sufficiently thin.

One current design for the window itself is a gold-plated hemisphere that will be attached to a glass-to-metal seal not unlike those tested here. What exactly the material that will be coated with gold is still being decided but aluminum and copper are two potential candidates. A major challenge is to have a window that is only 2 mils thick while maintaining structural integrity. At present, we are most optimistic about an electroformed window. This technique is particularly interesting because Epner Technologies Inc. (the same company that does the gold plating for us) are able to electroform the window.

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