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Design and Construction of a Lithium Vapor Oven

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CERTIFICATE OF ORAL EXAMINATION

Corey A. Leon, having **been** admitted to the Carl and Winifred Lee Honors College in 1988, has satisfactorily completed the senior oral examination for the Lee Honors College on July 14, 1993.

The title of the paper is:

"Design and Construction of a Lithium Vapor Oven"

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DESIGN AND CONSTRUCTION OF A LITHIUM VAPOR OVEN

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A lithium vapor oven to be used as a source of lithium target atoms in atomic collision experiments was designed and constructed. The oven was installed in the beamline of the Van de Graaff accelerator at Western Michigan University. Test results indicated the oven produced a diffuse cloud of evaporated lithium instead of the intended concentrated jet spray. Recommendations concerning the design are made for future efforts.

DESIGN AND CONSTRUCTION OF A LITHIUM VAPOR OVEN

by

Corey A. Leon

A Thesis Submitted to the Lee Honors College in partial fulfillment of the requirements for the Honors College program

Western Michigan University Kalamazoo, Michigan June 1993

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Corey A. Leon

CHAPTER I

INTRODUCTION

Atoms are composed of two basic units: the nucleus, which consists of densely packed, positively-charged protons and neutral neutrons, and the surrounding negatively charged electrons. The electrical charge of protons and electrons is equal in magnitude but opposite in sign. Protons have a charge of $+1$ and electrons have a charge of -1 . Normally, the number of protons and electrons in an atom are equal and thus the atom is electrically neutral. Figure 1 shows a schematic diagram of a lithium atom.

Ions are atoms with an excess or deficit of electrons. Ions may gain a net plus charge by removal of one or more electrons, while a net minus charge is the result of the addition of one or more electrons. Ionization (or electron loss) is the process of electron removal from an atom and is an important fundamental process. As part of the testing of the apparatus described here, ionization of lithium atoms was attempted using a "projectile" ion beam traveling through a "target" consisting of a spray of lithium vapor. Other elements, such as helium, which are much easier to work with since it is a gas, have been used in previous ionization experiments.

Single ionization (see figure 2) is a well understood process. Here, the incoming projectile ion interacts through an electrical disturbance with one of the target electrons causing the electron to escape from the atom. The atom then adjusts itself to a stable ionized state.

Double ionization (i.e. removal of two electrons) is not as well understood as single ionization. Double ionization is usually attributed to two basic processes. In the first (see figure 3), the incoming projectile ion interacts through seperate electrical disturbances with two of the target electrons causing both to escape from the atom. In the second process, a single ionization first occurs due to a disturbance between the projectile and a target electron followed by the ejection of a second electron during the subsequent stabilization of the resulting ion (figure 4).

Similarly, triple ionization can occur through three processes. The first consists of direct disturbance of all three target electrons by the incoming ion as shown in figure 5. The second process occurs first with a direct double disturbance followed by ejection of a single additional electron during stabilization. The third process consists of a single direct disturbance followed by the ejection of both of the other electrons during the stabilization process (see figure 6).

Investigation of the triple ionization of lithium by high velocity ions is an extension of recent studies made at WMU. In these previous studies, double ionization of helium, which has just two electrons, was induced using fast ions. Studies of the double ionization process give information on how two electrons interact or "communicate" with one another, thereby giving insight into the fundamental structure and properties of atoms. Likewise, triple ionization gives information on how three electrons interact or communicate, thereby probing further into the fundamental nature of atoms. To date, no experimental information exists on this latter process.

Lithium is used to study triple ionization because it is the simplest element in the periodic table which has just three electrons. Use of lithium is complicated because this material is a very reactive substance which spontaneously interacts with air producing sparks. To make a target, solid lithium must be heated to the gaseous state and then controlled carefully due to its metallic properties. Should lithium vapor drift onto any electrical components in the target region it could cause an electrical short circuit which may damage or destroy nearby components.

Further work on this project is continuing at the Ph.D. level based upon the knowledge of the lithium vapor oven design gained through the completion of this project.

Figure 2. Single Ionization.

Figure 3. Direct Double Ionization.

Figure 4. Indirect Double Ionization.

Figure 5. Direct Triple Ionization.

Figure 6. Indirect Triple Ionization.

CHAPTER II

THEORY

Estimated Density of Evaporated Lithium

A useful quantity for this work is the throughput of lithium in atoms per second. This measure allows us to estimate the amount of lithium being evaporated from the oven. Due to the low pressure and high temperature of lithium vapor, the ideal gas law can be used as an approximation to determine the density (in $m³$) inside the oven (Halliday & Resnick, 1970):

$$
n=133\times\frac{p}{kT}(atoms/m^3)
$$
 (1)

with $k = 1.38 * 10^{-23}$ J/°K

 $T =$ temperature (in Kelvin) inside the oven

 $p = pressure$ (in Torr where 1 Torr = 1/760 of an atmosphere)

The density helps determine the mean free path, which is the average distance between successive collisions, of the evaporated lithium atoms. As the size of individual atoms and the particle density increases, the mean free path decreases for the same volume. The mean free path of atoms is given by (Halliday & Resnick, 1970):

$$
L = \frac{1}{\sqrt{2}\pi n\delta^2} = \frac{kT}{133p\sqrt{2}\pi\delta^2} (meters)
$$
 (2)

with $\delta = 3.13*10^{-10}$ m (diameter of the lithium atom).

For temperatures between 600°C and 700°C, the corresponding lithium vapor pressure ranges between 0.05 Torr and 0.4 Torr (see figure 7) and the mean free path is between 4.16 mm

and 0.58 mm. Since these values are of the order of the radius of the nozzle $(r=2.38 \text{ mm})$, through which lithium vapor emerges from the oven, the vapor can be assumed to exhibit the properties of molecular flow. Molecular flow describes the case where the atoms of the lithium vapor emerging from the oven do not "stick" to one another during collisions, much like billard balls. In this case, the throughput (in atoms per sec) of lithium vapor is given by Moore et al. (1989) as:

$$
Q = (\frac{T}{295M})^{1/2} \times 2.16 \times 10^{21} \times \frac{d^3}{l} p \tag{3}
$$

with $M = 6.941$ au (atomic mass of lithium)

 $d = 4.76x10^{-1}$ cm (inner diameter of the nozzle)

 $1 = 3.18$ cm (length of the nozzle)

 $T =$ temperature in \mathcal{C}_K of the oven

 $p =$ vapor pressure in Torr inside the oven

The vapor pressure can be obtained from Figure 7 (Gray, 1957). The resulting dependence of the throughput (in atoms/s) on the temperature T (in \degree C) is shown in Figure 8. This dependence of the throughput on temperature (Figure 8) was used to determine the minimum operating temperature needed to give the desired amount of lithium in the collision chamber.

While the throughput gives the total amount of lithium being evaporated, it does not give the density of the lithium vapor in the collision chamber. One of the goals of the lithium oven design is to produce a concentrated jet of evaporated lithium spray rather than a diffuse cloud of vapor. Thus, the density of the lithium vapor spray at the position of the projectile beam is of prime importance. The throughput in atoms per **meter** can be calculated by dividing the

throughput in atoms per **second** by the average velocity (in meters per second) of the lithium atoms. The target thickness (in atoms/ $cm²$) is then calculated by dividing the throughput (in atoms/m) by the assumed width of the lithium jet spray (in mm). Atoms/cm² is a unit for the thickness by mulitiplying the thickness of the spray (in cm) by the atomic density (in atoms per volume [cm3]). For a 10 mm spray width, the dependence of the target thickness on the temperature is graphed in Figure 9.

M Sigure 7. Vapor Pressure vs. Temperature for Lithium (Gray, 1957

Figure 8. Estimated Throughput of Lithium Vapor in Atoms/sec Into the Collision Chamber Assuming Molecular Flow Through the Nozzle (see Figure 10).

Figure 9. Estimated Target Thickness in Atoms/cm2 Inside the Collision Chamber Assuming a Lithium Jet Width of 10 mm.

CHAPTER III

EXPERIMENT

Experimental Setup

The lithium oven apparatus was installed on an experimental station in the WMU tandem Van de Graaff accelerator laboratory in Rood Hall. Fast projectile ions (protons and He⁺) were aimed at the spray of lithium vapor which was released through a small nozzle at the top of the oven and passed through two defining slits to ensure that the spray was directed across the path of the fast ion beam as shown in figure 10. After crossing the ion beam, the lithium spray was recovered in a cooled cup at the top of the chamber.

While the ions pass through the spray, some remain unchanged in terms of charge, while others gain or lose electrons. The outgoing beam of ions is then passed through a magnet which exerts a force on the ions proportional to their charge as shown in figure 11. Ions of greatest charge are deflected the most, while ions of lesser charge deflect less. Therefore, the different charges become spatially separated and the number of outgoing ions with each respective charge can be separately counted as indicated.

Figure 10. Schematic of Lithium Vapor Oven Apparatus with Defining Apertures Shown.

Figure 11. Schematic of Experimental Setup

Experimental Procedure

The goal of this work was to design and test a lithium vapor oven. The oven was tested by determining if sufficient lithium for use in electron-capture, electron-loss, and multipleionization (loss of several electrons) experiments was evaporated. To distinguish lithium from contaminant materials which could be in the oven, e.g. residual air, dirt, oil, and oxides, the electron-capture yield vs. time was measured. Since lithium has a higher boiling point than most of these other materials, this yield (figures 14-17,19-20) is expected to converge to a nearly constant value after the other materials have boiled off.

Primary Design Considerations

Lithium is a very difficult substance to work with, especially in the setting in which we plan to use it. When working with ionized particles, electric fields are used to propel and steer the charged ions so that these ions may be spatially separated and individually counted. Electric fields are set up using two metallic plates with a voltage difference between them. Charged ions are attracted towards one plate and repelled by the other. Since the voltage difference across the plates is often large (e.g., 1000 volts) it is necessary to keep them electrically separated from one another. The oven used in the experiment is also metallic, so special care must be taken to prevent the oven from coming into contact with the electric-field producing plates. Lithium, as a metal, will also conduct electricity. Therefore, one of the main requirements of the oven design is that the lithium vapor spray be sufficiently controlled to stay off a particular set of plates which will be within a quarter of an inch of the spray.

There are two ways used to control the lithium spray, each one largely dependent upon the other. The first method for keeping lithium off the high voltage plates is to keep the lithium

spray very well defined. To do this we have designed for a spray size 9mm long by 3mm wide. This spray forms the region where the lithium vapor and the projectile ion beam will overlap and collide (see figure 10). Confining the lithium to this area is accomplished by a set of two defining apertures. One inch above the nozzle of the oven (see figure 10) is the first defining aperture and one inch above that aperture is the second. Each defining aperture is mounted in a removable disc in case the lithium spray size needs to be expanded or contracted . Figure 11 is an overall schematic of the lithium oven.

The second method of controlling the lithium spray utilizes this element's low condensation temperature. By cooling the defining apertures to the temperature of cold tap water, lithium will "stick" to the surface of the apertures upon striking them; otherwise, the lithium vapor may "bounce" off the apertures and the walls of the target chamber and eventually make its way to the surfaces of insulators and voltage plates. The path of the water flow may be traced from figure 13.

Cooling the target chamber also aids in the collection of the lithium spray after it has passed the target area. A deep, cooled cup is placed above the target area as shown in figure 13 so that entering lithium vapor condenses in this cup. The top of the cup has been made removable for easier access and cleaning.

To prevent lithium vapor from entering the beam line a set of cooled, removable aperture discs which closely match the diameter of the ion beam will be used to greatly reduce the escape of lithium into the beam line. The aperture discs will be placed in holding wells at opposite ends of the collision chamber (see figure 13).

Due to the constraints of the experimental setup, all water and electrical connections must

be made on the top of the target chamber. Figure 12 is a cross-sectional side view schematic of the lithium oven apparatus. Water flows only in the target chamber while the electrical connections are needed only for the oven below. An o-ring seal system is used to keep the chamber vacuum tight and the heater and thermocouple (used to measure temperature) wires (not shown) are fed down to the oven section. The heater wire is wrapped around the oven and nozzle. By passing an electrical current through the wire, the lithium is heated by conduction and a set of three heat shields reflect heat radiation back onto the oven. These heat shields not only help in heating, they also prevent heating of the cool target chamber. The thermocouple, which rests in a small well on top of the oven, provides a continuous monitor of the temperature. The lithium (a piece of wire of Vs" diameter and 2" length) was loaded through a hole in the bottom of the oven. A 115 W heating wire was used to heat the oven into the 500 to 600°C range (Shah et al., 1985 used 570°C).

Figure 12. Schematic of the Lithium Oven and Target Chamber.

Figure 13. Cross Sectional View of Collision Chamber.

Results

The oven was heated to 563°C for about 3 hours. Measurements of the yield of electron capture were compared on the basis of both time and temperature. The yield vs. time diagram (Figure 14) for a proton beam shows three peaks at about 10 min, 25 min, and 40 min. Figure 15, the yield vs. temperature diagram, shows these at 200°C, 390°C, and 430°C respectively. Then the yield decreased steadily over the next two hours. Following the run, the oven was inspected and it wasfound that almost all the lithium remained in the oven cell. It is speculated that the reason for this was that the pressure was not high enough to cause a significant portion of the lithium to escape through the nozzle (see Figure 7) and that the nozzle got plugged due to its small diameter. In the next run, the maximum temperature was increased to 648°C after 3 hours. At about 620°C, which is well above the boiling points of the expected contaminants, a single peak appeared at 130 min in the yield vs. time diagram (Figure 16) corresponding to about 620°C in the yield vs. temperature diagram (Figure 17). This peak coincides within one minute of an abrupt drop in the beam current by about 50% and, therefore, was assumed to be an anamoly. As in the first run, the nozzle was found to be plugged and no lithium was found in the cooled cup above the target chamber.

The fact that the nozzle became plugged in both runs suggests that the upper part of the oven cell, including the nozzle, was not sufficiently heated. This led to lithium vapor rising from the bottom of the cell to condense inside the nozzle and thus never reach the collision chamber. To compensate, one heater wire was wrapped around the nozzle and a higher capacityheater wire (700W) was wound around the oven cell. Together, these wires can easily heat the oven cell to 700°C.

Figure 14. Fractional Yield of Single-Electron Capture vs. Time for the H⁺ Beam. Maximum Temperature = 563° C.

Figure 15. Fractional Yield of Single-Electron Capture vs. Temperature for the H⁺ Beam.

Figure 16. Fractional Yield of Single-Electron Capture vs. Time for the H⁺ Beam. Maximum Temperature = 648° C.

Figure 17. Fractional Yield of Single-Electron Capture vs. Temperature for the H⁺ Beam.

In the third run, the oven cell was raised as seen in figure 18. Although this bypassed the defining apertures, this was done to try to increase the amount of lithium getting to the collision region by bringing the nozzle closer. In experiments where electric plates are not used within the collision region, the danger of creating a short is not applicable and lithium can be allowed to form a diffuse spray instead of a well-defined jet. The yield vs. time diagram (Figure 19) shows more changes than the first two runs largely associated with changes in the applied power (shown as A-D). The yield vs. temperature diagram (Figure 20) shows little change from the first two runs (Figures 15 and 17) however. (The main peak is at a different temperature because the thermocouple was not permanently fixed and thus could have differing degrees of contact with the oven.)

The nozzle heater wire did seem to prevent the nozzle from getting plugged, however considerable lithium still remained inside the oven cell. It is speculated that the nozzle was too small in diameter to allow sufficient lithium out of the oven.

Figure 18. Diagram of Modified Oven and Collision Chamber Setup.

Figure 19. Fractional Yield of Single-Electron Capture vs. Time for the H⁺ Beam.

Figure 20. Fractional Yield of Single-Electron Capture vs. Temperature for the $H⁺$ Beam.

A third set of measurements was performed using a larger nozzle and a reconfiguration of the heater wire around the top plate of the oven cell. The electron capture measurements confirmed that a significant amount of lithium was evaporated from the oven. However, lithium was found to have evaporated throughout the collision chamber which conflicts with the primary design specifications (see Chapter III). See Woitke, 1992.

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CHAPTER IV

CONCLUSION

From the results obtained, it was established that the oven did not evaporate significant lithium vapor into the collision region. This was shown by measuring the single-electron capture yield for H⁺ projectiles.

In the design, it is speculated the nozzle of the oven from which the lithium evaporated was too small so that only a small fraction of the vapor reached the collision region. A compromise in the nozzle size (between the first design and the larger size used in Woitke, 1992) is needed to provide a well defined cloud that is of sufficient density.

A reason for the poor definition of the cloud (determined by the amount of lithium on surfaces outside the cooled cup) is lithium not sticking to the walls of the "cold" cup (see figure 13). This sticking could be enhanced by cooling the cup with liquid nitrogen which is much colder. In case the cold cup starts drawing heat from the attached oven section, the connection between the two should be minimized.

It was also found that lithium condensed at the aperture between the end of the nozzle and the collision chamber. A redesign of the system to include the apertures as part of the oven cell instead of the aluminum block should avoid this problem. Another consideration is moving the nozzle closer to the collision chamber by reducing the distance between the nozzle and the apertures, between the two apertures (possibly even eliminating one aperture), and between the (top) aperture and the projectile beam line.

Due to the difficulty of tightly winding the heater wire around the oven and the inability to cover all parts of the oven evenly, it is suggested that the oven be constructed out of a material with a higher thermal conductivity. This will allow for a more uniform temperature distribution. It was found that having only one thermocouple at the top of the oven is not sufficient in light of the lithium condensation at various other places on the inside of the oven. It is recommended that at least three thermocouples be used: one for the nozzle, one for the top of the oven, and one for the bottom of the oven.

A shutter that would completely shut off the nozzle was originally intended to be included in the oven, however space constraints prevented this from being added. Future designs should include such a shutter as a priority. Also, a gas leak which would permit the introduction of He, for which capture and loss cross sections are known, into the collision chamber should be added. This would enable tests of the efficiency of the detection setup without risking the destruction of delicate instruments.

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