

# UCI KCCAMS Facility

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Swipe protocol  
April 28, 2011

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## I. Introduction

The use of  $^{14}\text{C}$  as a tracer has increased dramatically over the past decade, consequently contamination is becoming a significant problem. This is a danger that you should be aware of constantly. Reasonable precautions should be taken to avoid sample contamination (as well as contamination of the  $^{14}\text{C}$  processing labs and other people's samples). Precautions that should be practiced include ascertaining information about your labs' equipment history, and avoiding samples, equipment, and areas likely to be contaminated. Be wary of shared facilities and borrowed equipment. Remember that contamination does not glow in the dark (unfortunately!), and that even if you could do scintillation counter tests, in many cases the samples we deal with are so small that you would see no detectable increase in activity above the counter background.

One way of testing for contamination is by performing a swipe. In this process a quartz fiber filter is rubbed over a surface suspected of being contaminated with  $^{14}\text{C}$  tracer. The sample is taken to a special prep lab (isolated from the one where regular samples are prepared), where approximately 1 milligram of  $^{14}\text{C}$ -free carbon carrier is then added. The sample is combusted, graphitized, and the  $^{14}\text{C}/^{12}\text{C}$  and  $^{13}\text{C}/^{12}\text{C}$  ratios are measured by AMS. If the  $^{14}\text{C}/^{12}\text{C}$  ratios are significantly elevated compared to the carrier, tracer  $^{14}\text{C}$  was likely present.

## II. Conducting a swipe

Equipment seriously at risk for contamination include the three f's - fridges, freezers and fume hood. These are the most likely suspects; but ovens, vacuum centrifuges, rotovaps, bench tops, balances, and doorknobs are also frequent sources of contamination. One swipe per piece of equipment (e.g. per hood or per bench) is enough. If contamination is present, a single swipe will pick it up.

First contact the AMS facility and discuss the potential problem. We will NOT process swipes that just appear out of the blue.

To take a swipe,

1. Any surfaces that are really dusty or dirty should be wiped down beforehand e.g. with a damp paper towel, to avoid getting large amounts of dirt on the filter. If the surface is contaminated, there will still be excess  $^{14}\text{C}$  left to pick up even after cleaning. Pre-

cleaning avoids the accumulation of large amounts of modern dirt that will itself contain  $^{14}\text{C}$ , which can bias the final result.

2. Take a baked (900°C, 2-3 hrs) **25 mm diameter** quartz (not glass) filter and moisten with alcohol. The filters that are used at the KCCAMS facility are from SKC-West, Inc. (Figure 1). Any analogous filter is acceptable. The SKC-West filter can be ordered from the following address, but if only a few swipes are involved, it is usually simpler if we supply the precombusted filters.

Product	Company
Quartz Filter, 25mm, 0.4µm, 100/pk Item #: 225-1825	SKC-West, INC P.O. BOX 4133, Fullerton, CA 92834-4133 (714) 992-2780, skcwest.com

Note: i) Larger and thicker filters break apart easily and are extremely difficult to put into combustion tubes.

ii) Quartz rather than glass filters are used in order to protect the combustion tubes – glass can adhere to a quartz combustion tube and melt through it.

iii) At least in the US, ethanol is usually made from corn (i.e., contains contemporary levels of  $^{14}\text{C}$ ) whereas methanol is usually  $^{14}\text{C}$ -free. We have seen significant levels of  $^{14}\text{C}$  on filter blanks when ethanol was used (perhaps from traces of acetaldehyde residues in the ethanol). Use methanol if you can get it, but ethanol is acceptable. **In either case, blank filters moistened with alcohol must be provided (step 5 below).**

3. Rub the filter over the area to be swiped with a zigzagging motion a few times so you cover a reasonable area of the surface. Wear disposable gloves and change them after each swipe to avoid transferring contamination to the next swipe.
4. Wait a few seconds for the alcohol to dry, then drop the swipe in a Ziploc bag labeled with the name of the object swiped and any identifying information (i.e. location of object, room # etc., as appropriate).
5. Take two extra filters and moisten them with alcohol to mimic the others and put them into bags without touching any surfaces, to act as blanks. It's a good idea to do one at the very beginning of the procedure and one at the end (record which is which).



Figure 1

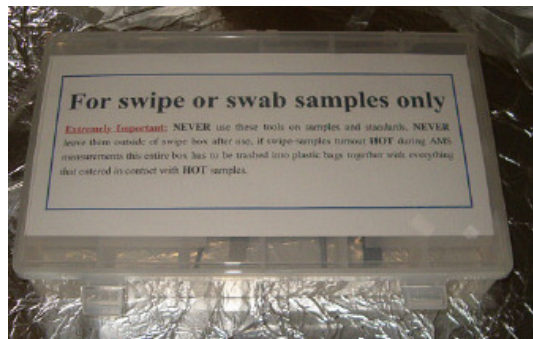


Figure 2

Fig. 1 Example of filters used for swiping a potentially “hot sample”.

Fig 2 Box containing tools for use with potentially “hot samples”. The box is located in the “AMS tools drawer” in room 239, Rowland Hall.

### III. Combustion of swipes

Each sample that comes into the lab is assigned an individual UCIG# and index card. To do this, after entering the pertinent information about the sample into the Master List on Irvine 5, locate the desktop icon **20 Index-Cards** and open it. This excel file has further instructions on how to print out the cards properly. There is also a copy of the directions on the side of the computer for further reference. Ask a lab assistant for help if you have questions.

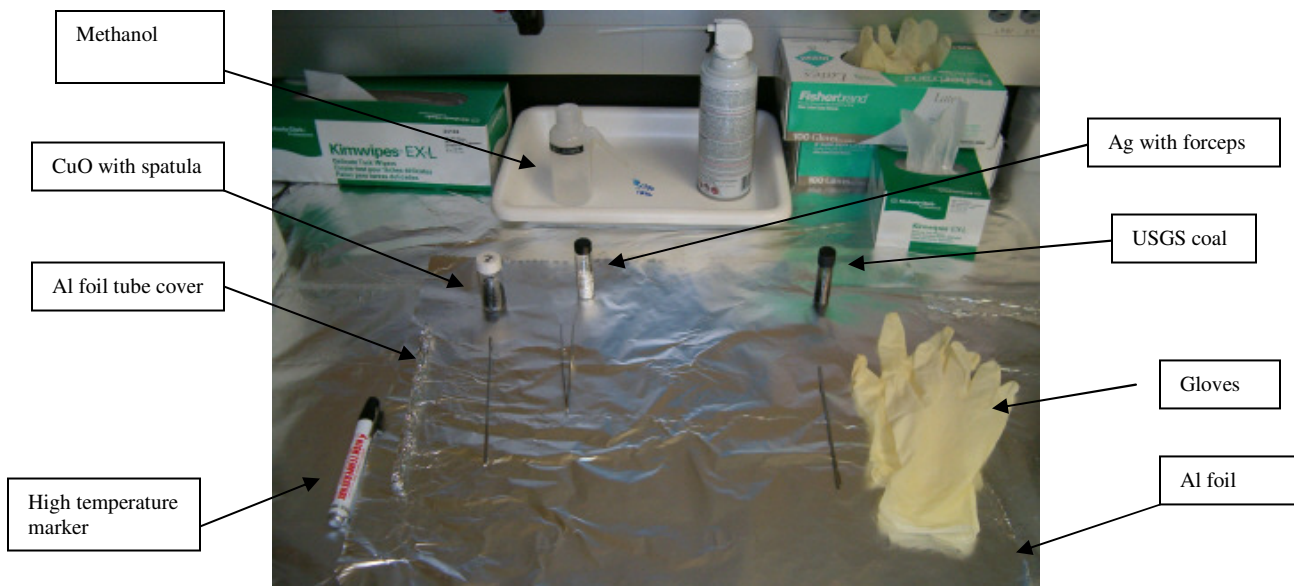
For combustion, a filter is placed inside a quartz tube (pre-baked at 900°C for at least 3 hours) along with CuO, Ag and coal. Theoretically a swipe sample contains no carbon; therefore 1 mg of coal is added to provide a <sup>14</sup>C-free carbon carrier. The quartz tube with the swipe sample is evacuated and sealed. The sample is then combusted at 900°C for 6 hours. Tools used to handle potentially hot samples are located in the swipe tools box (Figure 2) in the Swipe Lab in 239 Rowland Hall.

#### **Extremely Important:**

It is extremely important to take the necessary precautions to keep the combustion preparation station at the KCCAMS prep lab contamination free. Prior to sitting at the station, wipe the entire area with methanol. Then place an additional sheet of Al foil (to be thrown away upon completion of the combustion preparation procedure) on top of the already fitted layer. Place the vials containing Ag and CuO, as well as a spatula and tweezers on the foil. These items are found in the “Combustion Tools” drawer. You will also need a reaction tube cover (made of

Al foil) (Figure 3). USGS coal that is also used in the reaction is located in the standards drawer and should also be placed on top of the foil. It is very important that these tools never come into contact with the tools that are used for the potentially hot sample.

1. Combustion reactions for swipes are carried out in quartz 6mm x 8” reaction tubes. These are located in the wet cabinet in the KCCAMS prep lab. Place one of these tubes in an Al foil tube and tare the balance. The foil tube cover reduces the static charge generated from the quartz tubing which can affect the performance of the balance.
2. Add approximately 1mg of USGS coal.
3. Add approximately 60mg of CuO and record the weight.
4. Re-tare the balance.
5. Add one piece of silver wire.
6. Repeat steps 2-5 for one extra tube to be used without a swipe. This tube will now act as a “line blank,” which will be used to determine if the swipe graphitization line has been contaminated from the swipe samples.
7. **Take the tubes to the Swipe Lab at room 239, Rowland Hall. The following procedures will be done in this location. Be aware that the room 239 at Rowland Hall is shared with another group of people for C-14 tracer. Use only the area designated to the swipe line of the AMS lab.**
8. Using the forceps from the “HOT BOX (Figure 2)” add the filter. In order to make insertion easier, either roll up the filter or cut it into strips.
9. Always clean the tools with solvent such as methanol after each swipe and never use them on regular samples and standards. **NEVER** take them out of the Swipe Lab or leave them outside of the “swipe box” after use, if swipe-samples turn out **HOT** during AMS measurements the entire box has to be trashed into plastic bags together with everything that entered in contact with **HOT** samples.
10. Write the lab number of the sample (taken from the sample master list) using the high temperature marker on the outside of the tube and place it on the evacuation line (the top section of the line in Figure 4). Once the needle of the vacuum gauge reaches baseline,
- 11.
12. the tube is ready to be sealed.



*Fig 3 Necessary tools for combustion.*

#### **IV. Sealing quartz tubes**

The torch located in front of the evacuation line is used for sealing the tubes. Put on the safety goggles for quartz sealing.

1. Turn on the gas supply valves of the natural gas and O<sub>2</sub> bottle (O<sub>2</sub> should be at approx 10psi).
2. Turn the red knob on the torch in the direction opposite of the arrow just enough so that you hear gas coming out.
3. Point the torch away from you and use the striker to light the torch.
4. Use the green knob to add oxygen. The more oxygen applied, the smaller (hotter) the triangle will become.
5. The red and green knobs are then adjusted to get the appropriate flame. The bright blue part should make up approximately ½” of the overall 8” flame.
6. Sealing the tubes involves heating and rotating until the quartz becomes soft (figure 4). This can be accomplished by leaving the flame in one position on the tube until that area becomes white hot (it will appear bright green through the safety goggles). Once the glass has become white hot, rotate the tube to an area that has yet to be heated. Continue this procedure until a bright, narrow, white hot ring persists around circumference of the tube. If the ring does not form, the flame is probably too cold. Adjust the knobs to increase gas and oxygen.

7. At this point the quartz should be soft. Slowly pull the lower part of the tube away from the upper portion.
8. In order to make the newly sealed tube stronger and prevent breakage, melt back the sharp point into a smooth blob. Place the tube on a metal surface to cool.
9. Turn off the oxygen (oxygen first!) and gas valves at the torch.
10. Turn off O<sub>2</sub> supply valve and bottle.
11. Turn off the gas supply valve on the bench.

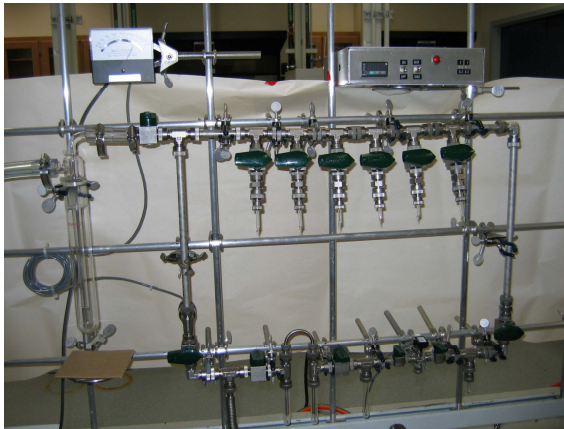


Fig 4 The top section of the line is for sealing sample tubes.



Fig. 5 NEY 2-525 furnace and ceramic tubes.

## V. Placing combustion tubes into the furnace

1. Place tubes by numerical order into the ceramic tubes in the furnace (Figure 5). Combust the tubes at 900°C for 2 hours.
2. The combustion process can be programmed as follows:

Rate 1 = 15

Rate 2 = 15

Temperature 1 = 500

Temperature 2 = 900

Time 1 = 0.1 hour

Time2 = 2 hours



- Check the settings every time before using the furnace. To change the settings, press the PARAMETER button, punch in the desired number and press ENTER. Press the start button to start the program for combustion.
3. Once combustion has been completed, retrace the numbers on the tubes with a sharpie marker to make them more legible.
  4. Place them into a mug together with their index cards. They are ready for graphitization.

## VI. Making tubes for graphitization

Pyrex tubes to be used must be pre-combusted for 3 hours at 550°C. The 8-inch long Pyrex tubes are made from 9 mm Pyrex tubing. A small dimple is made about one inch from the base of the tube. A small 6mm tube will rest on the dimple as shown in Figure 6. The 9mm Pyrex tubing and 6mm insets are in room 2222, Croul Hall. In the bottom of a 9 mm Pyrex tube put 30 mg Zn powder and 15-20 mg of TiH<sub>2</sub> (flammable) using the designated scoops. Be sure not to get Zn or TiH<sub>2</sub> on the sides of the tube.

Zn, TiH<sub>2</sub> and Fe powders and the designated scoops are located in the upper right drawer of the counter near the balance in room 2222, Croul Hall.

Put 5 mg of Fe powder in the bottom of a small (1.5 inch) 6mm Pyrex tube using the designated scoop. Be sure to wear gloves while handling the 6mm tubes, any carbon from your hands will become part of the sample.

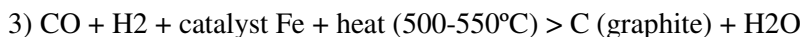
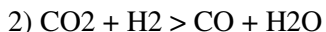
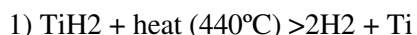
Gently place the small tube inside the 9mm tube by tilting the 9mm tube at a 45° angle and letting the small tube slide gently to the bottom.



*Fig. 6 Reactor of 9 mm Pyrex tube with 6 mm tube inside.*

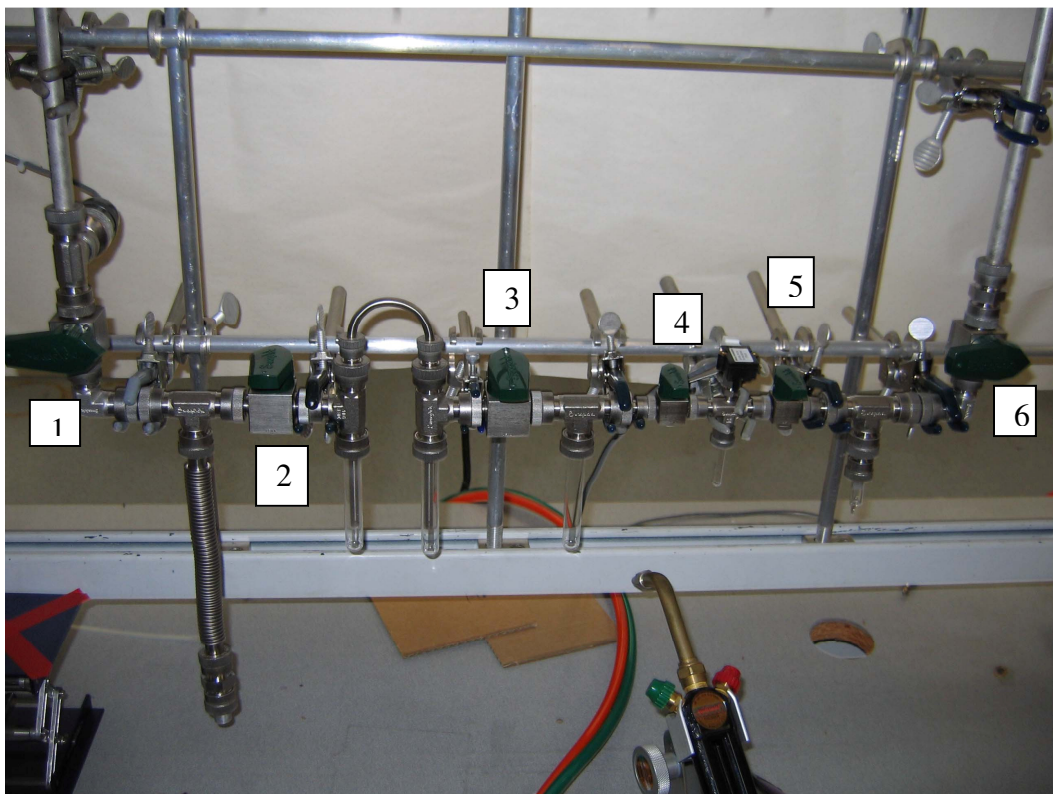
## VII. Graphitization (zinc reduction method)

The reduction reactions of CO<sub>2</sub> to graphite involved during graphitization:



The vacuum line for swipe samples is located in room 239, Rowland Hall (Figure 4). The procedure of graphitization is as follows:

1. Fill liquid nitrogen into the main trap at the upper left corner. Remember to top off this dewar periodically throughout the process.
2. Open all the valves of the entire line to the pump. Check for leaks by making sure the vacuum has reached baseline.
3. Prepare a methanol and dry ice slush for the water trap.
4. Place the slush on the water trap. This will remain on the line all the time while you are working. You will need to refresh the slurry by adding dry ice every 2-3 hours.
5. Score the sample tube with a glass cutter. Isolate the bellows by closing valve 1 and valve 2 (valve locations are shown in Figure 7) on both sides of the bellows, then remove the bellows. Gently place the sample inside the bellows. Be careful not to drop the sample into the bellows. Return the bellows to the line and tighten finger-tight. Open valve 1 to evacuate the bellows. Then open valve 2.
6. Close valves 5 and 6 to put a new Zn-TiH<sub>2</sub>-Fe tube on the line. Then, open valve 6 slowly to evacuate. If you open the valve too quickly, the powders inside the tube will jump around and could potentially mix with each other. Make sure all the valves are open to evacuate the entire line and that the vacuum reaches baseline.
7. Close valves 1, 2, 3, 4, 5, and 6.
8. Place a dewar filled with liquid nitrogen under the CO<sub>2</sub> trap. Let cool for a minute before continuing.
9. Crack open the sample by bending the bellows gently. Open valve 2. Wait for half a minute to allow the water to be frozen into the trap. Then, open valve 3. Wait for two minutes for all the CO<sub>2</sub> to be collected in the CO<sub>2</sub> trap. Top off with liquid nitrogen before continuing.
10. Open valve 4 to measure non-condensable gases and record the reading of the pressure. If the pressure can not be read here, close the valve to the pump and open valve 1. Now read the non-condensable gas pressure at the pump gauge and record.



*Fig. 7 The bottom line for graphitization.*

11. Open valve 1 to pump away any non-condensable gases. Or if you have read the non-condensable gas pressure at the pump gauge, then open the valve to the pump to pump them away.
12. Close valves 1, 2, and 3. Switch the liquid nitrogen dewar from the CO<sub>2</sub> trap to the cold finger under the pressure gauge head. Let the CO<sub>2</sub> be transferred from the CO<sub>2</sub> trap to the cold finger by warming the CO<sub>2</sub> trap to room temperature with a heat gun or with your hand. Be careful not to over heat the tube. Raise the liquid nitrogen level in the dewar and wait for 30 seconds to ensure a complete transfer.
13. Close valve 4. Warm the cold finger with your hand.
14. Record the measured volume shown on the pressure gauge. One milligram of carbon yields about 480 torr of CO<sub>2</sub>. It is very important to record the actual CO<sub>2</sub> yield, because no more than 480 Torr is expected for 1 mg of carbon. Any excess CO<sub>2</sub> can only be from two sources: dirty swipes or <sup>14</sup>C tracer.
15. Fill a mini-dewar with liquid nitrogen and place on the Zn-TiH<sub>2</sub>-Fe tube.
16. Open valve 5 to freeze sample CO<sub>2</sub> into the Zn-TiH<sub>2</sub>-Fe tube.
17. Top off the liquid nitrogen level in the dewar and wait for 30 seconds. Seal the Zn-TiH<sub>2</sub>-Fe

tube with torch. The sample tube is ready for combustion.

18. Place sample tubes in the holes of the heating block as shown in Figure 8.



*Fig. 8 Furnace with heating block inside.*

19. Put the heating block in the furnace to combust at 500 and 550°C for 7 hours. The program for graphitization settings is as follows:

R1 = 15

R2 = 15

Temperature 1 =500

Temperature 2 =550

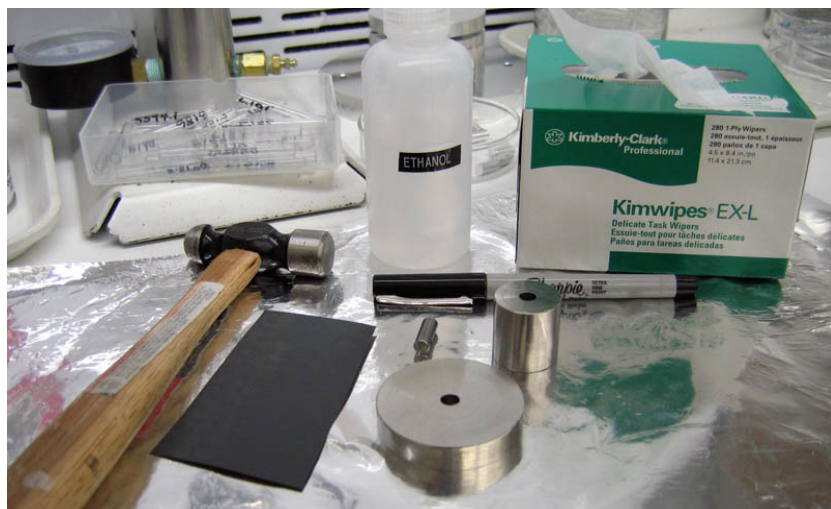
Time 1 = 3 hours

Time 2 = 4 hours

When the graphitization is done, take the sample tubes out from the furnace with the heating block. They are ready to be pressed into the cathodes for AMS analysis. Pressing is also done in the Hot Lab, room 239, Rowland Hall.

### **VIII. Procedure of how to press using UCI cathodes**

1. Put a sheet of aluminum foil down on the bench top.
2. Take out the pressing tools (Figure 9), which consist of the portable pin and the UCI cathode holder. Clean the pressing tools with both methanol and kimwipe, and then with the air duster. Sand the pin on sandpaper, clean with a kimwipe and finally air dust it.



*Fig.9 Pressing tools.*

3. Take a drilled UCI cathode and write down the UCIG number on the cathode using an ultra-fine sharpie.
4. Place the cathode into the UCI cathode holder.
5. Take the correct sample. Score the bottom of the tube just above the dimple. Gently pop off the end of the tube. Carefully slide out the inner tube containing the graphite. Turn the graphite tube upside down so that the graphite falls into the funnel shape of the cathode (Figure 10).
6. Using the portable pin, gently crush the graphite, if necessary, into powder.
7. Press graphite into the hole of the UCI cathode using force from your hand. Do this until all of the graphite is in the hole.
8. Place the sleeve over the cathode. Place the pin into the top of the sleeve. Lightly tap the pin with the hammer. Then give the pin 2-3 hard hits. Remove the pin and sleeve (Figure 11).



*Fig.10 Putting graphite into the cathode.*



*Fig.11 Using a hammer to pound the graphite.*

9. Remove cathode and tap it upside down to eliminate any residual graphite from cathode funnel and then place into the correct position on the wheel.
10. Both the portable pin and the UCI cathode holder need to be cleaned as well as the area surrounding each sample.
11. When all the samples are pressed, make sure everything is put away.
12. Throw away the foil that was used on the bench surface.
13. Wrap all the cathodes in a piece of aluminum foil. Take the cathodes along with their index cards to the BACK of the AMS machine lab. **DO NOT TAKE THE SAMPLES INTO THE PREP LAB!**