UCI KCCAMS Facility

Graphitization protocol -
Hydrogen reduction method
June 14, 2004

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I. Introduction

In the ^{14}C AMS technique, the element of interest (carbon sample) is chemically separated from the original sample and loaded as a solid target (graphite) in the sputter ion source of the tandem accelerator. The procedure to convert the raw sample material into a graphite target suitable for the ion source includes a series of necessary steps (removal of macroscopic contaminants, chemical cleaning procedures, combustion and graphitization). These steps are essential for a reliable AMS measurement.

This protocol outlines the graphite sample preparation procedure at UCI KCCAMS prep-laboratory for **organic** and **carbonate samples**. Through this process the CO_2 produced from carbonaceous raw materials are cryogenically purified (separated from non-combustible gases) and reduced to solid graphite.

II. Graphitization reaction

To catalyze the production of small amounts of elemental carbon from CO_2, we use the Bosch reaction (Manning and Reid, 1977) that can be summarized as:

$$CO_2 + 2H_2 \xrightarrow[\text{Catalyst (Fe or Co)}]{\text{550 - 650°C}} C + 2H_2O$$

The reaction takes place as two successive reductions: first to carbon monoxide and then to carbon, which permeates and adheres to the surface of the iron powder (catalyst). More details on graphitization can be found in Vogel et al, 1984.

During the graphitization procedure at the KCCAMS prep-lab, cryogenically purified CO_2 is transferred into pyrex culture tubes (Lloyd et al, 1991) and reduced to graphite, using hydrogen over pre-cleaned (preconditioned or reduced) iron powder at 550°C a for maximum of 3-4 hours.

III. Graphitization Procedure

The graphitization line (figure 1) at UCI KCCAMS prep-lab has 12 H_2/Fe reactors, allowing us to graphitize 48 organic or carbonate samples per day. The vacuum lines are made of glass and stainless steel and are pumped by turbomolecular pumps backed by oil free diaphragm pumps

(Santos et al, 2004). The design was based on sample graphitization lines from CAMS/Lawrence Livermore National Laboratory (LLNL).

In order to begin the graphitization procedure, make sure that you are signed up for the correct day and time on the sign-up sheet located on the upper left of each line. Before beginning check and sign the "magnesium perchlorate use" card above the line. If the three spaces to sign are filled then you must change the magnesium perchlorate (section X). However, if there are more spaces available, then sign in your name and proceed to section IV (Preconditioning the Fe catalyst).

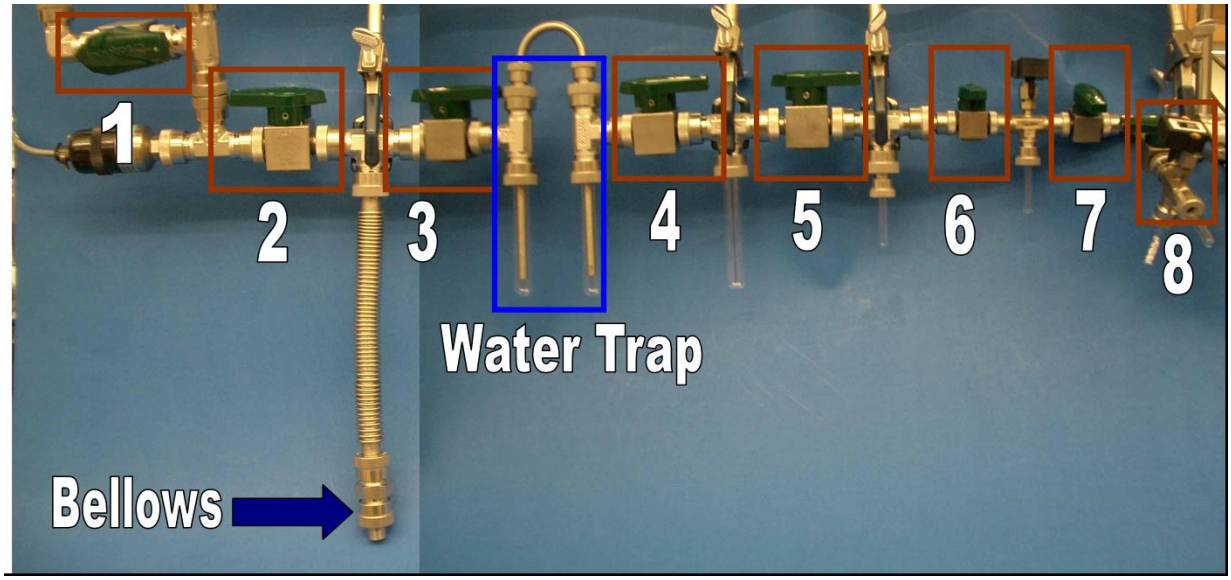

Fig.1: Picture showing the main section of the graphitization line with labeled valves

If you are using a Fe catalyst: When you use Fe as a catalyst you MUST FIRST PRE-CONDITION the Fe. This procedure takes 1 hour to complete.

1. If there is a card that states "Preconditioned" on the graphitization line then you can skip this step.
2. If the graphite-reactor tubes have something in them but there is no sign to clarify what is in the tubes, talk to an AMS assistant.

3. If the graphite-reactor tubes are empty follow the procedure below.

IV. Preconditioning the Fe catalyst:

1. Take 12 baked pyrex graphite-reactor tubes from the wet cabinet on the island tabletop.

2. In a drawer labeled “graphitization tools” there is a box (Each graphite line has its own tools in its corresponding island). From inside the box take out the following (figure 2): a) vial of Fe powder (catalyst) and b) Fe scoop.

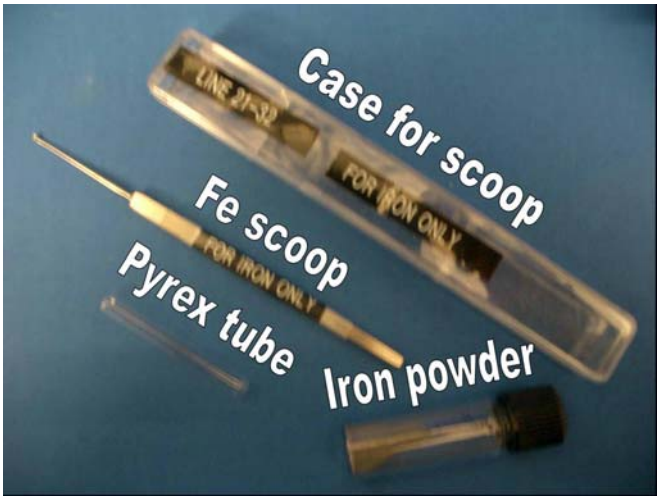

Fig. 2 Tools necessary to load Fe-catalyst into the graphitization line.

3. WEARING GLOVES, scoop 2 spoonfuls (level the spoon of excess Fe) of Fe into each tube. Evenly distribute iron inside the pyrex tube walls (only at the tip) by rolling the tube on a sterilized surface before placing into reactors.
4. After all of the tubes have been filled remove the reactor tubes already on the reactor and replace with the tubes containing Fe. MAKE SURE THE TUBES ARE SECURELY FASTENED INTO THE REACTOR
5. Leave valves # 1-7 (figure 1) completely open and wait until the vacuum reaches baseline (vacuum gauge 0-5 millitorr). Vacuum out all reactors by opening the valves slowly (Fig.3a and 3b). Pause when you see the pressure gauge reading (left-hand end of the line - above valve 1) start to rise, open the valves fully when the pressure reading starts to fall.

Fig. 3a Valve in closed position

Fig. 3b Valve in open position

6. Monitor the vacuum by looking at the pressure gauge. A good vacuum should be about 0-5 millitorr.
7. Flush entire line (including bypass line) with H_2 at least 3 times. Open the H_2 valves # 9 and 10 (figure 4) to allow H_2 to flow into the reaction line. Then add H_2 by carefully opening valve #11. Evacuate the entire line and wait until the millitorr gauge reaches its baseline again (0-5 millitorr).
8. Once the gauge reaches 0-5 millitorr again close valve # 1 (Fig. 3a)
9. Now you will add between 1/2 to 1 atmosphere (380 - 760 Torr) of H_2 to each reactor to aid in the cleaning of the Fe (preconditioning of the Fe). Open carefully valve # 11 again. Close it when pressure desired is achieved. You can monitor the amount of H_2 added by looking at the pressure readings above the line.

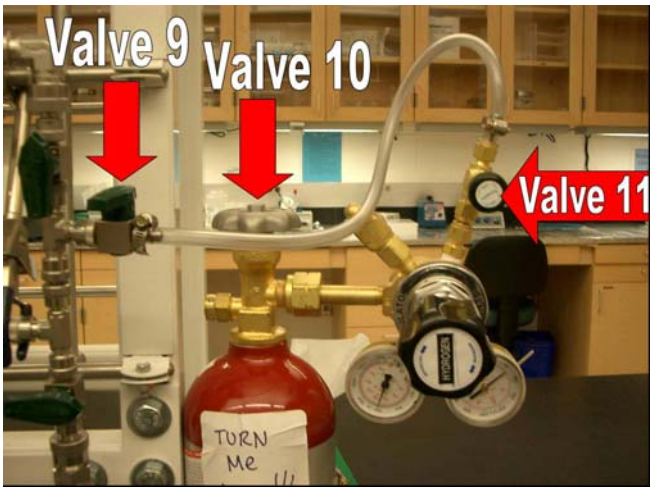

Fig. 4 Hydrogen cylinder with labeled valves

10. If you have added between 700-800 torr proceed to step 10. Avoid exceeding these values so that H_2 is not wasted.
11. If, however, you have exceeded 1500 Torr then you must vent some H_2. Close valve # 5 and then open valve # 6 (figure 1). You will notice that the pressure readings have lowered. If your new reading is < 1500 Torr then proceed to step 10.
12. If you have not exceeded 1500 Torr, then close all the graphite-reactor valves and the H_2 valves that were opened.
13. Pump the excess H2 that is trapped in the line. DO NOT BE ALARMED IF THERE IS A LOUD SOUND, THIS IS THE EXCESS H_2 BEING PUMPED AWAY.

Be careful not to flood the pump with more than 1 atm (760 torr) of H_2. Excessive amounts of gas may cause the pump to auto shut off or even damage the pump. To check that the pump is still operational look for the little green light on the pump controller and check that the pump cooling fan is still working. If the light is red, seek assistance.

14. Slip reaction tubes into the corresponding heaters and set the timer and temperature controls (figure 5). To set the temperature make sure all the controllers are on (the current temperature should be indicated for each reactor) **FOR PRE-CONDITIONING THE Fe THE TEMP. MUST BE 400°C.** Check the set temperature by pressing the "sel" button next to each pressure monitor. This will display the set temperature.
    a. If the set temp is 400°C proceed to part c.
    b. If the set temp is not 400°C then use the up and down buttons to raise or lower the set temp and then reselect by pressing "sel".
    c. Double check that the temp set is at 400°C by pressing the "sel" twice.

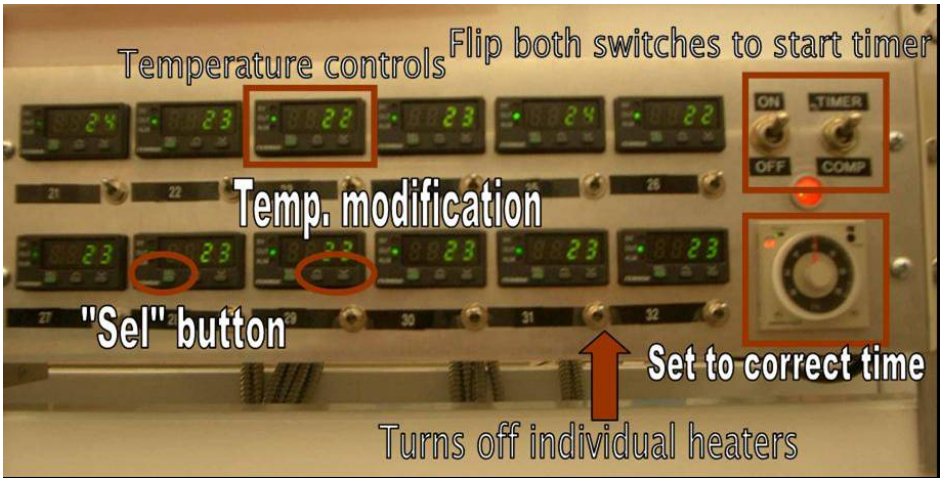

Fig. 5 Control locations to set temperature and timer

15. To set the timer, turn the dial to 1 hour and then start the timer by flipping the power switch off and then on again.

V. Preparation for graphitization:

Before you begin graphitization you must

1. Prepare the slush (dry ice + ethanol) for the water trap (figure 1). If you do not know how to prepare the slush go to section XI.

2. Get 2 dewars of liquid nitrogen. Liquid nitrogen is provided in the SW corner of the prep lab next to the pressing station. IF YOU HAVE NEVER GOTTEN LIQUID NITROGEN BEFORE PLEASE ASK FOR ASSISTANCE.
3. Vacuum out H_2 from preconditioning. Vacuum out all reactors by opening valves # 1-7, then open all the reactor valves (slowly). Monitor the vacuum by looking at the pressure gauge. A good vacuum should be about 0-5 millitorr (will vary from gauge to gauge). Then close reactor valves, and valves # 2-7 and both bypass valves.
4. Start to fill out your cards. For each sample YOU MUST HAVE AN INDEX CARD WITH YOUR ASSIGNED UCIG NUMBER. Every sample must be entered into the computer and have a corresponding index card. **At this point you should write down the date of graphitization, your name and the number of the reactor(s) that each sample will be in. Following, example of UCIG card (new info is in blue. The info in green will be explained later):**

UCIG#		**SUBMITTER NAME**		
DATE		**SAMPLE ID**		
		DESCRIPTION		
		(PRETREATMENT APPLIED)		
DATE OF COMBUSTION		**SAMPLE WT.**	**CuO Wt.**	**Ag**
GRAPHITZATION DATE		**YOUR NAME**	**Measure Volume (Torr)**	**X (multiplier value)**
REACTOR #	**CO2 (Torr)**	**H2 (Torr)**	**CO2+H2 Total (Torr)**	**Res (Torr)**

Fig. 6 The correct way to fill out an index card

VI. Graphitization:

VI.1. Using combusted organic samples

1. Score the glass using a glass cutter found in the "graphitization tools" drawer in the plastic box. This prevents unnecessary wear and tear on the bellows.
2. Clean your combustion tube using ethanol and a Kimwipe. Be aware that this will erase any label you may have written on the tube.
3. Check that valves # 2, 3, 4, 7, and the bypass valves are closed, then unscrew the bellows. If there is glass in the bellows discard into the glass waste. Do not accidentally discard the end support for the bellows (figure 7) – either take it out or hold it so it does not go into the glass waste container.

Fig. 7 Bellows with detachable end support

4. Securely fasten the bellows back into the line
5. Open valve # 2 to evacuate the air in the bellows.
6. Monitor the vacuum by looking at the pressure gauge. A good vacuum should be about 0-5 millitorr. If the vacuum does not reach 0-5 millitorr in one minute or so then open valve # 3. Some water may be trapped in the bellows. Once the gauge reaches baseline close valve # 2.
7. Crack the tube that is in the bellows between valves # 2 and 4.
8. If valve # 3 is not open yet, open it to allow the gasses to go through the water trap for about 1 minute to 3 minutes depending on your sample. During this step, water that is in your sample will be "trapped". So if your samples contain a lot of water you should wait for at least 2-3 minutes.
9. While you are waiting place a dewar of liquid nitrogen on the tube between valve # 4 and 5 (figure 8). Fill the dewar to the top and adjust the height of the dewar with the retractable stand (jack lab). The liquid should be about an inch from the top of the tube.

Fig. 8 Placement of liquid nitrogen. Numbers indicate valve number.

10. After you have trapped your water open valves # 4 and 6 to allow the CO_2 to freeze in the presence of the liquid nitrogen for 2 minutes. Non-condensable gasses will not freeze. Watch the digital pressure monitor labeled "meas. vol." and record any non-zero readings on the card and in the computer (i.e. Non-C 8 torr). You should see a white cloudy ring of frozen CO_2 in the tube.
11. Evacuate the non-condensable gasses by opening valve # 2. Wait until the vacuum reaches baseline. IT WILL TAKE WHILE.
12. Close valve # 4 and then remove the dewar to allow the frozen CO_2 to thaw. You can use the heat gun or your hand to accelerate the process (but DO NOT GET THE TUBE TOO HOT).
13. Move the dewar to the tube on the measure volume between valve # 6 and 7. The CO_2 will be transferred to this tube to quote the CO_2 amount. Let it set for 10 seconds.
14. After the CO_2 is once again completely frozen close valve # 6, isolating the section between valves #6 and 7.
15. Allow the CO_2 to thaw. You can use the heat gun or your hand to speed things up.
16. Watch the digital pressure monitor labeled "meas. vol." and when the numbers stop going up you know your CO_2 is completely thawed. Write the amount of CO_2 (the digital reading from "meas. vol.") on the index-card (figure 6 - emphasized in green). Also write the multiplier value: (1) for 1 aliquot sample.

If you are preparing 4 aliquot samples, you will have to isolate CO_2 gas between valves # 5 and 7, instead of valves #6 and 7, as was explain in the step 14. The combined volume between the valves #5 and 7 is enough to accommodate 4 times 1mgC sample. See section VII of this protocol (Graphitizing a standard - x4 standard) on how to transfer this amount of gas to the next 4 reactors.

17. If there is more than 650 torr of CO_2 **you must evacuate** some of the CO_2 by following the procedure below.
    a. To allow 1/8 of the total CO_2 open valves # 4,5,and 6. To allow 1/4 of the total CO_2 open valve # 6. Before vacuuming out any CO_2 calculate how much CO_2 will be lost to ensure you will be left with enough CO_2 (between 480-550 is best). If you are unsure ask for assistance.
    b. Close valve # 6 and use the backline to vacuum out the CO_2

c. Collect the CO_2 to the measure volume using the liquid nitrogen. Recheck the CO_2 amount and repeat if the CO_2 level is still too high.

18. Move the liquid nitrogen to the reactor that you are going to transfer the CO_2 into. Submerge the magnesium perchlorate tube in the nitrogen. Do not allow liquid nitrogen to touch the ultra torr fittings.
19. Check that both bypass valves are closed then, open valve # 7 and the valve to your reactor.
20. Monitor the pressures of the measure volume and reactor. Wait at least 5 seconds after both numbers are 0 to ensure that complete CO_2 transfer has occurred. Close the reactor valve.
21. Vacuum out by opening valves # 2-7 (some may already be open) and both bypass valves, monitoring the millitorr gauge.
22. Allow CO_2 to thaw. DO NOT USE HEAT GUN. Write the amount of CO_2 displayed on the digital screen labeled "reactor" on the index-card (figure 6). Make sure you are reading the correct reactor each time.
23. Repeat steps 1-22 for each sample.

VI.2. Leaching carbonate samples.

Prior to acidify a carbonate sample to generate CO_2 for graphitization, the user should leach the sample to get rid of secondary carbonates. This procedure is required for anything that has been in the ground, fresh water, etc. Leaching is conducted using HCl as shown in the equation below:

$$\mathbf{CaCO_3 + 2HCl \rightarrow Ca^{+} + 2Cl^{-} + H_2O + CO_2}$$

The user should add 2mL of 0.1N HCl for every 10mg of CaCO3 that you wish to remove or submerge sample in 2ml of 0.01N HCl to extract 10% of the surface of the sample.

1. Weighing the sample into the Vacutainer (vial reactor for carbonates where the user can apply the leaching and later on acidify the sample for CO_2 production).
    a. Place an Aluminum foil wrapped Vacutaineer (Santos et al, 2004) or weigh paper on the scale.

b. Tare the scale and then weigh out 9-10mg of a carbonate sample (for 1mg C graphite). When conducting the cleaning procedure (leaching), you have to calculate an extra amount of material to compensate for material that will be consumed by the HCl. If you are leaching using 2ml of 0.01N HCl, you have to add at least more 10% of total amount.
c. Replace the septum after done with the weight.

2. Turn on the heating block to low on setting 8, monitor until the thermometer is at 80°
3. Submerge sample into 2ml of 0.01N HCl to extract 10% of the surface of the sample (leaching procedure).
4. Place sample into heat block and wait at least 25 minutes.
5. Upon completion rinse twice with MilliQ water, pipette excess water with an ultra-fine disposable pipette and dry on the heating block. DRY UNDER AN ALUMINIUN FOIL "TENT" TO SPEED UP THE PROCESS.

VI.3. Acidified Carbonate Samples:

1. Go to the graphitization line and check that valves # 2-7 (ALL VALVES) are closed.
2. Remove bellows and replace with a needle fitting (Fittings are found in the “Carbonate sample prep” drawer on the island containing line 1-12).
3. To evacuate each Vacutainer + your sample, place one drop of MQ water on the septum and press the needle through the water and septum (figure 9)

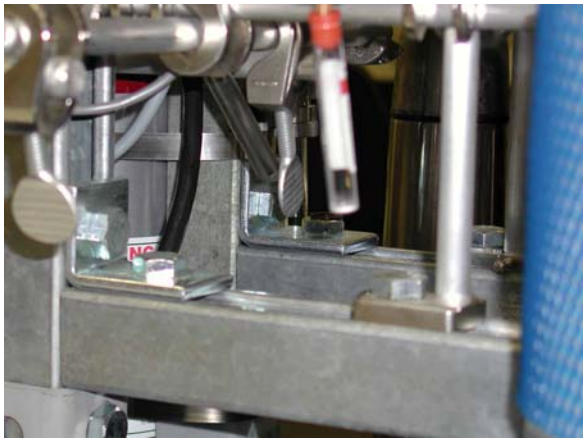

Fig. 9 Vacutainer on graphitization line to make a vacuum in the tube

4. Monitor the vacuum by looking at the pressure gauge. A good vacuum should be about 0-5 millitorr. Once the gauge reaches baseline close valve # 2.

5. To remove the Vacutainer tube and avoid the drop of water on septum be sucked by the vacuum pump, carefully pull the Vacutainer half way down, blow out the water with the Dustoff and take away the tube. Repeat for all tubes.

Be aware that the needles rapidly become blunt and get increasingly harder to push the needle through the Vacutainer septa. After you have used a needle 6 times, replace it. Discard the old needle into the glass waste.

6. Bring tubes to the hood and gather the syringe, needles, a beaker on the tray (in drawer in labeled "Carbonate Sample Prep") and phosphoric acid.
7. Place a big kimwipe on the tray, which will be your work surface.
8. Attach the needle to the syringe.
9. Wearing gloves fill beaker with about 0.8mL of phosphoric acid per sample that needs to be acidified.
10. Remove the plunger from the syringe and fill the syringe to 5mL with phosphoric acid.
11. Replace the plunger and expel any air bubbles (figure 10).

Fig. 10 Syringe turned upside-down and plunger pushed in to expel air bubble.

Fig. 11 Needle piercing through septum, then plunger pushed to expel acid

12. Take your sample tube and puncture the needle through the septum (figure 11). Put a little drop of acid onto the septum and push the needle in through it – this will help prevent leaks.
13. Expel 0.8mL of acid into the tube. Wipe off the drop of acid from the top of the septum. Place the tube in the heating block (figure 12) for 20-40 min. or until the liquid is clear.

Fig. 12 Sample containing Vacutainers on the heating block

14. Repeat until all samples are done. Replace the needle and refill the syringe every 6 samples.
15. Discard needle in glass waste, discard excess acid in acid/base waste, and wash beaker and syringe with ample amounts of hot water and then rinse with MQ water. Dry with the hot air gun and put back in the drawer.
16. Make sure all valves are closed.
17. When acidification is complete remove the bellows from the line and replaces it with the needle adapter.
18. Insert the needle on the adapter halfway through the septum being **very careful** not to fully penetrate the septum. If that happen, your sample was mix with air in the needle and it is not suitable for ^{14}C measurements.
19. Open valve # 2 and allow the atmospheric gases in the needle to evaluate.
20. When the gauge reactor reaches 0-5 millitorr close valve # 2, then push the needle completely through the septum. Continue procedure by going to section VI.1 (Using combusted organic samples - steps 8 to 22).

VII. Graphitizing a standard (4x standard) sample.

This section illustrates a variation of the method (section VI.1. Using combusted organic samples) to allow share organic combusted samples into 4 aliquots, e.g. 4 graphite samples. It is being applied at UCI KCCAMS prep lab to prepare the standards (OX-I's, OX-II's, ANU's, etc).

See also the UCI KCCAMS combustion protocol to learn how to accommodate 4mg C samples into the combustion tube.

1. If you are preparing 4 aliquot samples, you will have to isolate CO_2 gas between valves #5 and 7, instead of valves #6 and 7, as was explain in the step 14 (section VI.1. Using combusted organic samples). The combined volume between the valves #5 and 7 is enough to accommodate 4 times 1mgC sample. Write the amount of CO_2 (the digital pressure reading from "meas. vol.") on the index-card (figure 6 - emphasized in green). Also write the multiplier value: (4) for 4 aliquot sample.
2. Standards must be split into 4 different reactors. This step requires to share the amount of gas that is trapped between valves #5 and 7.

To ensure almost equal amounts of CO_2 in each reactor, Dos Santos systematic evaluated splitting processes and chooses this splitting order:

1 2 3 4 4 3 3 2 2 4.

a. This numbering order means that you have to transfer the amount of gas trapped between valves #6 and 7 to the first reactor available.
b. Again share the gas between valves #5 and 7, opening valve #6 (wait 30 secs).
c. Close valve #6 and transfer the CO_2 trapped between valves #6 and 7 to the second reactor available.
d. Repeat (b) and transfer the CO_2 trapped between valves #6 and 7 to the third reactor available.
e. Repeat (b) and transfer the CO_2 trapped between valves #6 and 7 to the fourth reactor available and so on.
f. The final amount of gas (when you are on the last number in the splitting order) trapped between valves #5 and 7 can be completely transferred to the fourth reactor.

By using this transferring method none of the CO_2 gas will be wasted.

3. During CO_2 transfer to reactor, move the liquid nitrogen to the reactor that you are going to transfer the CO_2 into. Submerge the magnesium perchlorate tube (1/2 tube) in the liquid nitrogen.
4. Open valve # 7 and the individual valve of the particular reactor.

5. Monitor the pressures of the measure volume and reactor. Check that both bypass valves are closed then; wait at least 5 seconds after both numbers are 0 to ensure that complete CO_2 transfer has occurred. Close the reactor valve.
6. After completed the splitting order, vacuum out the line by opening valves # 2-7 (some may already be open) and both bypass valves, monitoring the millitorr gauge.
7. Allow CO_2 to thaw on each individual reactor. DO NOT USE HEAT GUN ON THE MAGNESIUM PERCHLORATE. Write the amount of CO_2 (displayed on the digital screen labeled "reactor") on the index-card (figure 6 - CO2 Torr- emphasized in green). Make sure you are reading the correct reactor.
8. Repeat the entire section VII for each standard (4X standards).

VIII. Hydrogen addition:

Twice the amount of hydrogen (compared to CO_2) must be added to each reactor (Vogel et al, 1984, Santos et al, 2004). Since each reactor has a varying amount of CO_2 each reactor must be dealt with separately. At this point the valves to the reactor tubes should remain closed until the H_2 is ready to be added.

1. Before adding H_2 close valves # 3 and 4 then take off the water trap. Allow frozen water to sublimate.
2. Flush the entire line three times with H_2 (figure 4) to allow flow of H_2 into the line and then quickly open and closes valve # 1 to flush the line with H_2. Allow the gauge to reach baseline
3. Calculate the amount of H_2 needed for each reactor. Before you add H_2 to a reactor, put liquid nitrogen on the perchlorate tube. BE CAREFUL DO NOT SUBMERGE THE ENTIRE TUBE INTO THE LIQUID NITROGEN. Watch the reactor pressure (make sure the reactor dial is measuring the correct reactor), when it reaches 0 (your CO_2 is now frozen) you may proceed.
4. Close valve # 1 and open the reactor valve.
5. Now you will add the appropriate amount of H_2 to each reactor. Begin with the reactors that require the least amount of H2 addition.
6. Open the H_2 valves # 9 and 11 (figure 4) to allow H_2 to flow into the reaction line. Regulate the amount of H_2 by using H_2 valve #2. Monitor the amount of H_2 added by looking at the pressure readings.

7. If you have added the appropriate amount +/- 5 torr you may proceed to step 8. If, however, you have exceeded your target then you must vent some H_2 and try again.
8. Close the reactor valve, move the reactor dial to the next reactor and repeat steps 3-7 until all reactors have H_2.
9. When all the reactors have H_2 allow the gases to thaw. Write the TOTAL amount of gas (displayed on the digital screen labeled "reactor") on the index-card (figure 6 - CO2+H2 Total- emphasized in green). Make sure you are reading the correct reactor.
10. Put heaters onto each graphite-reactor tube. CHECK THAT THE HEATERS DO NOT COME TOO CLOSE TO THE ULTRA-TORR FITTING. THERE SHOULD BE 1/8 INCH – 1/4 INCH CLEARANCE (figure 13). If the heater is too close ask for assistance.

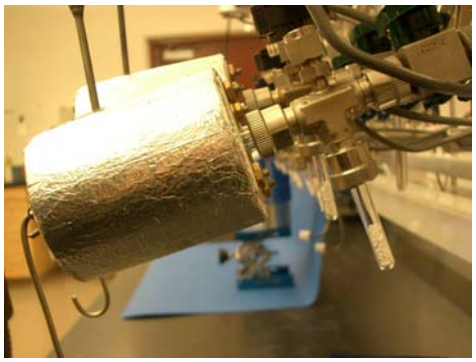

Fig. 13 Showing proper distance of the heater from the ultra torr fitting.

11. Set the timer and temperature controls (figure 5) to set the temperature make sure the controls are on (the current temperature should light up) **FOR GRAPHITIZATION THE TEMP. MUST BE 550°C.** Check the set temperature by pressing the "sel" button next to each pressure monitor. This will display the set temperature.
    a. If the set temp is 550°C proceed to part c.
    b. If the set temp is not 550°C then use the up and down buttons to raise or lower the set temp and then reselect by pressing "sel".
    c. Double check that the temp set is at 550°C by pressing "sel" twice.
12. To set the timer, turn the dial to 6 hours. And then start the timer by flipping both switches off and then on again.
13. After you have begun graphitization use the following procedure to clean the line:
    a. Pump all excess gas from the line (except reactors).
    b. Empty the bellows of broken glass.
    c. Flush the entire line with more H_2 three times.

d. Evacuate the entire line.

14. Take index-cards to the computer and record all numbers into the **Sample Master list** in the sample list folder of the computer. Place index-cards back on the line.

IX. Post-Graphitization Procedures (collect your graphite)

1. Take heaters off of the reactors and hang them on the bar. Ensure the leads all hang the same way to avoid tangles (figure 14).

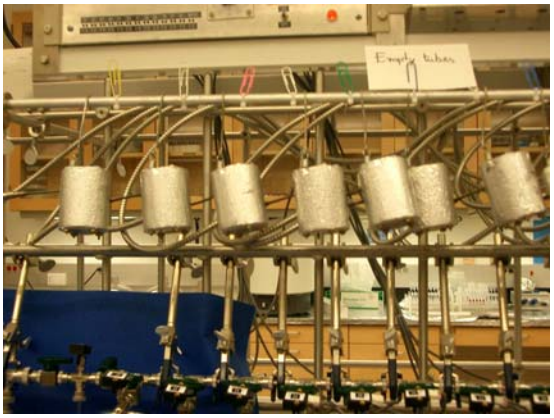

Fig. 14 Example of how heaters should be put away

2. Obtain caps for your sample tubes from the drawer labeled "graphitization tools".
3. Record the residual pressure for each sample on the index cards (figure 6).
4. Evacuate the residual H_2 from each reactor.
5. Flush the entire line with fresh H_2 (3 times).
6. Close valve # 1 and fill the entire line with H_2 until the pressure reaches approximately 1 atm (maximum). DO NOT EXCEED 1 ATM OR THE PYREX TUBE MAY FLY OFF THE REACTORS, CAUSING INJURY OR SAMPLE LOSS.
7. One reactor at a time, take off the graphite sample, then replace with a tube containing new Fe catalyst. Have baked pyrex tubes with new catalyst ready to put into the reactors immediately after you take each sample off. The magnesium perchlorate is very hydroscopic and upon contact with the air will trap water (Santos et al, 2004).
8. Apply a cap to the graphite sample tube and label with a sharpie the UCIG # assigned to the sample, put on a new tube with Fe catalyst.
9. Repeat this for all samples and then Pre-condition the line for the next user.
10. When the line is not in use leave it under vacuum (including the bypass line).

11. Take index -cards to the computer and record the residual values into the **Sample Master list.**
12. Place your graphite samples and attached index-cards in a box with your name on it in the dry dessicator for further AMS measurements.

X. Changing the magnesium perchlorate

1. Get 12 baked pyrex tubes from the wet cabinet.
2. Obtain a large kimwipe, the magnesium perchlorate, shifter and magnesium perchlorate spatula (in drawer labeled magnesium perchlorate supplies on island containing line 21-30).
3. Remove the black tape on the container lid. Using gloves shift a small amount of the magnesium perchlorate from the jar through the large mesh shifter and onto the kimwipe. Close jar immediately when done and place black tape back onto jar (if the tape appears to be old replace with new tape from the magnesium perchlorate drawer).
4. Using the spatula or the pyrex tube, gather the magnesium perchlorate into the pyrex tube until 1/4 full. It is important to avoid collecting any powdery magnesium pechlorate.
5. Replace tubes onto the line and throw away the old tubes into the glass waste.

XI. How to make a slush

1. If there is no dry ice in the lab, go to the **School of Physical Science supply store** and buy 2 pounds of dry ice per line
2. Use the ice shaver to grind the ice (figure 15)
    a. Lift the top of the shaver and add dry ice to the ice shaver (use leathern gloves). Make sure there is a plastic container below the machine to catch the shaved ice.
    b. Replace lid and switch the motor "on". Press down the lid lever to push dry ice through the machine and into the plastic receptacle, then turn off the motor.
3. Take a dewar containing ethanol (1/3 – 1/2 full) and place into a tray.
4. Slowly add shaved dry ice to the ethanol being careful not to add too much, at first, which will result in a loss of ethanol by "boiling" out of the dewar.
5. Add dry ice until the mixture becomes the consistency of a stiff slushy. Stir with the spatula provided.

6. Place slush on the water trap located between valve # 3 and 4 (figure 1).

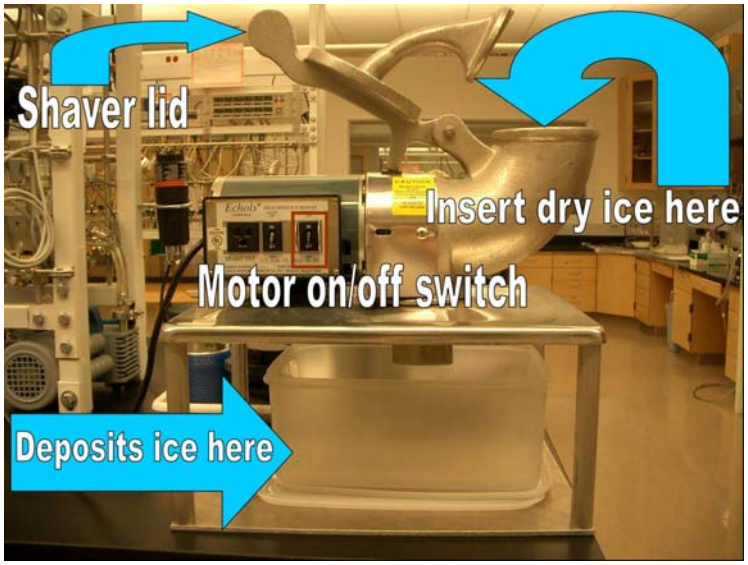

Fig. 15 Ice shaver with labeled parts

XII. References

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