

UCI AMS Facility

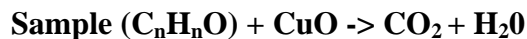
Combustion Protocol
Dec 26, 2011

SUMMARY

1. Assemble the samples and index cards
2. Clean the combustion area and tools
3. Clean the combustion line fittings and grease the O rings
4. Cover a quartz tube with Al foil, and tare the balance.
5. Add sample and weigh
6. Tare, add 60 mg of CuO (180 mg for x4 standards), reweigh, add Ag wire
7. (For powdery samples) add a plug of glass wool.
8. Label the tube with the high temperature marker.
9. Valve off a combustion line port, insert the tube into the fitting and evacuate.
10. When you have a full set of samples on the line, torch off the tubes
11. Turn off the torch, the oxygen, and the gas when you're done.
12. Place the tubes in order in the furnace in ceramic tubes, check the timer and temperature, cycle the power to start.
13. Input the combustion information to the Irvine6 spreadsheet
14. Hang the cards on the furnace door
15. After combustion, relabel the tubes with a Sharpie

I. Introduction

Carbon dioxide (CO₂) is obtained from organic samples is obtained by combusting them at high temperature. The following summarizes the combustion reaction:



Samples are placed in quartz tubes (pre-baked at 900°C for 3 hours) along with CuO and Ag wire. The CuO is the oxygen source for the reaction. The Ag wire acts as a getter to remove sulfur as well as chlorine from the raw sample material. The quartz tubes with the samples are evacuated and sealed, and the samples are then combusted at 900°C for at 3 hours.

II. Pre-Combustion Procedure

Balance setup .To reduce static problems in the Sartorius balance, moisten the sponge that is kept inside the balance enclosure; and if conditions are very dry, use the sponge to wipe down the inside surfaces of the plastic panels of the enclosure. Note that the new Kern balance self-calibrates when first turned on, and this takes several minutes

Clean the combustion line fittings and grease the O-rings. Sealing tubes is difficult if the O-rings in the combustion line fittings are dry. O-ring grease and Q-tips are kept in the pencil drawer below the combustion line. Check the baseline pressure, then valve off each port in turn, remove and dismantle the 1/4" fitting. Remove the O-ring, clean out the fitting with a Q-tip, and blow off with an air duster to remove any residual lint.. Lightly grease the O-ring, reassemble and replace the fitting and the blankoff tube, and slowly open the valve. The line should return to baseline pressure within a few minutes - check..

Sample cards. 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 Irvine6, 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 reference. **Ask a lab assistant if you need help.**

Work area. Put a clean layer of Al foil on the counter top. From the combustion toolbox, (located in the combustion tool drawer on the top left) obtain Ag wire and CuO vials, as well as spatulas, tweezers and combustion tube cover (made of Al foil.) Make sure the cover is long enough to completely cover the tubes you'll be using: if it's too short, make another. Clean all tools with methanol and Kimwipes and blow them off with an air duster. **Remember to wear gloves! Also, keep a designated spatula for CuO and tweezers for Ag in order to avoid cross-contamination.**

Standards. Working vials of primary standards (NBS OX-I and OX-II oxalic acid), plus some other commonly used standards and blanks (ANU sucrose, USGS coal, etc) are in the top drawer to the right of the workstation. These have their own spatulas that should always be cleaned before and after use, and should never be used with other samples. Vials of other (secondary) standards and blanks, are located in the bottom left hand drawer of the workstation and in the overhead cabinet (bone standards).

II Weighing samples into quartz tubes

Unless you know exactly what you're doing and where your hands will be at all times, wear gloves while loading tubes.

1. Most samples are combusted in 6" long 6mm tubes. However, if too much material is combusted the tube may explode due to excess pressure, so if you are unsure of the carbon content of the sample, use an 8" long tube for greater volume. Routinely used primary standards (OX1, OX2, etc) are often combusted with sufficient material in one tube to make four aliquots of graphite, and 8" tubes are used
2. Place a tube in the Al foil cover (to reduce static), and tare the balance.
3. Add the appropriate amount of sample and record the weight on the index card. The amount required will depend on the carbon content of the particular material. The table below gives %C for some common materials, and the corresponding weights of pretreated material required for a single 0.75 mg C sample. Other materials can vary widely: sediments can range from tens of percent C to almost zero.

Material	%C	Amount required (mg) for .75mgC
Wood, cellulose, bone	40	1.7
Oxalic acid	19	4 (use 16mg for x4)
Degraded wood	40-50	1.5
Coal	60-90	1.2

Notes: a) It is sometimes easier to use weighing paper instead of weighing the sample directly into the tube, depending on sample type, static conditions, etc. The sample must then be transferred without loss from the weighing paper to the tube, which is then wrapped in Al foil and placed on the balance.

b) Static samples can be very difficult to get down into the tube. Holding the tube upright and repeatedly dropping it 6" on to the bench sometimes helps, or you can try using the back end of a spatula to push the sample down. There are anti-static guns in the drawers to the left of the workstation, that sometimes help.

4. Re-tare the balance.
5. Add 60-70 mg of CuO for a single sample (this is several times stoichiometric to allow for surprises) and record the weight. The amount is not critical: try for 60-70 but 80 or even 90 mg is not a disaster. Use 180mg for a large (4x) standard.
6. Add one piece of silver wire.
7. Label the tube 1-2" above the bottom with the UCIG number, using the high temperature marker pen. Shake the marker pen to ensure the ink is flowing, and you may even have to press the tip on to a scrap piece of Al foil. Go over the numbers again if necessary to ensure they're good and black
8. Evacuate the tube on the combustion line: close the valve, remove the blankoff tube, insert the combustion tube and slowly open the valve.
9. If the sample is a very fine powder, a small plug of baked glass wool should be placed into the opening of the combustion tube prior to evacuating, and the valve should be opened very slowly. This will keep your powdery sample from being sucked up into the fittings. Also, evacuate each tube individually: Closing off the previously evacuated tubes will ensure that the glass wool plugs do not get pushed down the tubes towards the samples by back pressure in the line when a new tube is pumped. Once the tubes have been evacuated individually, all of the valves can be opened for further pumping.

III. Sealing quartz tubes

When all tubes have been pumped and the line vacuum is has reached baseline, the tubes can be sealed off using the gas torch located by the combustion line. Note that OX1 and OX2 (and some samples) contain loosely bound water that must be pumped away, and this can take up to an hour.

1. **Put on welding goggles.** This is important: the UV from white-hot quartz can cause retinal burns. **Do NOT wear gloves while sealing tubes:** if you inadvertently touch hot quartz, a clean burn is preferable to one filled with molten rubber..
2. Turn on the gas supply valve along with the oxygen bottle and supply valves. (The regulator should be at approx 10psi – don't touch).
3. Turn the red knob (gas feed) on the torch in the direction opposite to the arrow just enough that you hear gas coming out. (The arrows indicate the direction to close the valve).
4. Point the torch away from you in a safe direction (think about what the flame could hit) and use the striker to light the torch.
5. Use the green knob to add oxygen. Adjust until you get a bright blue triangle at the base of the flame. The more oxygen, the smaller and hotter the triangle will become.
6. The red and green knobs are then adjusted to get an appropriately large and hot flame. The bright blue part should make up approximately 1/2" of the overall 8" flame.



Sealing the combustion tube. Continue twisting the tube until the two halves separate.

7. Sealing the tubes is most easily done by holding the flame steady and rotating the combustion tube. Pick up the torch out of the holder and hold it so the blue triangle almost touches the quartz, at a point about 1" below the fitting (and below any glass wool plug). The quartz will heat up white hot (it will appear bright green through the

goggles). Do not allow it to overheat in one spot: rotate the tube slowly until a uniform white-hot ring forms. If the ring doesn't form the flame is not hot enough. Adjust the knobs to increase gas and oxygen.

8. At this point the quartz should be soft. Gently pull the lower part of the tube down, while continuing to twist and keeping the torch midway between the two halves. When they are about 1" apart, the neck between them will thin out and eventually break.
9. To prevent breakage of the tip of the tube and to seal off possible "wormhole" leaks, melt the tip back into a smooth blob, then place the tube on the tile surface under the combustion line.

Caution: Hot quartz looks like cold quartz: the quartz may be very hot even though it's no longer glowing. Be careful what you touch.

10. Turn off the oxygen valve on the torch followed by the gas valve (turn the knobs in the direction of the arrows, and always turn off the oxygen first).
11. Turn off the oxygen supply valves (2) and the bottle.
12. Turn off the gas supply valve on the wall.

IV. Combustion:

Once all tubes have been sealed they are baked in the furnace at 900°C for 3 hours.

1. Place the tubes inside the ceramic tubes in the furnace (If one combustion tube blows up these protect the others). Leave about 1/2" protruding, to make it easier to pull the combustion tubes out when they're done. As a precaution in case the high temperature ink fades, keep the combustion tubes in strict numerical order (left to right, top to bottom),
2. Check that the temperature setting is correct (900°C). If not, set it by manipulating the arrow keys and then pushing the set button.
3. Check that the oven timer is set to 3 hours, and start it by cycling the power off and on.
4. Enter the combustion info (date, sample wt, CuO wt) into the sample master list spreadsheet on Irvine6.

5. Sort the cards into numerical order and hang them from the clip on the oven door.
6. Once combustion has been completed, retrace the UCIG number on the tube with a Sharpie marker to increase legibility
7. Place combusted tubes and index cards in coffee mugs in the appropriate sample trays. Combusted standards not associated with any particular sample set are stored in the cabinet above the combustion area.