**Appendix 24**

# RULES AND PROCEDURES FOR OPERATION OF THE TRITIUM GAS CELL

J.E. Edwards Accelerator Laboratory
Ohio University
Athens, OH 45701
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1. <u>EMERGENCY PROCEDURES</u>

   A. If a leak of tritium gas from the cell or the tritium gas handling system to the target room or vault at large should occur, or with any indication of a possible leak from the tritium sniffer or the tritium cell pressure gauge:

      1) Evacuate the target room immediately and close door if the room is being occupied at the time of the leak.

      2) Turn on overhead fan.

      3) Notify the Radiation Safety Officer, TALC Chairman, and the Tritium Safety Officer -- phone numbers are posted in the control room.

      4) All people should remain in the accelerator laboratory area until the people in 3) above arrive.

      5) Record immediately: amount of tritium lost, individuals possibly exposed, length of time of exposure for each individual, approximate location of each person who might have been exposed from the time of rupture until leaving the target room, and finally, the sequence of events leading up to the indication of leakage.

      6) Do not re-enter the large target room or vault under any circumstance until assured by the Radiation Safety Officer that it is safe.

      7) Resumption of the experiment or any further experiments using a tritium cell should be postponed until a full critique of the gas release is completed.

   B. If a <u>slow</u> leak of tritium gas from the cell to the accelerator vacuum system should occur as evidenced by a) bad vacuum on the beam leg--with possible automatic closing of the pneumatically operated gate valves <u>and</u> b) a gradual decrease of the tritium pressure in the cell below 14 psi, then:

1) Check tritium monitor. If it reads in excess of 50 $\mu Ci/m^3$ follow procedures in A above.

2) If reading is less than 50 $\mu Ci/m^3$:

   a) Turn on target room overhead exhaust fan.

   b) Enter target room and close hand operated gate valve.

   c) Open the two valves between the gas cell and the tritium furnace to allow the tritium gas remaining in the cell to be reabsorbed by the uranium furnace.

   d) Proceed with steps A1-A7 above.

C. If a large leak of tritium gas from the cell to the accelerator vacuum system should occur as evidence by a) automatic closing of the pneumatically operated gate valves and/or b) a tritium pressure gauge reading of O psi:

   l) Proceed with steps Al-A7 above.

D. If the tritium monitor reading should exceed 10 $\mu Ci/m^3$ for an extended period of time (3-4 hours) during the course of a run, the experiment should be temporarily suspended and the source of the high reading ascertained. The TALC Chairman and Radiation Safety Officer and Tritium Safety Officer should be notified and the experiment resumed <u>only</u> if approved by them.

   NOTE: The maximum permissible concentration of tritium in air for normal working conditions (40 hrs/wk) as specified by regulation is 5 $\mu Ci/m^3$.

E. If a slow leak of tritium from the cell in excess of 0.1* psi/day is indicated during the course of an experiment, the experiment should be suspended, the entire gas cell removed and replaced.

2. <u>GENERAL RULES</u>

A. A list of individuals who have been checked out in the operation of the gas cell will be placed in the Training Flow & Documentation book located in the control room. Any experiment using a tritium cell must be supervised by at least one person on this list. That person will do all operations concerning the cell. Persons not on this list will under <u>no circumstances</u> attempt to use the cell.

* These numbers are not fixed absolutely. They may be changed as further experience is gained.

B. A Tritium Safety Officer shall be appointed whose responsibilities will be the general overseeing of the use of the tritium gas system. People desiring to be approved should apply to the Radiation Safety Officer and a series of orientation classes will be set up. These will consist of:

   l) General safety and operational procedures at the lab

   2) Familiarization of applicant with the rules and procedures regarding the tritium system

   3) Observation by applicant of the entire preparation and set up of a tritium gas cell by the Tritium Safety Officer or by someone else approved by the Tritium Safety Officer

   4) Preparation and set up by the applicant of a tritium gas cell under the direct supervision of laboratory personnel previously certified for tritium usage

   5) Final approval will be made when the applicant is able to demonstrate:
      a) proficiency in the set up and operation of the gas cell
      b) ability to use properly the helium leak detector
      c) ability to use the tritium monitor
      d) knowledge of laboratory safety procedures

C. Tritium gas cells will be used on any beam leg only if backing pumps are properly vented.

D. The Tritium Safety Officer will keep a stock of gas cells and all parts (O-rings, foil, etc.) for gas cell operation. Any approved individual requiring a cell or parts will obtain them from this person. The shop will not be authorized to fabricate gas cells for any person other than this designated individual.

E. It is the responsibility of each experimenter to keep an accurate estimate of the amount of beam run on each cell he has in his possession. No cell shall have more than 2 Coulombs of charge run on it. All used cells should be stored in the fume hood in the back of the large target room and labelled as to amount of charge, type of particle, incident energy of particle and supervisor in charge of experiment.

F. No gas cell should be used more than one time.

G. Two logs will be kept for the tritium gas system operation. One of these will be located on the gas board and information regarding filling and emptying gas cells will be recorded. A second log will be located in the control room and during a run with tritium indicated quantities concerning the gas cell shall be recorded with (at least) each shift change during the experiment.

H. Negligent operation of the cell will not be tolerated. The Radiation Safety Officer, the TALC Chairman and the Tritium Safety Officer have the authority to suspend or terminate any experiment if any of them determine the operation of the cell is unsafe.

I. The procedures outlined below were written for pulsed beam operation of the accelerator. In D.C. experiments because of the possibility of both very sharply focused beams and large current fluctuations, different procedures and precautions may be required. Before any D.C. experiment is undertaken, any necessary changes in procedure's should be formulated and agreed upon by the TALC committee.

3. PROCEDURE BEFORE BEGINNING AN EXPERIMENT USING A TRITIUM CELL

A. Submit a written request to the TALC Chairman for beam time, specifying:

1) tritium pressure to be used

2) approximate beam current

3) incident particle and energy

4) names of all people to be associated with the experiment

5) name of authorized individual in charge

B. Obtain new foil, O-rings, and a new gas cell from the Tritium Safety Officer.

C. Reread these rules and procedures to refresh yourself of the details involved in using the cell.

4. INITIAL SETUP

The procedure outlined below for attaching and filling a cell should be strictly followed.

A. The gas cell to be used will be thoroughly leak tested prior to attaching to the tritium system.

B. Tritium monitor

1) Replace filter in the tritium monitor.

2) Turn monitor on and let it run for at least 1/2 hour.

3) With intake hose disconnected, record background in the control room in the tritium filling log. The background should be about 2 to 4 on the X1 scale. If it is not, check with Don Carter to be certain the monitor is working properly.

4) Move the target room end of the sampling hose away from the tritium gas handling board and the gas cell. Connect the input hose to monitor and take a background reading. Record in the log. If this reading is higher than that in 4.B.3) above, do not proceed until the reason for the high background is ascertained and corrected. Check the tritium filling log for background levels following last use of the system.

5) The $T_2$ gas handling system should be found in the following configuration: (1) A blank-off Cajon fitting should be attached at the connector where the gas cell is to be attached. (2) The four valves between the Cajon connect and the vacuum pump should be open. (3) All other valves should be closed. BE CERTAIN THE VALVE IMMEDIATELY ABOVE THE TRITIUM FURNACE IS CLOSED.

6) Be certain the hose attached to the tritium monitor is near the gas handling system and record the $T_2$ monitor reading in the log.

C. Attaching the Cell

1) Place a fan so any possible contamination from the system would be blown away from you as you work on the board. It should be assumed that surfaces of the gas handling system are contaminated with tritium. Put on plastic gloves and wear a ring badge for the next step.

2) To attach the cell to the gas handling system, first close the valve nearest the Cajon fitting and slowly remove the blank-off cap on the Cajon fitting. Attach your cell using a new metal gasket on the flange.* Place the used gloves and old gasket in plastic garbage bags marked with "Radioactive Material" tape.

3) Slowly open the valve nearest the Cajon fitting and pump on the gas cell with the vented fore pump.

D. Leak Checking the Cell

1) The O-ring in the tritium cell provides a three-way seal and must be leak-checked in all three ways.

2) Attach leak checker to gas handling system at the flange indicated in Figure 1.

3) Get leak checker operational. When ready, close the valve nearest the vented rotary pump and leak check the cell and the entire gas handling system.

4) If no leak appears, close the valve nearest the leak checker and open the valve nearest the vented pump. If a leak is indicated, fix it and recheck the entire system.

5) Disconnect leak checker from gas handling system, immediately cap off the "vent" flange and then attach leak checker to the pumping port on the beam line.

6) With leak checker attached on the port, leak check the entire gas cell assembly. If a leak is indicated, disassemble cell and locate cause; then reattach cell and restart with Step C above in the procedure.

* See Appendix A.

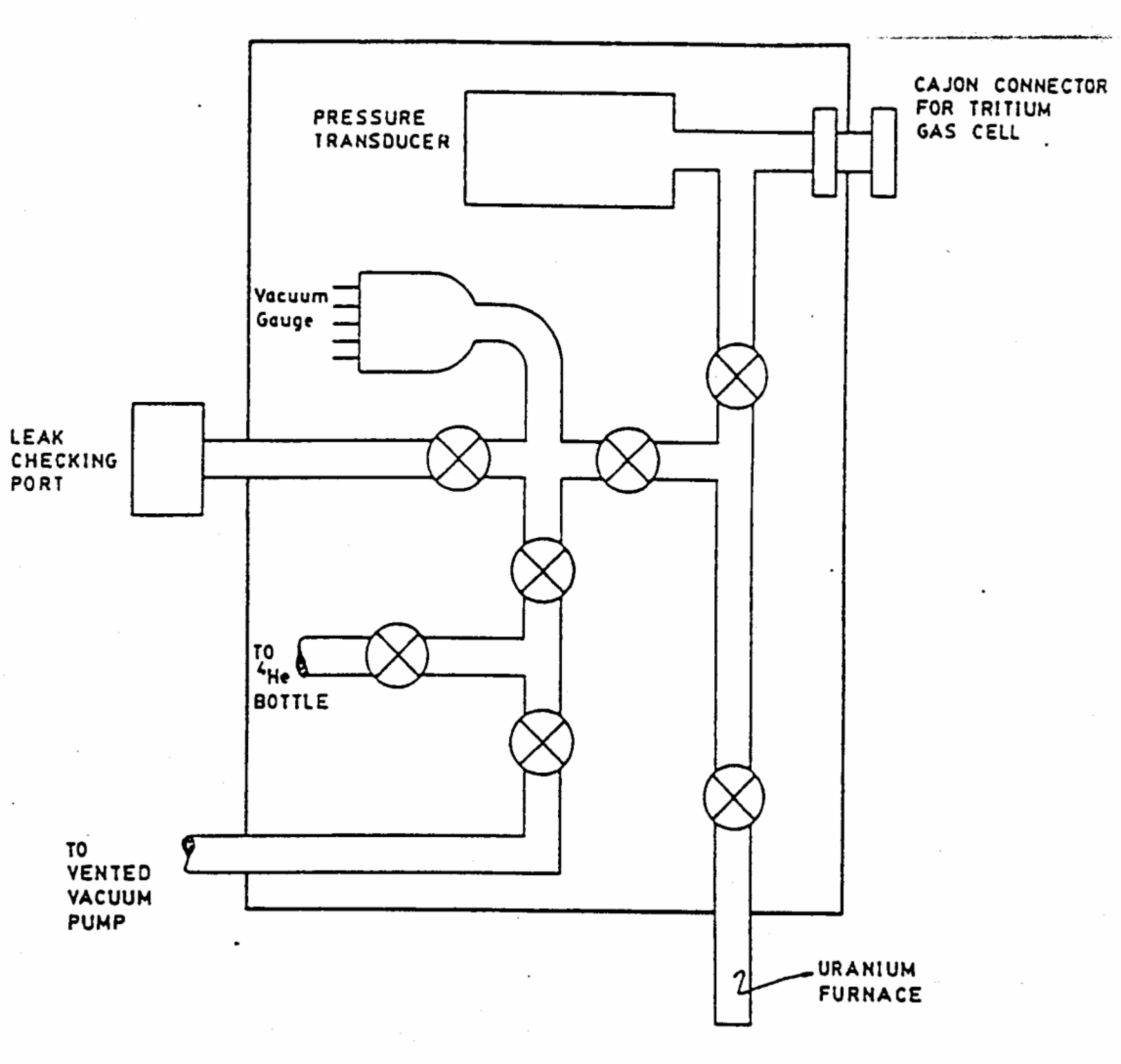



**Figure 1**
**Tritium Gas Handling System**

7) Make sure valve on helium bottle is closed and the regulator valve is turned counterclockwise until loose. With vented pump, pump out the volume of the gas handling system up to the helium bottle. Then close the valve to the vented roughing pump. Open valve on helium bottle; bottle pressure gauge should indicate gas in the regulator. Watching the tritium cell pressure gauge, slowly open the needle valve on the helium regulator. With regulator on helium bottle, very slowly increase helium pressure in the cell until the pressure there is at least 20% greater than the tritium pressure you intend to use in your experiment. Close the valve nearest the gas cel1.

8) Close main valve on helium bottle, the needle valve, and the valve on the $T_2$ gas handling system nearest the helium bottle. Open valve to vented pump and pump helium out of gas handling system.

9) Observe leak rate on the helium leak checker for at least 1 hour to be certain the gas cell does not leak into the vacuum system and record the leak rate as a function of time in the log. Compare your leak rate with past fillings in the log. If yours is similar disconnect the leak checker.

5. RUN BEAM ON HELIUM FILLED CELL FIRST

A. After leak checking and while the cell is still filled with helium, get the water cooling for the gas cell working**.**

B. Be certain solder link interlock is working.

C. Set up T.V. camera and monitor in control room to view pressure gauge on tritium handling system. If a rupture of the gold beam stop should occur during the experiment, the gauge should read atmospheric pressure. If the entrance foil should rupture, the gauge would read zero. Be certain camera is in place so these possibilities can be observed.

D. After water cooling of target is started and interlocks checked, bring the beam down the leg. Run at least the amount of beam current on the helium-filled cell as you plan to run in your experiment. This provides another check of the integrity of the cell and eliminates tuning up the beam on a tritium-filled cell.

E. After the check, remove the helium from the cell by slowly pumping on cell with vented pump.

F. Pump on the cell for at least 15 minutes (thermocouple gauge should read ~ 5 μ) to be certain all the helium is out of the cell. It is also a good practice to pump on the uranium furnace before filling the cell.

6. FILLING THE CELL WITH TRITIUM

A. Clear all people other than the person filling the cell from the target area.

B. There should be two people involved in the filling: one at the cell and one at the tritium monitor. During the filling, the monitor will give an indication of some tritium in the air during the heating of the furnace.

C. Turn on overhead exhaust fan, in large target room.

D. Close the pneumatically operated gate valve nearest the gas cell.

E. Check the vacuum gauge on the beam line: notice the reading. It should be on the $10^{-6}$ scale. Leave the gauge on the most sensitive scale possible. If there is a tritium leak to the accelerator vacuum system during filling, it will be indicated on this gauge. If the leak were large enough, the gauge would automatically turn off. If this occurs while filling, stop the filling process by removing the heater and investigate the cause of the poor vacuum.

F. Prior to filling all valves, the gas handling system should be closed except the two valves between the gas cell and the tritium furnace. The valve nearest the gas cell should be opened about one full turn.

G. Insert the thermocouple in the hand-held heater and slide the heater over the furnace. Turn on thermocouple. The uranium is near the bottom of the furnace. Hold the heater so it will heat this portion of the furnace.

H. When the furnace is heated to evolve tritium into the gas cell, the tritium monitor will indicate some tritium in the area around the furnace. To avoid any unnecessary exposure to the person filling the cell, the cell should be filled quickly but carefully. Turn the variac all the way on. The pressure in the gas cell should begin to rise at about 275º C. Remember the temperature at which you first see a rise in pressure for recording in the log after the filling, is completed. Under no circumstances should the temperature exceed 450º C while heating the furnace. The uranium furnace will be ruined if this happens.

I. When the pressure gauge reads the desired pressure, QUICKLY close the valve nearest the gas cell, remove and turn off the heater and LEAVE THE TARGET AREA. Some tritium will be in the target area immediately after heating the furnace. To avoid any unnecessary exposure, go into the control room and observe the $T_2$ monitor reading. When the reading is below ~ 5 $\mu Ci/m^3$ resume work in the target area.

J. Turn off overhead fan.

K. Keep the valve nearest the uranium furnace open for at least one hour after the filling to reabsorb tritium in the volume above the furnace. Then close it.

L. Fill out information in the log book attached to the gas handling board. The format should be as follows:

Date_____________________ Time_____________________ Op._____________________
Max. Helium Leak Rate Through Foil__________________________________________
Sampling Tube Location_______________________________________________
Tritium Monitor Background (C.R.)____________________________________
Tritium Background (T.R.)___________________________________________
Tritium Background (Manifold)_______________________________________
Gas Evolved at________________________ºC Highest Temp.____________________ºC
Final Pressure________________________ Room Temp.________________________
Comments:

7. BEFORE RUNNING BEAM ON THE CELL

A. Be absolutely certain that the water cooling on the gas cell is operating. Without this cooling, it would be possible to melt a hole through the beam stop resulting in tritium being released to the interior of the laboratory. Be certain water reservoir is full.

B. The ion gauge on the beam line being used should be operating on the $10^{-6}$ range. Any leak of tritium through the foil should be detected by this gauge and should result in the gate valve nearest the gas cell being closed.

8 WHILE RUNNING BEAM ON THE CELL

A. Be certain that the beam spot on the gas cell entrance window is as diffuse as possible. A too sharply focused beam can burn a hole through the foil. Always require a small amount of beam current to register on the entrance collimator to the gas cell 1% of total beam current). If the beam is properly aligned this will insure a sufficiently diffuse spot.

B. Never put more than 4 µA of beam current on the cell.

C. Check the gas cell pressure as monitored by the T.V. camera frequently.

D. Check the water level in the gas cell water cooling reservoir frequently.*

E. The tritium monitor alarm should be set at 8 on X1 scale. Make sure the alarm knob is on "internal."

* See Appendix B.

F. Record information in the "Tritium Run Log" at each shift change. The necessary information is:
Date______________________ Time__________________ Op.______________________
Gas Cell Pressure_____________ B.C.___________________ Energy___________________
Sampling Tube Location________________________________________________________
Tritium Monitor Reading_______________________________________________________
Cooling Alarm and Water Level Checked by___________________ at______________(time)
Comments:

G. Keep an estimate of the amount of beam current and the energy of particles incident on the tritium cel1. These numbers will be needed in the studies of radiation damage on the cell.

H. Check ventilation in large target room whenever opening the door to the target room. Air should always be flowing from the control room to the target room--<u>never the reverse</u>!

I. Always carry a chirper, and, if appropriate, the Bicron GM counter when working around the gas cel1. It should be remembered that both of these instruments are insensitive to tritium. Their purpose is to monitor any radiation coming from the region of the activated beam stop.

9. <u>EMPTYING THE CELL OF TRITIUM</u>

A. Close the gate valve on the beam line nearest to the gas cell.

B. All valves on the gas handling system should be closed at this point. Open the two valves between the gas cell and the uranium furnace.

C. Pressure should immediately drop and within a few minutes should read less than a few tenths of a psi on the gauge.

D. The uranium furnace is capable of lowering the pressure to the 10-30 micron range. However, due to the small diameter of the gas cell filling tube, this may take a while. Leave the uranium furnace open to the gas cell for at least 4 hours.

E. Close the value immediately above the uranium furnace.

F. Any remaining gas in the cell, which is likely $^{3}He$, can now be removed by pumping on the cell with the mechanical pump vented to the outside of the building.

10. <u>DISASSEMBLY OF THE CELL</u>

A. Allow a day or so for the radioactivity of the cell to die down.

B. Remember the gas cell and all associated parts are contaminated with tritium.

C. Use plastic gloves, ring badges and remote handling tools when handing the cell or associated parts.

D. Be certain all valves on the gas handling system are closed. Slowly loosen the Cajon fitting attaching the gas cell to the gas handling board. When cell is disconnected, attach a blank-off connector to the Cajon fitting using a new washer. Open the valves between the Cajon fitting and the vented vacuum pump. All other valves should be closed. Check TC gauge to be certain there is a good vacuum on the blank-off connector. Check $T_2$ monitor to be certain there was no contamination of the outside surface of the gas handling system.*

E. Label the cell with a tag indicating pressure used, type of incident particle, energy of incident particle, amount of beam, etc. Render the cell unusable by cutting the hypodermic tubing between the cell and Cajon connector.

F. Temporary storage of the used cell will be in the hood in the darkroom off the large target room.

G. Record in the log on the tritium filling board the readings of the tritium monitor when sampling at the manifold and in the target room. If these are higher than normal locate the contamination, contact the Radiation Safety Officer and the Tritium Safety Officer, and with their aid clean up the area. Information to be recorded is:

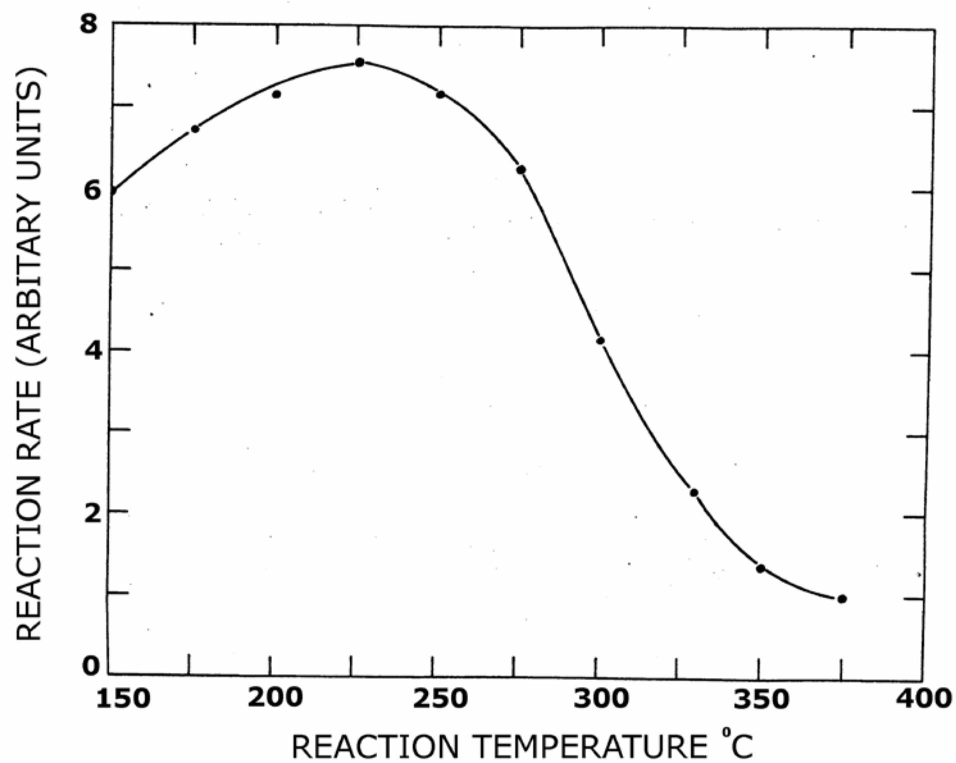
Date________________ Time________________ Op.___________________________
Furnace Open at____________________ (time)______________________________________
Residual Pressure____________________ PSI at_________________________________(time)
Furnace Closed at____________________ (time)______________________________________
Tritium in Cell_________________________________________________________total hours
Estimated Total Charge Incident on Cell____________________________________________
Tritium Background (Manifold)_________________________________________________
Tritium Background (T.R.)_____________________________________________________
Comments:

11. THE URANIUM FURNACE AND ITS USE WITH TRITIUM

A. Theory--The tritium gas is stored in a uranium furnace. This furnace consists of a stainless steel chamber which is filled with about 3 gm of pure uranium turnings. Activated uranium is an exceedingly good absorber of hydrogen gas, forming the compound $UH_3$ very readily. The rate of absorption is directly proportional to the active surface area and is strongly temperature dependent. The absorption rate has a maximum at 225º C. The rate of absorption is still rapid at room temperature. At -80º C absorption still occurs, though only very slowly. No reaction is observed at liquid nitrogen temperatures. The reaction rate decreases at higher temperatures until at 440º C, no hydrogenation takes place.

The temperature dependence of the reaction rate is shown in Figure 2. The absorption rate is also a non-linear function of the hydrogen gas pressure as shown in Figure 3. The equilibrium pressure as a function of temperature for the system uranium hydride ⇆ uanium + hydrogen is shown in Figure 4.

* See Appendix C.



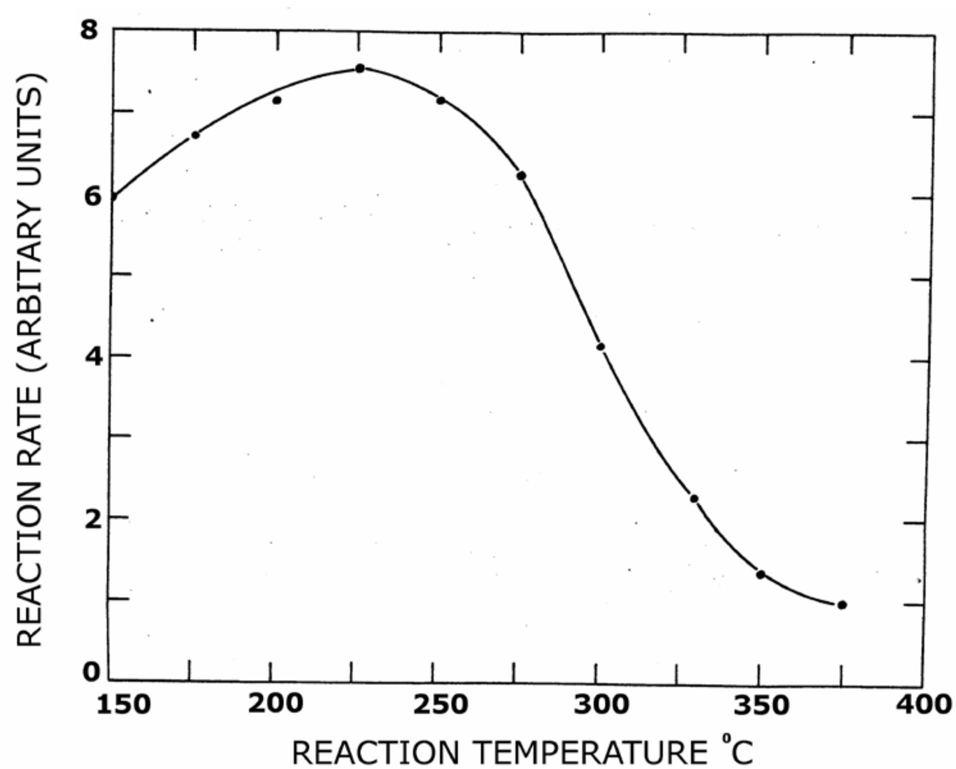




Figure 2

Figure 2

**Effects of temperature on reaction rate**

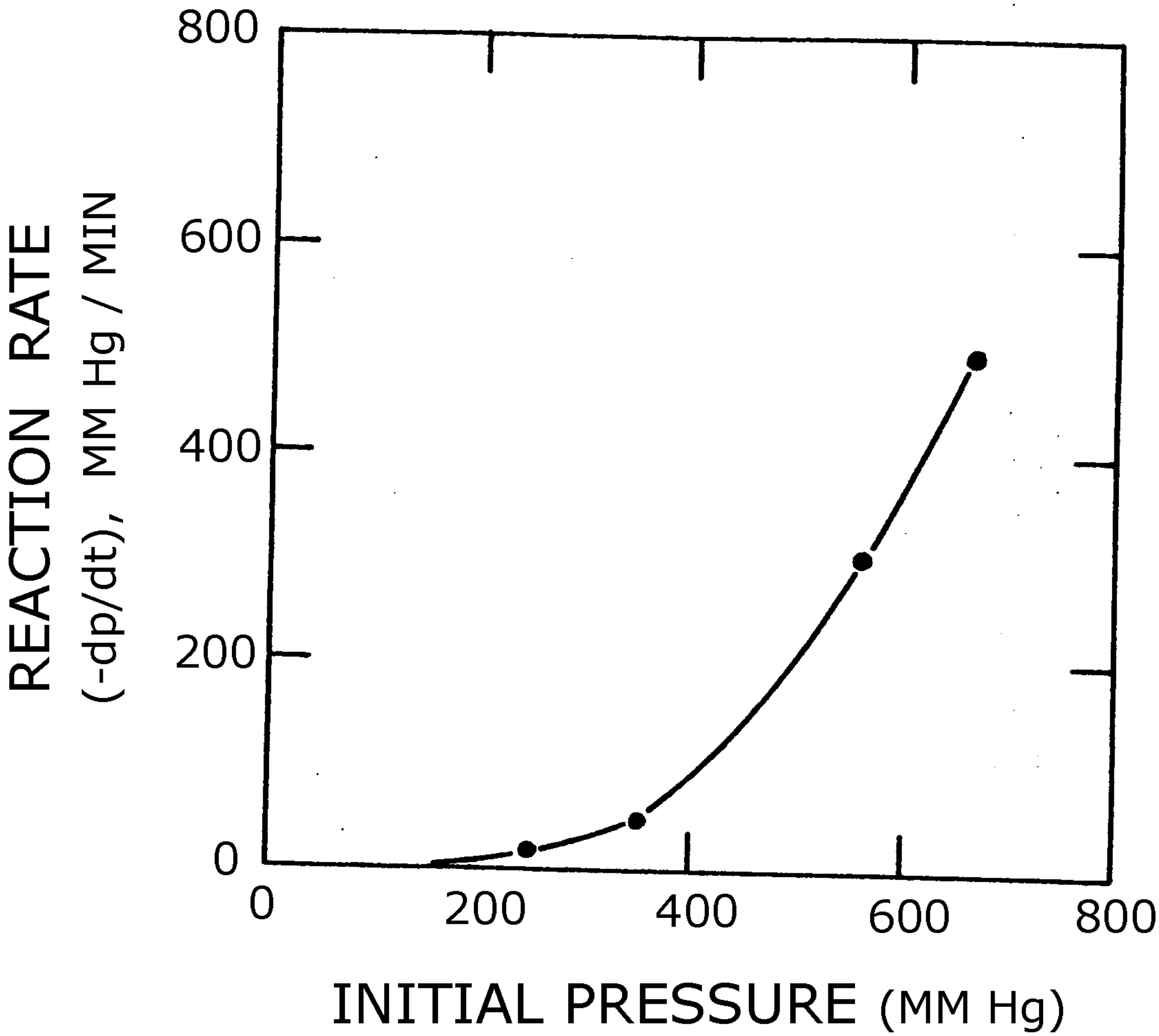



**Figure 3**

**Effects of initial pressure on reaction rate of Hydrogen & Uranium at 357 ° C**

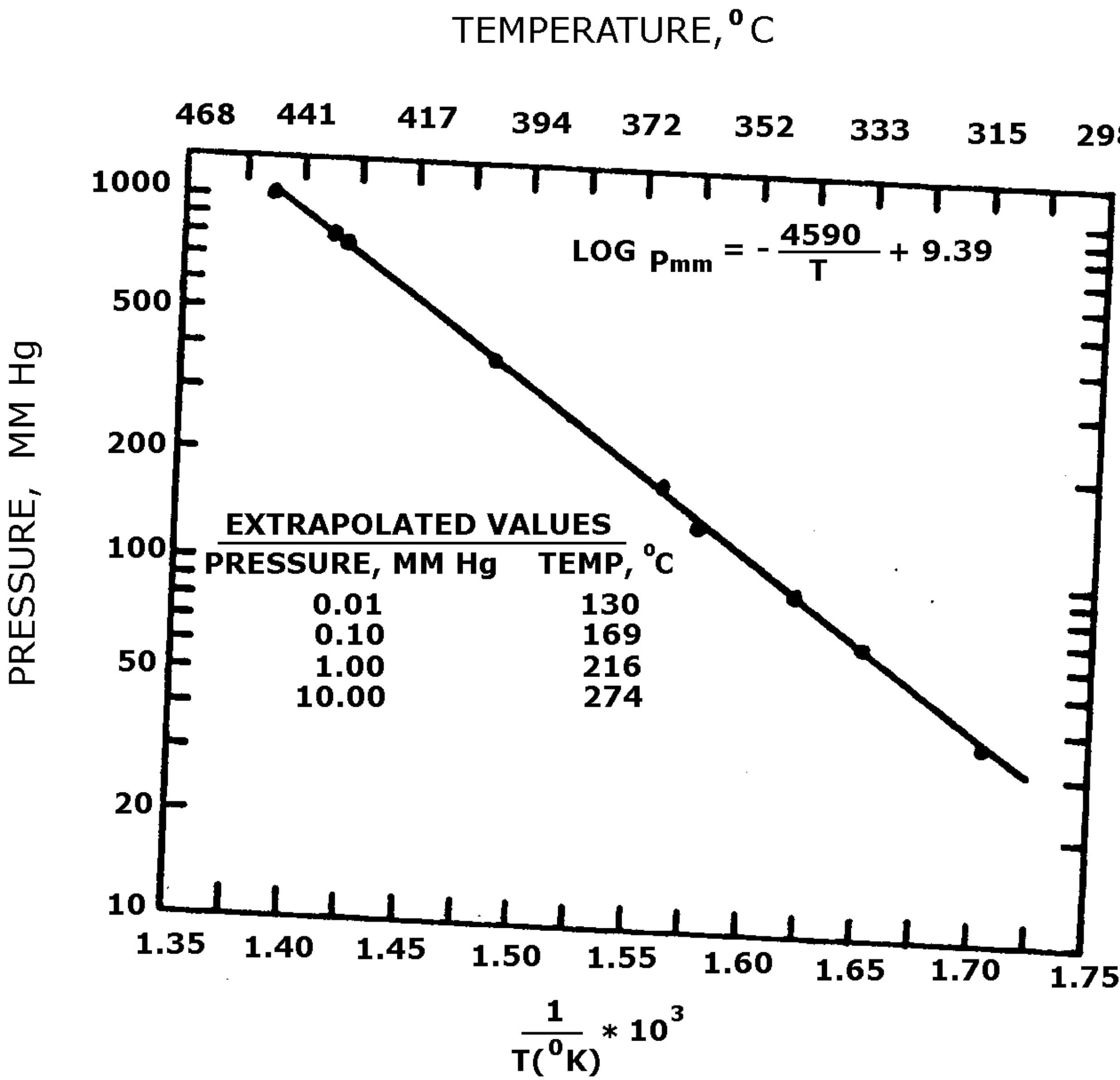



**Figure 4**

**Equilibrium pressure as a function of temperature for the system URANIUM HYDRIDE ⇔ URANIUM + HYDROGEN**

The above information is given for hydrogen. Since tritium is almost chemically identical to hydrogen, these data also apply to tritium. One gram of uranium is capable of absorbing 360 Curies of tritium. Thus, the furnace which is being used at OUAL is capable of absorbing about 1000 Curies of tritium. Presently, it is filled with 200 Curies.

A closed system of activated uranium and tritium can be used for the repeated absorption and generation of tritium gas.

B. Furnace Construction--The uranium furnace is shown in Figure 5. Basically, it consists of a stainless steel rod with the center drilled out. After being filled with uranium, the furnace was TIG welded (tungsten-inert-gas) to a stainless steel bellows valve.

All components of the uranium furnace were initially cleaned with a 30% nitric acid solution. The uranium turnings, which are normally covered with an oxide layer, were cleaned in a 50% nitric acid solution. After cleaning, the turnings were placed in the furnace (~ a 2" length) and glass wool was packed in to the remaining space. During this entire process of filling the furnace and welding, the uranium turnings were always covered with distilled water to prevent oxidation. During the welding process, all but the upper 1" section of the stainless steel furnace were held in a copper heat sink to keep the water, which covered the turnings, from boiling away. After the welding process was complete, the water was pumped out with a mechanical pump.

C. Activation of the Uranium Furnace

1) After the water was removed, the furnace was outgassed by pumping for 3 hours at 100º C. This was then repeated at 150º C for 3 hours and repeated again at 400º C for 1 hour. Finally, the system was now leaked chased. Note that during this process the system was connected to the gas handling system shown in Figure 1 with the exception that a TC vacuum gauge replaced the gas cell.

2) Approximately 85 cc's of hydrogen (same as the volume of tritium which was eventually placed in furnace) was now admitted to the vacuum manifold. This hydrogen was admitted to the furnace by opening the valve immediately above the uranium furnace. After ~ 15 minutes, all but 1% of the gas was taken up (this 1% was most likely non-hydrogen impurities which could not be taken up). These impurities were removed by pumping on the furnace and vacuum manifold.

3) The furnace was heated to 450º C to reevolve the hydrogen gas into the vacuum manifold. The temperature is held at 450º C until gas no longer evolves. The furnace is now allowed to cool and the hydrogen reabsorbed by the uranium. Any residual gas is removed by pumping on the furnace at room temperature.

4) This entire process of heating and reabsorbing the hyrdoqen is repeated 3 or 4 times. The time necessary to reabsorb the hydrogen should decrease each time until after 4 cycles. It takes only 1 or 2 minutes for the pressure to drop to ~30 microns.

5) The furnace is now heated to 450º C and all the gas pumped out. While maintaining the 450º C temperature the furnace is pumped on at diffusion pump pressures for one hour. The furnace is now ready to accept tritium.

6) The tritium is admitted to the furnace simply by connecting the tritium shipping bottle to the gas handling system and allowing the uranium to absorb the tritium. This entire operation is carried out under a hood with the "tritium sniffer" operating. As the

tritium is shipped at a pressure considerably less than 1 atm., it takes several hours for all of the tritium to be taken up by the uranium. Also, as there is about 1% of the $^{3}He$ daughter in the tritium, the pressure will not fall to 30 microns as it did with hydrogen. This $^{3}He$ gas, however, can be removed after the tritium has been taken up by simply pumping on the furnace at room temperature.

# APPENDIX A

The blank-off connector will be contaminated with tritium. When it is removed, place it inside a rubber glove and tape the glove closed with yellow "Radioactive Materials" tape. Attach the glove to the tritium gas handling system with tape so that the blank-off connector will not be misplaced during the experiment. The metal gasket used to seal the blank-off flange to the gas handling system will also be contaminated. Dispose this gasket in a properly labelled plastic garbage bag.

# APPENDIX B

What to do if solder link fails:

1. Check tritium monitor, tritium gas cell pressure and vacuum gauge nearest gas cell to be certain no leak of tritium has occurred. If a leak has occurred, follow instructions on page 1 of the tritium manual.

2. If no leak has occurred, remove tritium from the gas cell by following the instructions in Section 9 of this manual.

3. Before proceeding, be absolutely certain that valve immediately above the uranium and the one immediately below the gas cell are closed.

4. Determine the cause of the solder link failure, i.e., examine water cooling system, etc. Fix the problem.

5. Close the beam line gate valve nearest the gas cell.

6. Very carefully resolder the link. If you have never done this before, find someone in the lab who has and let them assist you. <u>DO NOT OVERHEAT THE GAS CELL</u>. <u>DO NOT ALLOW MOLTEN SOLDER TO COME IN CONTACT WITH THE GOLD BEAM STOP</u>. If molten solder should come into contact with the gold beam stop, the beam stop will dissolve and the gas cell will be ruined. Some tritium could be released if this happens.

7. Visually inspect the gas cell after the wire is reattached to be certain no damage has been done to the cell.

8. Pressurize the repaired cell with helium to a pressure slightly in excess of the tritium pressure used in the experiment.

9. If using the swinger, check the vacuum between the gas cell and the hand operated gate valve using the thermocouple gauge on the roughing line. If necessary, rough out this section. If vacuum is okay, open hand operated valve.

10. Open pneumatically operated beam line gate valve. Check the solder link interlock to be certain the gate valve will close if solder link melts.

11. Recheck gas cell cooling system to ensure adequate cooling water is getting to the cel1.

12. Run beam on the helium filled cell for several minutes to be certain the cooling system is working properly.

13. If everything appears to be working properly, remove helium from cell and refill with tritium. Continue experiment.

# APPENDIX C

After being certain that the blank-off connector is properly sealed, close the valve nearest the blank-off flange. Be certain the valve immediately above the uranium furnace is closed. When finished with the tritium system, leave only the valves between the vacuum pump and the thermocouple gauge open. ALL OTHER VALVES SHOULD BE CLOSED.