Creation of Precise Rb and K Mixtures for ^{3}He Polarization Studies

A thesis submitted in partial fulfillment of the requirement
for the degree of Bachelor of Science in Physics from
The College of William and Mary

by

Patrick Steele

Accepted for _____

_____

Todd Averett, Advisor

_____

Irina Novikova

_____

Charles Perdrisat

_____

Robert Pike

Williamsburg, VA
April 15, 2011

Contents

Abstract

Physicists are interested in studying the spin structure of neutrons via scattering experiments on neutron targets. We create highly polarized ^{3}He cells for use in these experiments, as neutrons have a short half-life and so are unsuitable for direct study. The quality of polarization of these targets is affected by the alkali mixtures used to polarize them. We construct apparatus to create precise mixtures of Rb and K to fill target cells in order to explore the polarizing properties of different mass ratios of Rb and K. We use electron paramagnetic resonance experiments to determine the polarization of cells filled with these mixtures.

Chapter 1

Introduction

Physicists are interested in studying the spin structure of neutrons. This can be accomplished via scattering experiments using neutron targets. Using only neutrons as targets proves difficult; neutrons have a half-life of approximately ten minutes, making any neutron target highly unstable. However, the structure of ^{3}He makes it a suitable replacement for the neutron in targets; as the spin of the two protons in ^{3}He conspire to cancel each other, the net spin of ^{3}He is due only to the neutron [2]. To increase the usefulness of ^{3}He targets, the spin of the ^{3}He nuclei can be polarized. These polarized neutrons are studied at Thomas Jefferson National Accelerator Facility.

We desire to create mixtures of rubidium and potassium for use in polarized ^{3}He nuclei targets, such as with Babcock et al. [1]. These mixtures require precise mass ratios of rubidium and potassium, and both alkali metals must be handled with extreme care. Both metals are highly reactive with both oxygen and water; exposure to either renders the metals useless for the purposes of ^{3}He targets. To prevent the alkali mixtures from being contaminated by oxidation reactions, they are manipulated in an inert gas such as nitrogen.

To avoid contaminating the materials, it is necessary to achieve atmospheric pu-

rity levels on the order of 1 part per million contaminants. We utilize a Vacuum-Atmospheres glovebox as a the housing for our inert atmosphere; we construct a air purification system to rid the atmosphere contained within the glovebox from most contaminants.

1.1 Target Cell

The ^{3}He target cells consist of a spherical glass bulb containing ^{3}He gas and a polarizing mixture, as in [3]. The bulb is attached to a tube through which an electron beam is fired; see Figure 1.1. These cells are hand blown for the laboratory and are made of aluminosilicate glass [2]. The pumping chamber allows the ^{3}He to come into contact with the alkali mixture away from the long tube where scattering experiments occur.

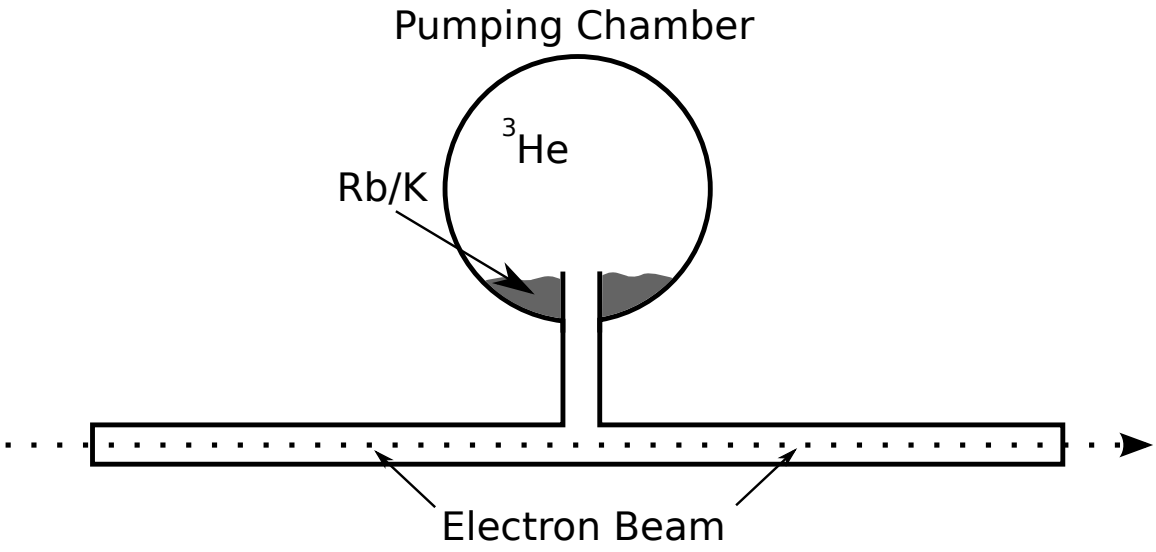


Figure 1.1: A target cell filled with rubidium and potassium. Note the small cusp around the tube entering the pumping chamber designed to prevent the rubidium from leaving the chamber.

1.1.1 Optical Pumping

A technique known as optical pumping is used to polarize the ^{3}He within the target cells. The goal of optical pumping is to first polarize gaseous Rb and K in the target cell, and then have the ^{3}He become polarized via contact-induced spin-exchange with the rubidium.

Recall the structure of the target in Figure 1.1. The circular pumping chamber is placed in an oven, heating the chamber to 230°C and encouraging rubidium vapor to evolve. Additionally, the target is located at the center of large Helmholtz coils which create a powerful magnetic field, causing the degenerate $5S_{1/2}$ ground state to split into two energy states, $m = \pm 1/2$. Zeeman splitting also occurs in the $5P_{1/2}$ state, the first excited state. A circularly polarized laser is the applied to the pumping chamber, causing Rb atoms in the $5S_{1/2}, m = -1/2$ energy state to be excited to the $5P_{1/2}, m = +1/2$ state. These excited atoms then either decay to the $5S_{1/2}, m = -1/2$ state where they are re-excited, or decay to the $5S_{1/2}, m = +1/2$ state. The N_2 gas in the cell absorb rogue emissions, preventing randomly polarized photons from interfering with the experiment. The $5S_{1/2}, m = +1/2$ state becomes the predominant energy state, and exchanges its polarization by colliding with ^{3}He and exchanging spin. The K is polarized through collisions with the Rb as well, which in turn pass their polarization to the ^{3}He via additional collisions [4]. The K is included because K-K collisions are less likely to lose their polarization than Rb-Rb collisions [9].

1.2 Measuring the Polarization of a Target Cell

1.2.1 Nuclear Magnetic Resonance

The polarization of ^{3}He in a target cell can be measured with nuclear magnetic resonance (NMR) using the adiabatic fast passage (AFP) technique as in [10]. However, this technique requires calibration using a water target to measure the absolute polarization of the ^{3}He target, which can be difficult.

1.2.2 Electron Paramagnetic Resonance

A different technique, electron paramagnetic resonance (EPR), can also be used to measure the polarization of the ^{3}He cell. Previous work in the laboratory has utilized this technique to successfully categorize the polarization of ^{3}He targets. EPR measures the polarization of the ^{3}He in the target indirectly by measuring the polarization of the rubidium also present in the cell [2].

This technique relies on the application of powerful magnetic fields to the cell in order to exploit the Zeeman splitting that occurs between the energy levels of the rubidium atoms. The Rb atoms are continuously optically pumped as in 1.1.1 to keep most Rb atoms in the $5S_{1/2}, m = +1/2$ state. These atoms transition between energy levels split by the hyperfine interaction at a frequency known as the electron paramagnetic resonance frequency. This frequency is dependent upon the magnetic field applied to the rubidium atoms, which is determined by the Helmholtz coils, the ^{3}He, and the spin-exchange interaction between ^{3}He and Rb. As the first two fields are fixed, the EPR frequency yields a measure of the spin-exchange interaction, which depends directly upon the polarization of the ^{3}He [2].

1.3 Contaminants

We fill the target cells with alkali metals. However, alkali metals react with both oxygen and water. These impurities degrade the quality of experiments, and so we manipulate the metals in a nitrogen-based environment. As such, we require purifying agents that do not react with nitrogen. Other contaminants, such as CO_2, are of little importance and acceptable levels should be obtained from the initial flush of the glove box with nitrogen.

1.3.1 Removing H_2O

A high quality desiccant is required to achieve the desired level of moisture in the glove box, which is approximately 1 part per million. To remove water molecules from the atmosphere, we utilize a bed of type 4A molecular sieve, which is capable of adsorbing molecules approximately the size of water, or 4Å in diameter. Type 4A molecular sieve also has the advantage that it can be regenerated through the application of heat and a special mixture of regeneration gases alone, allowing the purifying system to be left intact for regeneration.

1.3.2 Removing Oxygen

A high quality oxygen getter is required to achieve the desired level of oxygen in the atmosphere, which is approximately 1 part per million. There are a variety of oxygen getters available on the market, but we have several requirements that not all adsorbers possess. Specifically, we require adsorbers that can be regenerated simultaneously with the molecular sieve material used for water removal; this allows a single housing to be used for the purification system, rather than a series of purifying columns. This also requires that the adsorber's regenerative process requires no materials or gases that are hazardous to the molecular sieve material. For these reasons

we have opted to use the "Q5" material sold by Vacuum-Atmospheres Corporation, which was the oxygen scavenger originally used in the purification system with the glovebox. The "Q5" material is a proprietary oxygen getter than satisfies the requirements above. The primary active component of Q5 is copper, which tends to oxidize when exposed to oxygen. This allows the Q5 to be regenerated by the application of heat and a mixture of hydrogen and an inert gas, as described in Section 3.2 [8].

Chapter 2

Apparatus

2.1 Glove Box

The glove box is a Vacuum Atmospheres Company (VAC) Dri-Lab HE series glove box. VAC produced a purifying filtration unit designed to accompany the glove box, the Dri-Train HE-493. This glove box does not have a Dri-Train HE-493 purifying unit; as VAC no longer manufactures the Dri-Train or Dri-Lab, replacement units are generally unavailable for purchase.

The Dri-Lab (henceforth glove box) is a large aluminum, rectangular prism with a Plexiglas covering on the front. The Plexiglas has two circular ports allowing gloves to be used to manipulate the contents of the glove box when the box is sealed. To allow materials to be passed in and out of the glove box without contaminating the atmosphere, a sealed antechamber is located on the right of the box. This antechamber has doors to the interior and exterior of the box that can be sealed independently, and the chamber itself can be evacuated and then flushed with high purity nitrogen to remove contaminants from the antechamber atmosphere prior to opening the interior door. See Figure 2.1 for a schematic diagram of the front of the box.

The rear of the glove box contains a number of useful ports for plumbing, as well

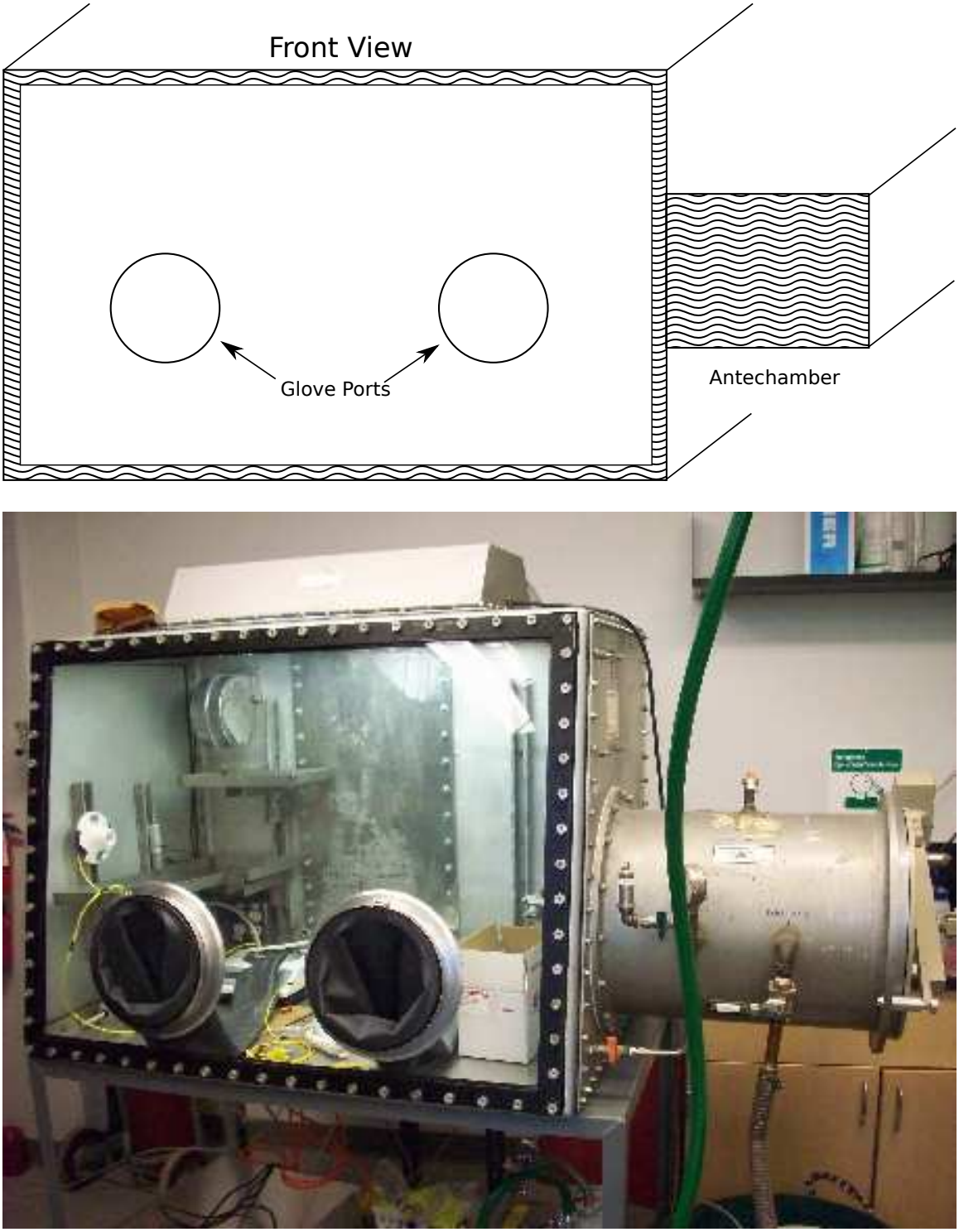


Figure 2.1: Top: a diagram of the front of the glove box. Bottom: a photograph of the front of the glove box.

as electrical conduits; see Figure 2.2. These ports will be used for gas in- and outflow, pressure control, and purging processes.

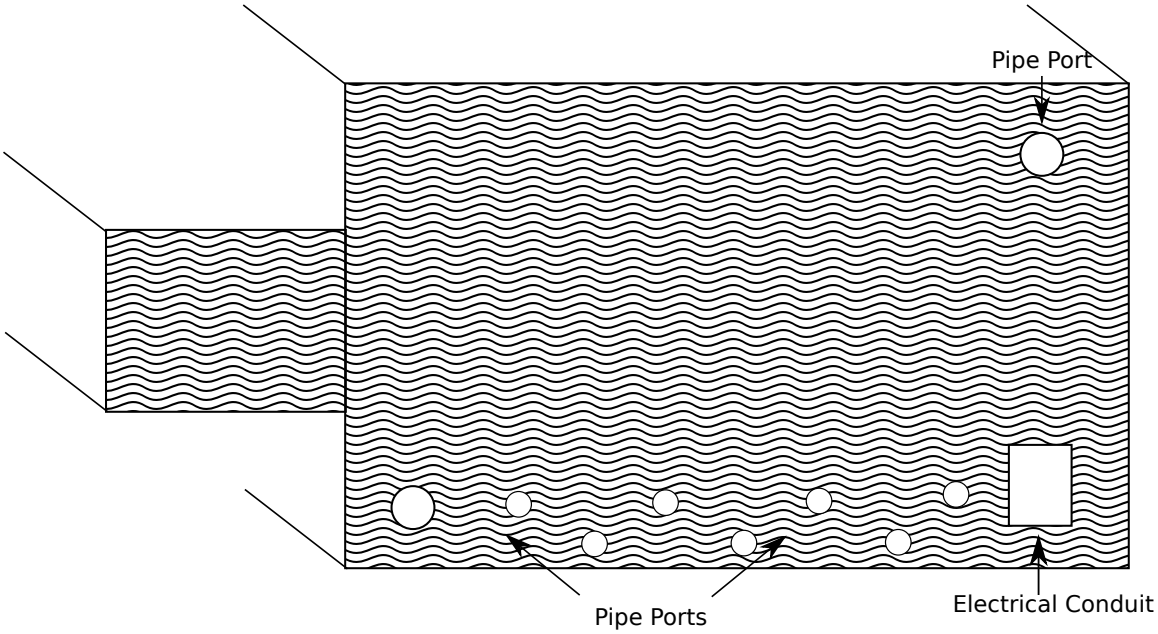


Figure 2.2: A diagram of the rear of the glove box.

2.2 Filter

The glove box will serve as a container for the inert atmosphere; however, a filtration system must be used in conjunction with the glove box to establish the initial inert atmosphere as well as to continually purify the atmosphere, as imperfect seals and porous surfaces let contaminants in over time. Additionally, the stock nitrogen available is not sufficiently pure, and so must be further purified.

2.2.1 Requirements

The primary requirement of any filter is to reliably purify a gas stream to the desired purity; no other qualities are important if the filter is fundamentally incapable of sustaining an inert atmosphere. Beyond this, it is desirable for the filter to be able to

be regenerated; it is neither time- nor cost-effective to continually replace the active components of the filter when they become saturated with contaminants.

To accomplish these goals, the new filter design was based on the original Dri-Lab design specifications. The original Dri-Train had a purifying column 6-1/4 inches in diameter and 16 inches in length; this column contained 5-3/4 pounds of molecular sieve and 5 pounds of Q5 oxygen getter. Additionally, the plumbing connecting the Dri-Train to the Dri-Lab glove box was 1-inch piping. These dimensions allow the Dri-Train to filter a sufficient volume of gas to maintain the atmospheric purity required, and so serve as a useful lower bound on the dimensions required for the new design.

The requirement that the purifying column be able to be regenerated poses an additional requirement on the housing of the adsorbents. The regeneration of the Q5 material and 4A molecular sieve requires the materials to be heated to 200°C [8]. To accomplish this a heating unit must be inserted inside the purifying column. Since the column is a sealed environment, it must have electrical feedthroughs for power and thermocouple leads.

2.2.2 Construction

To meet these requirements, a tubular steel casing was constructed to house the 4A molecular sieve and Q5 material to form a purifying column to pass a gas stream through. This tube is 6-inches in diameter and 18-inches in length. To seal the column while still allowing it to be opened to manipulate or replace its contents, Conflat flanges were welded to the pipe on both ends. This allows any compatible Conflat flanges to then be attached, providing interfaces for plumbing and electrical feedthroughs; see Figure 2.3.

The top seal on the column is a Conflat flange with seven additional Conflat ports through it; it has six 1-1/3 inch ports and one 2-3/4 inch port. The larger port is used for the gas input from the glove box, and two of the smaller ports are used

for electrical feedthroughs; the remaining four ports are sealed, but can be used in the future for additional heating or temperature sensing wiring. See Figure 2.4 for a schematic of the multi-port flange. The bottom seal on the column is a Conflat flange similar to the top one, but with only one 2-3/4 inch port which serves as the output of the purified gas.

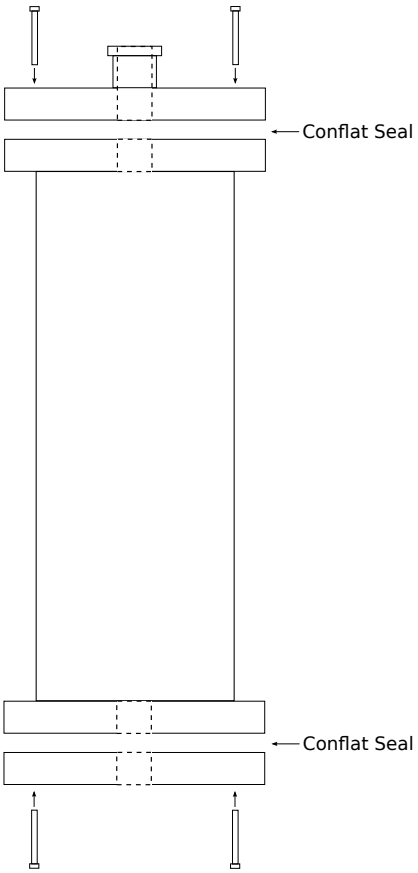


Figure 2.3: Schematic of the purifying column. Conflat flanges were welded to the pipe, allowing other Conflat flanges to complete the seal and to provide interfaces for both plumbing and electrical feedthroughs.

2.3 Plumbing

The glove box and filter require several different gas inputs and outputs to regulate the pressure of the box, feed nitrogen in, and to pass gas to and from the filter.

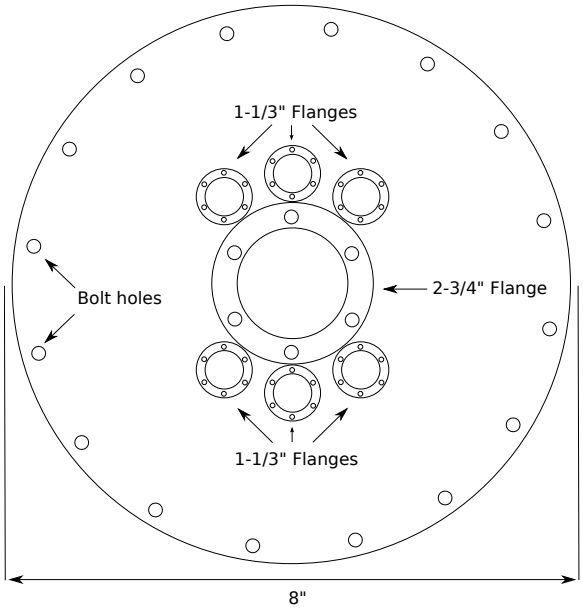
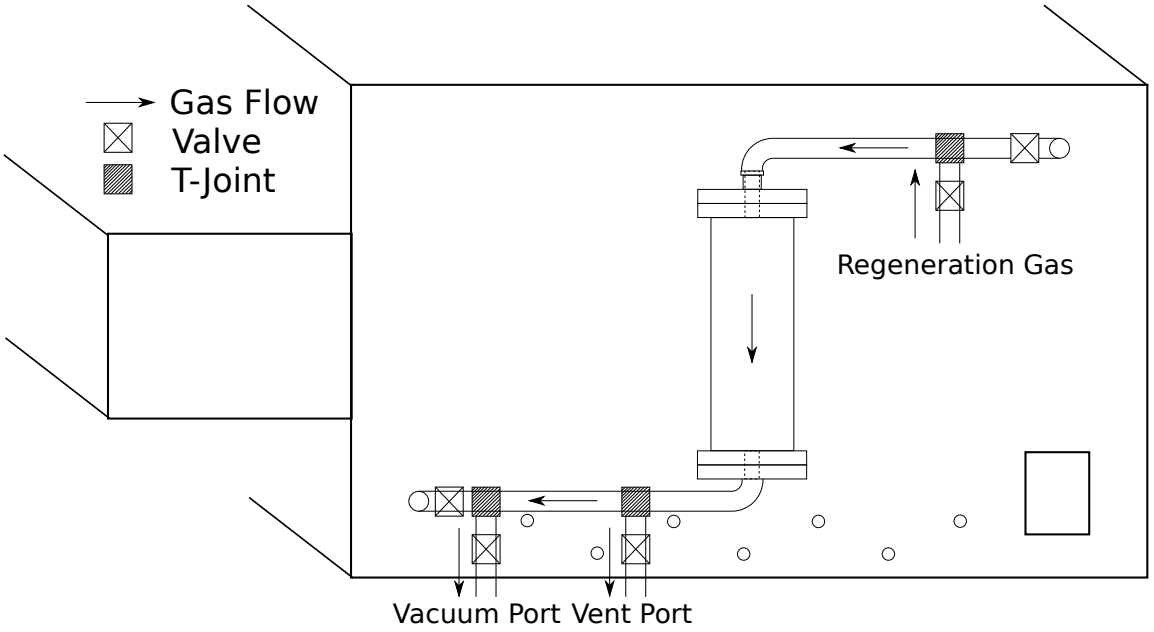


Figure 2.4: Schematic of the multi-port Conflat flange used to seal the top of the purifying column. Two of the smaller flanges are used for electrical feedthroughs, and the larger flange is connected to the glove box plumbing for gas input.

Additionally, the glove box and filter must be able to be completely separated when regenerating the filter, as the regeneration process requires both creating a vacuum in the filter and introducing a gas mixture to the filter as discussed in Section 3.2.



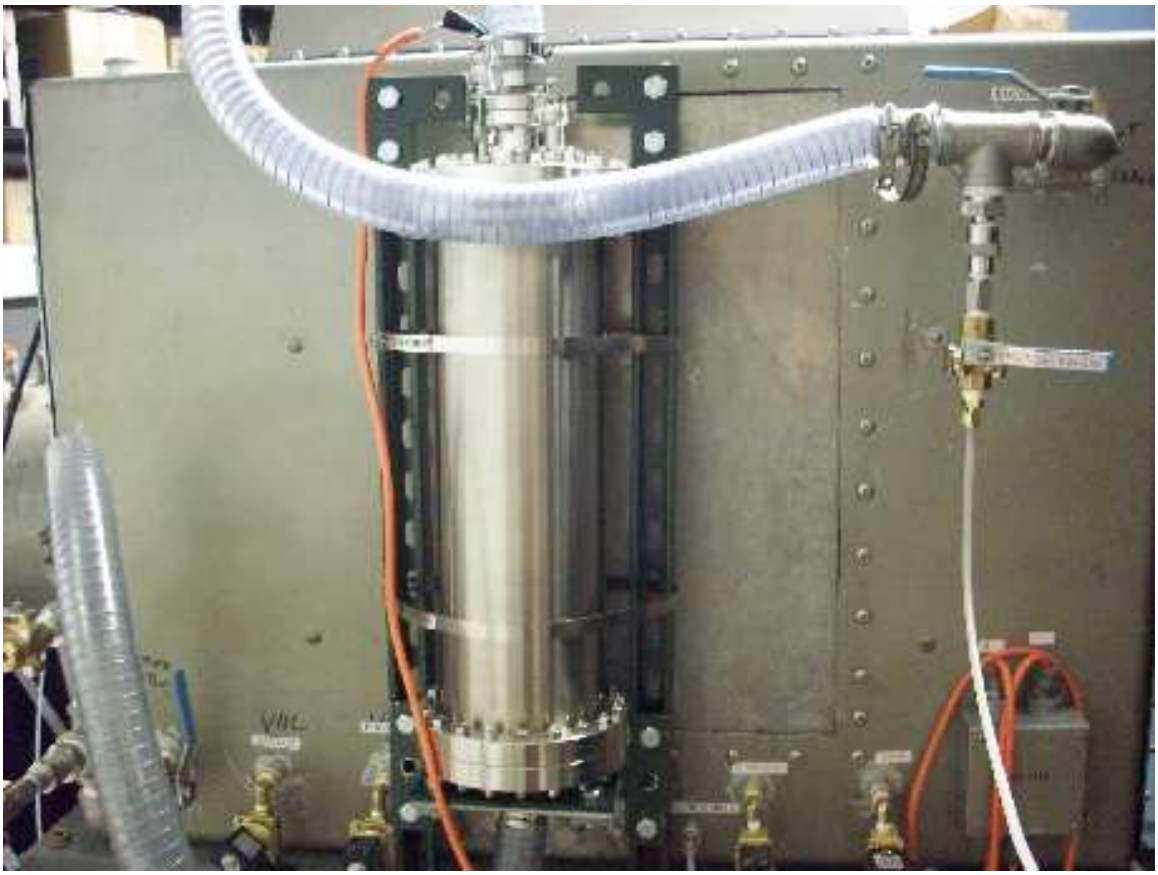


Figure 2.5: Top: a schematic of the plumbing on the rear of the glove box. Bottom: a photograph of the rear of the glove box with the purifying column mounted.

Chapter 3

Establishing an Inert Atmosphere

3.1 Leak Testing

The first step in establishing an inert atmosphere is to check for leaks in the glove box. Gross leaks can be detected by pressurizing the system and applying a leak detecting fluid – generally a dilute solution of water and soap – such as Swagelok Snoop® to all joints and seals. If a substantial leak is present the solution will foam as gas is forced out of the glove box by the positive pressure. This test is suitable only for locating gross leaks, and the failure to detect leaks by this method should not be viewed as an indication that no leaks exist. Once all gross leaks have been detected, one of the following more rigorous methods should be used.

As second method is utilized to detect aggregate leaks over the box. The box is filled to positive pressure and left to sit. An attached pressure sensor will document the change in pressure over some unit of time. Small decreases in pressure are expected, as the rubber gloves used in the glove box provide a porous surface through which gas will escape. The manufacture suggests that pressure decreases up to $^1/_8$ inches of water pressure over one hour are acceptable [7]. Both this and the previous tests are repeated with the antechamber door open and closed to test both the seals

between the antechamber and the main chamber as well as the antechamber and the exterior of the glove box.

A more precise leak detection method utilizes a helium detector. By filling the glove box to positive pressure with helium gas, any leaks will emit helium and so could be detected by the detector. This test was not used, as it is both time consuming and expensive, and the previous test yielded less than a 1/8 inches of water decrease in pressure over one hour.

3.2 Regeneration

When the adsorbents in the filtration system become saturated, they must be regenerated. This must also occur when establishing the initial inert atmosphere, since the adsorbents were exposed to an impure environment for an extended period of time, and so are completely saturated. There are two goals of regeneration: remove the oxygen from the Q5 oxygen getter, and remove the water from the 4A molecular sieve.

The filter is first isolated from the glove box environment by closing the gas in- and outflow pipes; this is accomplished by closing valves V1 and V5 in figure 3.1. These two valves will remain closed until regeneration is complete. The vacuum (V2), vent (V3), and regeneration (V4) valves are also closed. The purifying column is then heated to 200°C by activating the heating unit located inside the column. The temperature is regulated by varying the current through the heating unit, and is tracked by monitoring the two thermocouple leads embedded in the adsorbents. This temperature is maintained for three hours. The vent (V3) and regeneration (V4) valves are then opened, flushing the column with regeneration gas. All contaminants boiled off during the heating phase are flushed out, and the hydrogen in the regeneration gas regenerates the Q5 material. This gas is flushed through the column for one hour,

after which the vent (V3) and regeneration (V4) valves are closed, and the system is vacuumed for eight of more hours by opening the vacuum (V2) valve and turning on the attached vacuum pump. After vacuuming, the vacuum (V2) valve is closed, and the gas in- and outflow (V1, V5) valves are opened once again.

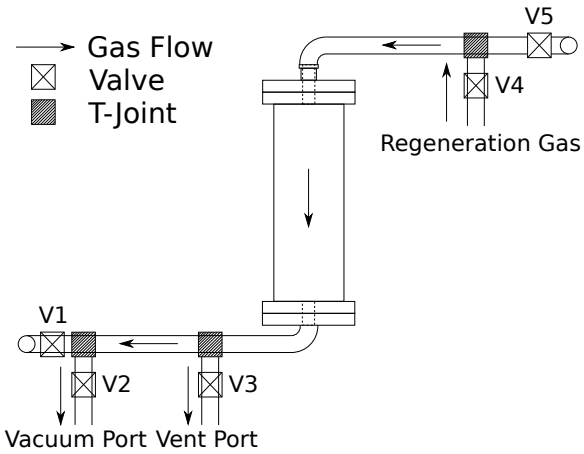


Figure 3.1: Reference schematic for the regeneration process. Each valve used during regeneration is labeled here.

3.3 Purification

3.3.1 Purge

Before utilizing the purifying filter to remove contaminants from the glove box, the box is continuously purged with high purity nitrogen. This is done primarily to avoid immediately saturating the filter with the large quantities of oxygen and water present in the laboratory atmosphere. However, this approach is also quite effective. Assume that all stock gas entering the system is perfectly mixed with the existing gas, and assume that the stock gas has purity α. Let the volume of the box be V, and assume that we wish to displace all gas currently in the box. If the flow of stock gas into the glove box is f, then the rate of gas leaving the box is also f. Then the fraction of

impure gas C in the box obeys

$$\frac{dC}{dt} = -\frac{f}{V}C, \tag{3.1}$$

and so

$$C(t) = e^{-ft/V}. \tag{3.2}$$

For $C(t)$ to drop to purity levels of 1ppm, we need $C(t) = .00001$, or

$$t = -\ln(.00001)\, V/f = -11.51V/f. \tag{3.3}$$

However, more interesting is the total amount of stock gas that must be used to attain this level of purity. Define V_{gas} as the amount of stock gas used; then

$$V_{\text{gas}} = ft, \tag{3.4}$$

and so

$$C\left(V_{\text{gas}}\right) = e^{-V_{\text{gas}}/V}. \tag{3.5}$$

Thus, to achieve purity levels of 1ppm, it is necessary to purge the box with

$$V_{\text{gas}} = -V \ln\left(10^{-6}\right) \tag{3.6}$$

of stock gas, or 13.8 box volumes of gas.

3.3.2 Purifying Cycle

Once the glove box has been flushed and the level of contaminants has been reduced to the 1000ppm range, the box is allowed to cycle gas through the purifying column to further reduce the impurity level. Assume that the gas in the glove box is perfectly mixed, so contaminants are evenly distributed throughout the box. As before, let the volume of the box be V, the flow through the filter be f, and the fraction of impure

gas in the box by C. If the purifying column can remove $0 < \alpha \leq 1$ of contaminants passed through it, then C obeys

$$\frac{dC}{dt} = -\alpha \frac{f}{V} C. \tag{3.7}$$

Then

$$C(t) = \beta e^{-\alpha f t/V}, \tag{3.8}$$

or defining V_{gas} as the volume of gas circulated through the purifying column,

$$C(V_{\text{gas}}) = \beta e^{-\alpha V_{\text{gas}}/V}. \tag{3.9}$$

When $V_{\text{gas}} = 0$, we have that $C = \beta$, and so β is the initial concentration of impurities in the glove box. To achieve purity levels of 1ppm when starting at a purity level of β, it is necessary to filter

$$V_{\text{gas}} = -\frac{V}{\alpha} \ln\left(\frac{10^{-6}}{\beta}\right), \tag{3.10}$$

of gas, or

$$-\frac{1}{\alpha} \ln\left(\frac{10^{-6}}{\beta}\right) \tag{3.11}$$

box volumes. The original Dri-Train had an impurity half-life of approximately 4.5 minutes, and so would require approximately 30 minutes to reduce impurity levels from 1000ppm to 1ppm [8]. As the filter was constructed to meet the specifications of the Dri-Train, similar performance is expected.

3.4 Purity Testing

After the initial purge and continuous purification, it is useful to measure the oxygen and moisture content of the glove box. There are several tests possible of varying levels of accuracy. The simplest test is the "lightbulb" test, which measures both

oxygen and moisture content. An exposed lightbulb filament burns with a length of time proportional to both the moisture and oxygen content of the air surrounding it. If an exposed 25-watt lightbulb filament burns out in less than five hours, then the oxygen and moisture content of the glove box is greater than 5ppm; if the bulb burns longer, then the oxygen and moisture content is less than 5ppm [6, 7].

Should impurity levels fall below 5ppm, it is likely that purity levels are acceptable. If, however, levels are above 5ppm, it is useful to know which substance is too abundant. To measure the moisture levels in the glove box, the titanium tetrachloride test can be used. If when exposed to the glove box environment a sample of titanium tetrachloride emits white smoke, then moisture is present at a level of 10ppm or higher [7]. To measure oxygen levels in the glove box, the diethylzinc test can be used. If when exposed to the glove box environment a mixture of diethylzinc and heptane emit white smoke, then there is oxygen in excess of 5ppm in the glove box [8].

Chapter 4

Mixing Procedure

The following procedures for mixing the alkali metals are adapted from [5].

1. Establish an inert atmosphere in the glove box, as per Chapter 3.
2. All of the following supplies are required inside the glove box. This list is representative of the supplies required, not exhaustive; a more complete list is given in [5]. Any missing supplies can be passed into the glove box via the antechamber.
    - Alkali metals
    - Ampules for mixing with stoppers
    - Torch with gas supply
    - Hot plate
    - Scale
    - Ampule heaters, possibly with variac transformers to control the heat
3. Melt the K contained in an ampule. Pour the melted K into a mixing ampule.
4. Melt the Rb and pour onto a dish, allowing it to form small chunks while cooling. Weight the proper amount of Rb chunks, and slowly add the chunks

to the mixing ampule filled with K.

5. After the Rb and K have been thoroughly mixed in the mixing ampule, seal the ampule in order to remove it from the glove box.

6. Bathing the ampule in Ar gas, replace the stopper on the Rb/K mixture ampule with a stopper connected to a gas hose. This hose will allow the gas to leave the ampule during heating, keeping high pressure from rupturing the ampule.

7. Using a torch, seal the ampule by cutting through the top of the ampule while pulling and twisting the ampule down. This will cause the bottom of the ampule to twist off, leaving a glass seal in place of the stopper.

8. Heat the ampule to melt the Rb/K mixture. Mix for 10 - 15 seconds.

Chapter 5

Conclusion

The glove box has been constructed and sealed. Initial tests indicate that the glove box is well sealed; as discussed in Section 3.1, no gross leaks are detected by Snoop®, and the glove box exhibits acceptable pressure losses. Automatic pressure controls are installed, which will be used to maintain a positive gauge pressure in the glove box. The purifying column has been installed, and the glove box is able to circulate its atmosphere through it.

The next step is to regenerate the purifying column using the methods discussed in Section 3.2. This process has begun. As the column is being regenerated, the glove box will be purged using high-purity nitrogen gas, evolved from a vat of liquid nitrogen, as in Section 3.3.2. Following the regeneration and purge cycles, the glove box will be left to cycle its atmosphere through the purifying column to further improve the purity of the atmosphere. Finally, the purity of the glove box will be verified using the "lightbulb" technique discussed in Section 3.4 using specially prepared filaments. A lightbulb socket has been installed for this purpose on the interior of the glove box, opposite the purified stream inlet.

Should the atmosphere be purified appropriately, we will proceed to mix Rb and K ampules for use in ^{3}He targets using the procedure outlined in Chapter 4 and

detailed in [5]. These ampules will be used to fill existing target cells. These cells will be polarized via the optical pumping techniques discussed in Section 1.1.1, which will then have their polarization tested by EPR experiments as in Section 1.2.2.

Bibliography

[1] E. Babcock, I. Nelson, S. Kadlecek, B. Driehuys, LW Anderson, FW Hersman, and T.G. Walker. Hybrid Spin-Exchange Optical Pumping of ^{3}He. *Physical review letters*, 91(12):123003–1, 2003.

[2] E. M. Buckley. Measuring the polarization of a ^{3}he target cell using electron paramagnetic resonance. Thesis, College of William and Mary, 2005.

[3] RE Jacob, SW Morgan, and B. Saam. ^{3}He spin exchange cells for magnetic resonance imaging. *Journal of Applied Physics*, 92:1588, 2002.

[4] L. M. Larkin. Investigation of Noble Gas Polarization Using Optically Pumped Rb and K. Thesis, College of William and Mary, 2004.

[5] T. Ross. Rubidium and Potassium Mixing Procedure. Technical report, University of Virginia, 2011.

[6] D.F. Shriver and M.A. Drezdzon. *The manipulation of air-sensitive compounds*. Wiley-Interscience, 1986.

[7] Vacuum Atmospheres Company. *Dri-Lab Technical Manual: GBX, HE & DLX Series*.

[8] Vacuum Atmospheres Company. *Dri-Train Technical Manual: Model HE-493*.

[9] T.G. Walker and W. Happer. Spin-exchange optical pumping of noble-gas nuclei. *Reviews of Modern Physics*, 69(2):629–642, 1997.

[10] X. Zheng. Precision Measurement of Neutron Spin Asymmetry A1n at Large x_{bj} Using CEBAF at 5.7 GeV. Technical report, Thomas Jefferson National Accelerator Facility, Newport News, VA (US), 2002.

Appendix A

Regeneration

The following are data taken while regeneration the purifying column.

This table shows temperature data taken during the heating phase of the regeneration. The "heater thermocouple" refers to the thermocouple embedded in the heating element. The "other thermocouple" refers to a secondary thermocouple embedded in the purifying column some distance from the heating element. Note the lag between the temperature at the heater, located roughly at the center of the column, and the other thermocouple, located closer to the wall of the column.

Time	Variac (%)	Heater Thermocouple (°C)	Other Thermocouple (°C)
2:27pm	18	208	102
2:30pm	14	207	109
2:33pm	14	207	114
2:36pm	14	209	119
2:39pm	14	212	124
2:43pm	13	212	128
2:46pm	13	212	130
2:49pm	13	213	132
2:55pm	13	215	135

3:00pm	12	216	138
3:08pm	12	210	139
3:13pm	14	216	141
3:16pm	13	219	142
3:23pm	12	215	144
3:35pm	12	214	145
3:51pm	12	215	147
4:20pm	12	220	152

Table A.1: Temperature data recorded during the heating phase of the column regeneration.

Appendix B

Photographs

The following are photographs of the glove box.

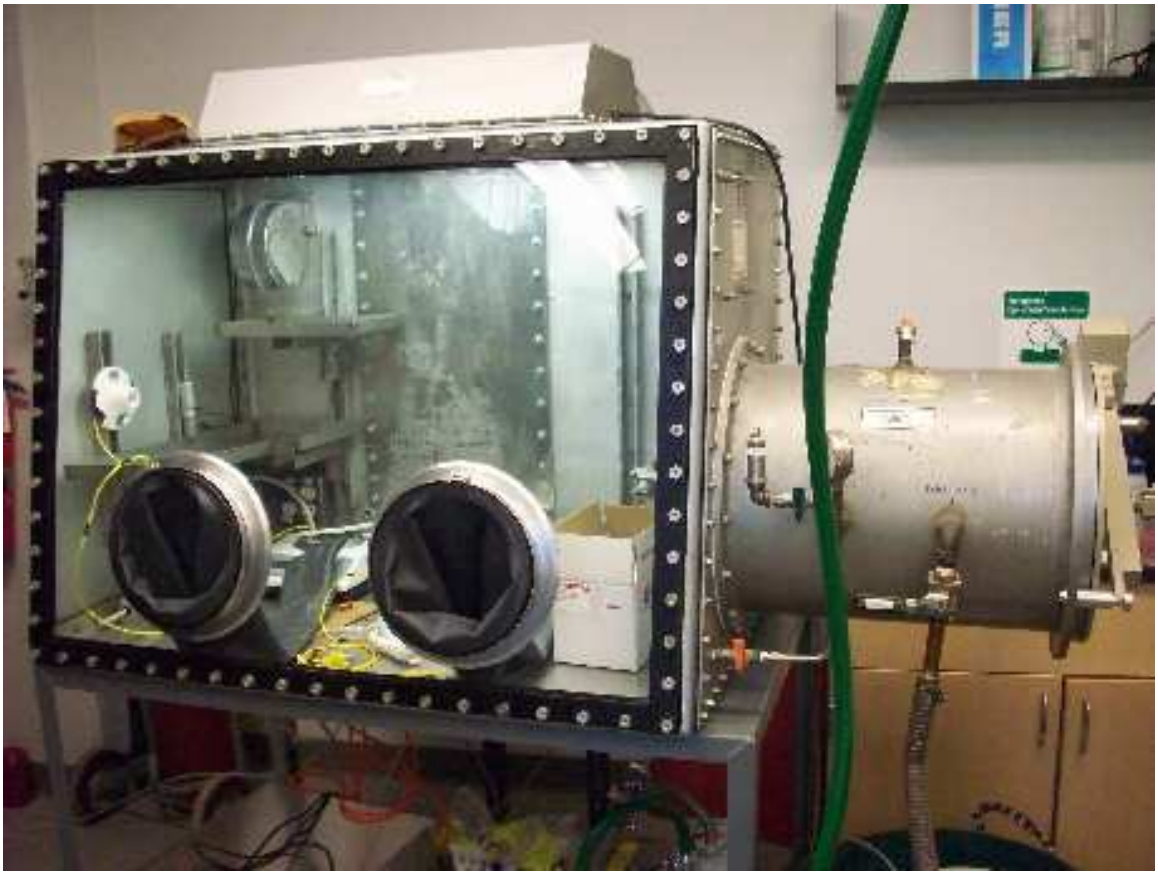


Figure B.1: A photograph of the front of the glove box.

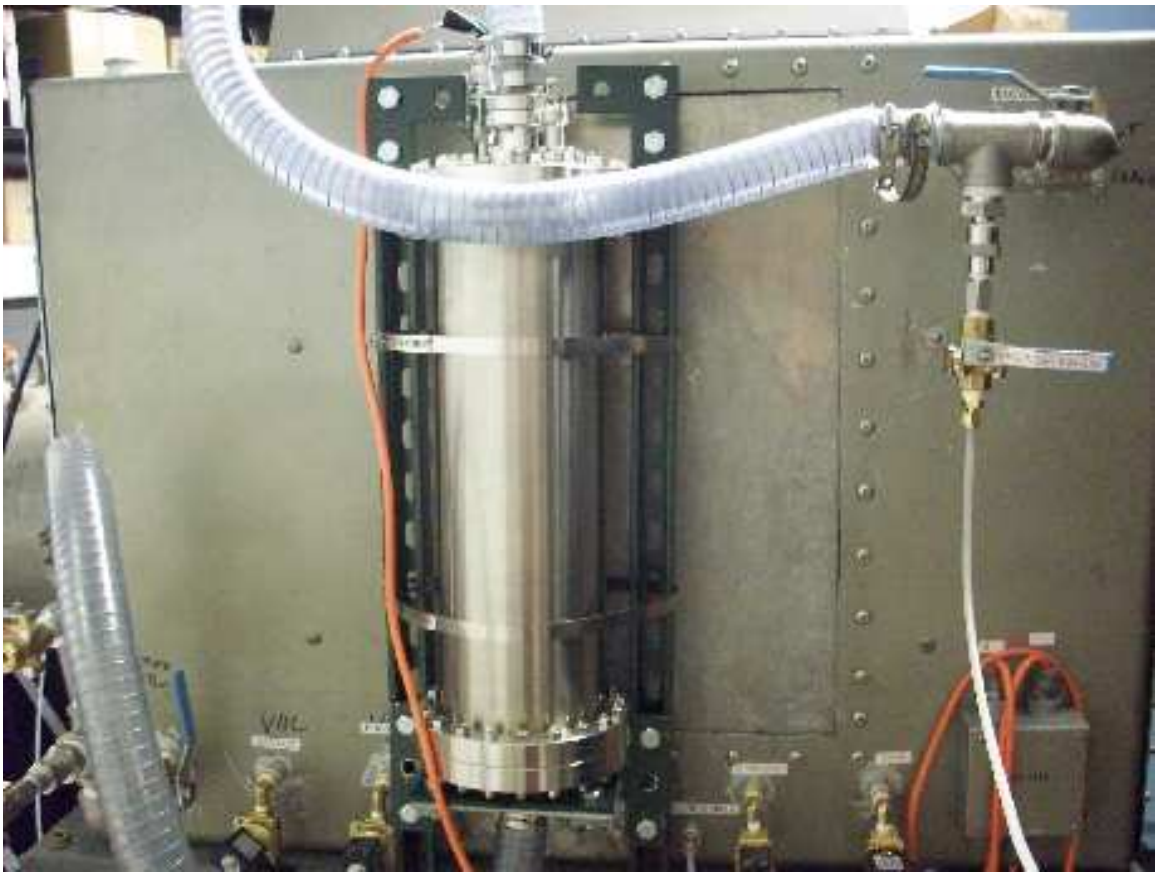


Figure B.2: A photograph of the rear of the glove box with the purifying column mounted.

Figure B.3: A photograph of the interior of the glove box. Note the ring stands for holding ampules, as well as the hot plate and scale.

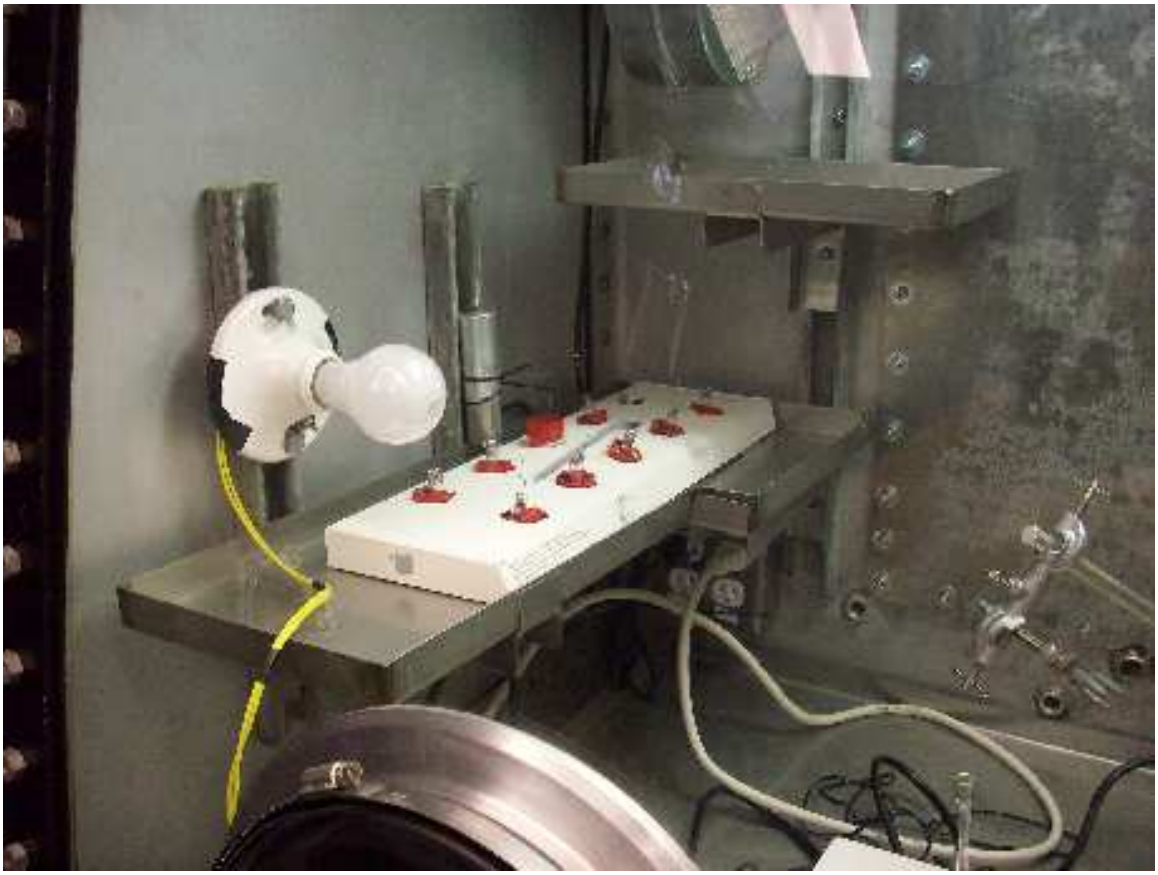


Figure B.4: A photograph of the filaments used to test the purity of the atmosphere. Note the working light bulb in the socket that the filaments will be placed in.

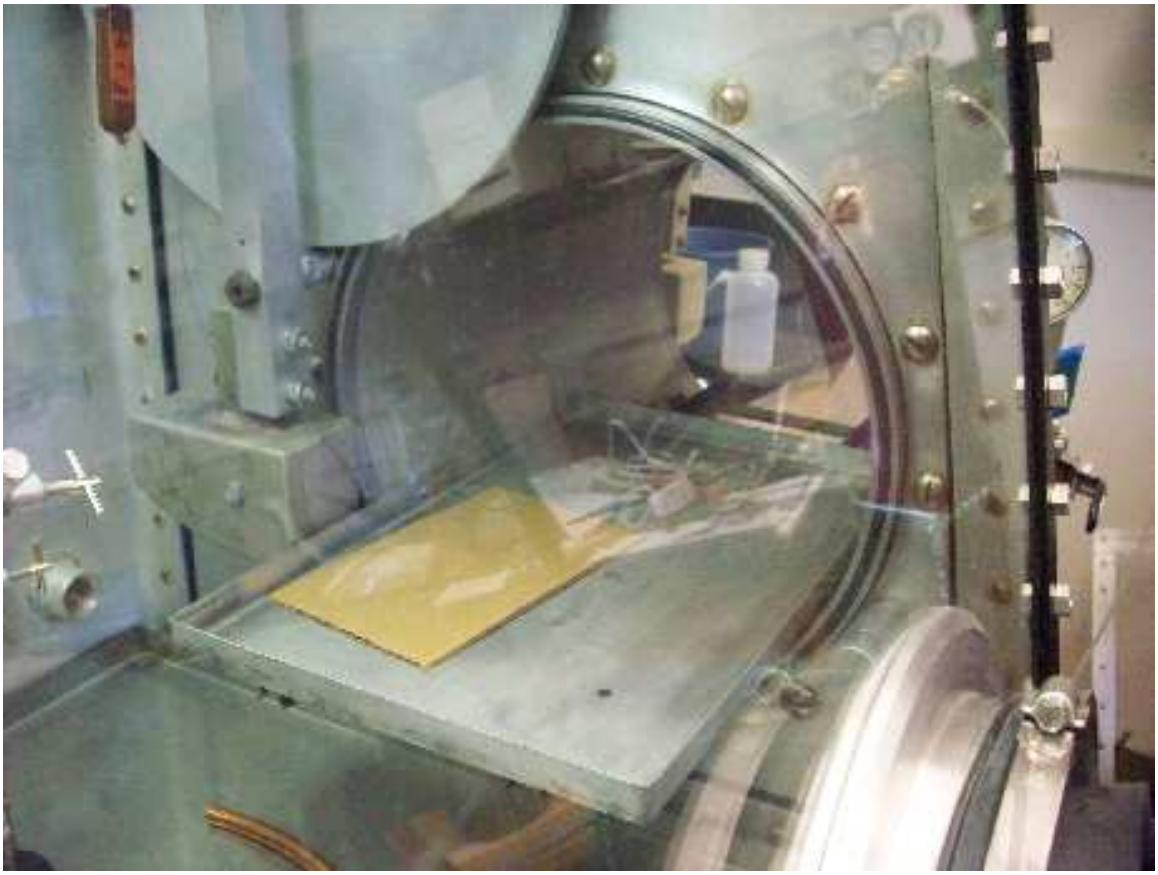


Figure B.5: A photograph of the glove box antechamber with both ends open. Note the sliding tray used to pass materials in and out of the glove box.

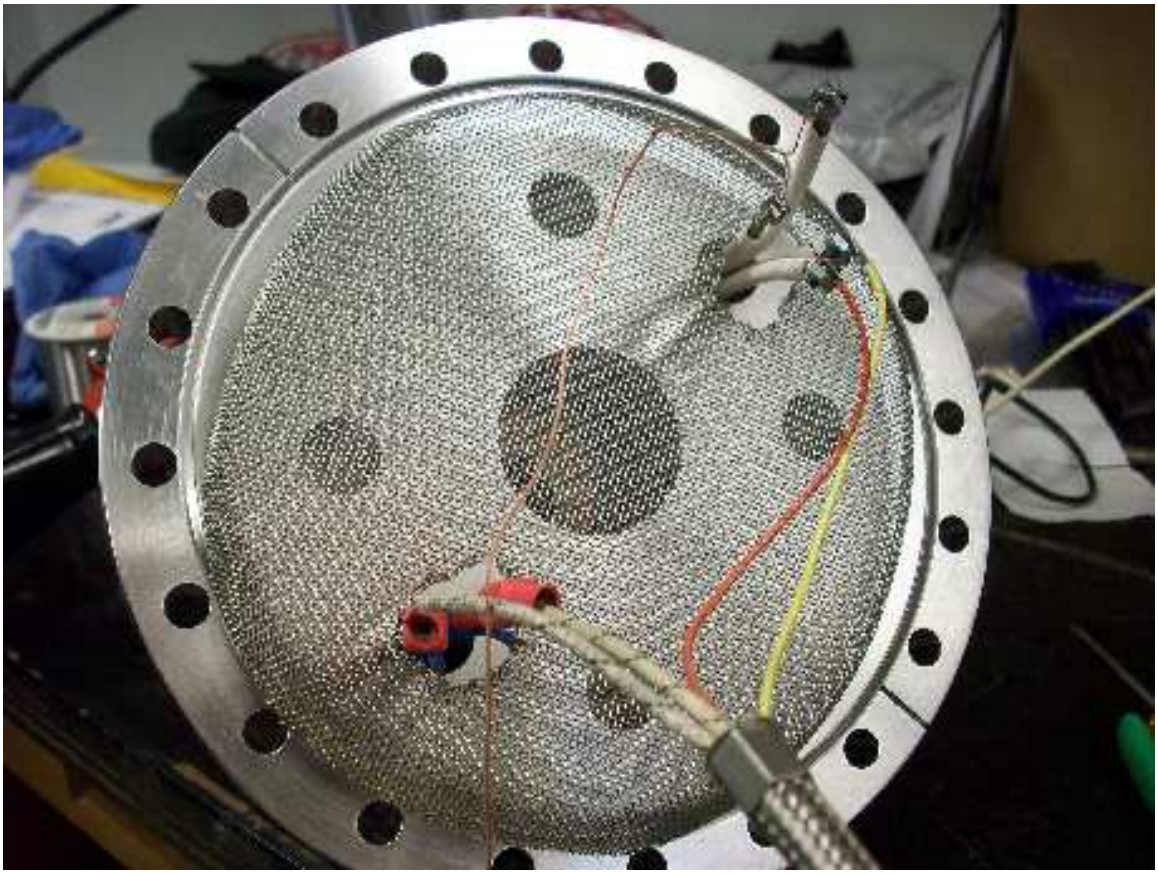


Figure B.6: A photograph of the interior of the multiport Conflat flange on top of the purifying column. On the left are the leads to the heating element, and on the right are the thermocouple leads. Note the wire mesh creating a plenum chamber at the top of the column. A matching wire mesh is at the bottom of the column.