X ray diffraction

9th floor at ETC building (9.142)
In order to use the machine it is necessary to make an appointment the same day including name, time, and for a period of 2 hours.
There is two machines, we must use the grayer one (the one at the right). It works better.

Preparation of the sample for X-ray diffraction.

We take one of the pellets or we can use powder sample. In this case $Ca_3Y_1Cu_5O_{10}$ is our sample that has been, bake already 3 times

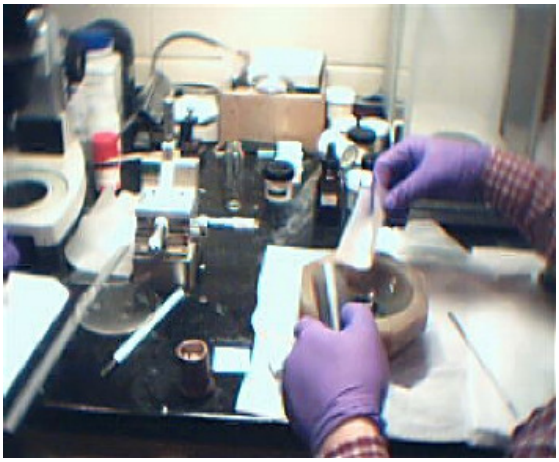



If we have a pellet, it needs to be ground. To do this without losses, we must cover the mortar with aluminum foil. Make a hole in the center and put the pestle through the hole. Then grind the pellet (do this dry) (Make sure to collect all the sample that has been spread out of the sides of the mortar and scrap off the sample left on the pestle).



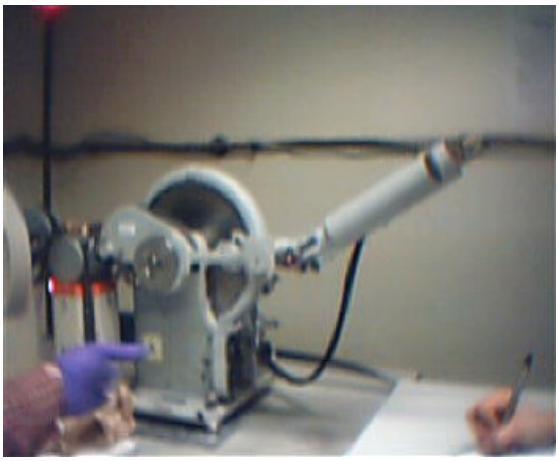
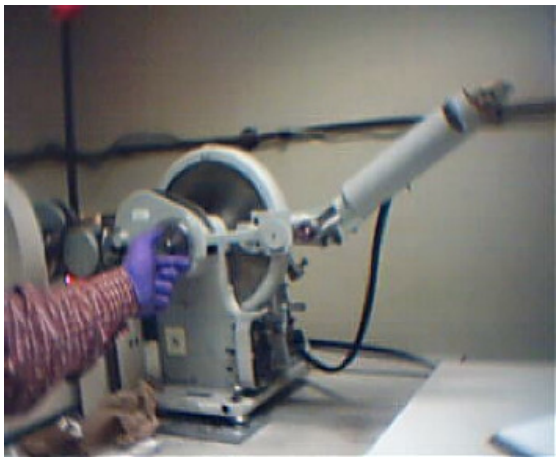



We need the pellet to become powder for the X-ray diffraction analysis. Put the powder in a clean piece of the glass slide that we cut before. Then put 1 drop of Amyl Acetate and then stir it with the spatula to make an even sheet. We must use Amyl Acetate (Take from Dr. Goodenough lab) (Break a glass slide in two pieces. We will put the sample in this glass.) Professor Goodenough will retire this year his substitute will be Dr Jianshi Zhou at ETC 9.140.

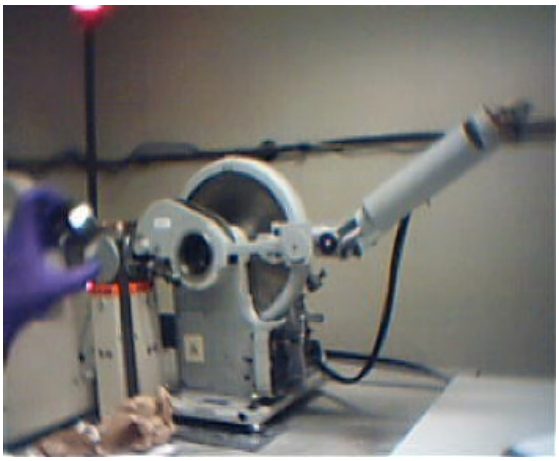
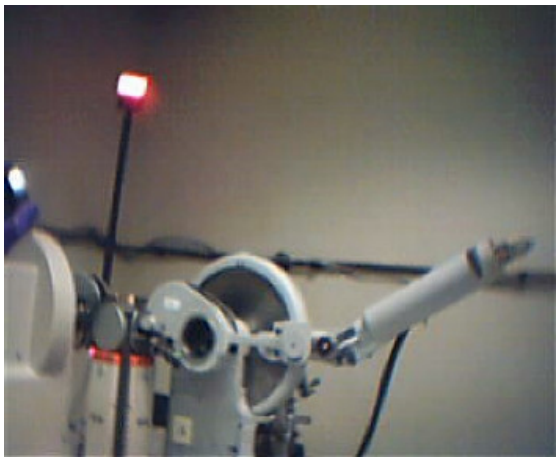



Then label the glass slide with a sharpee.

Running the X-Ray machine (ETC 9th floor)

To introduce the sample into the x-ray machine, first rotate the small knob counter clockwise and later the large knob. The hole is small. There is a holder inside the hole. In this holder we will put the sample to be analyzed. After set the sample in the holder we must replace the cover tight.

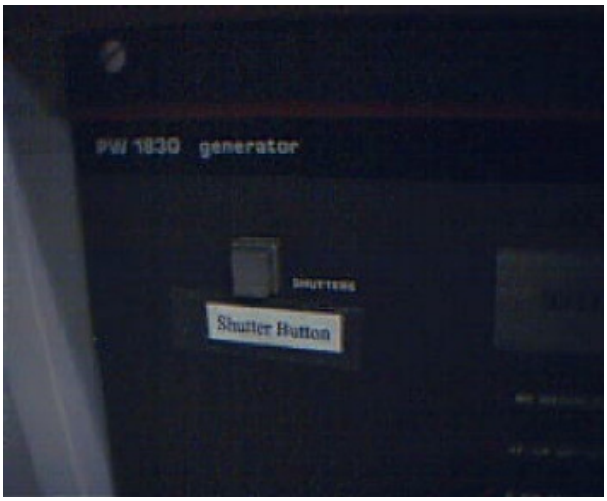
Control Panel

The control panel is situated under the x-ray machine.

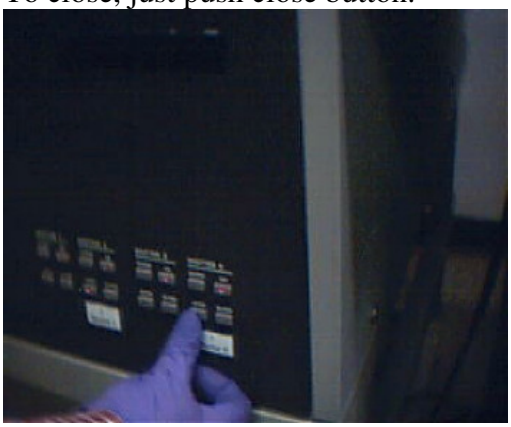

Shutter 4-

Carefully: X rays are always being emitted.

Hold shutter button and press open button under shuttler



To close, just push close button.



Software

In order to start taking data we must follow the directions on the screen of the monitor located at the right of the X-ray machine.
Example
First of all open windows internet explorer. There you must register as Cardenas Rosa or Manzanera, Isaac as Select User

Select User: Supervisor: Markert, John
Inst. Used: Phillips # A
Start time: hh mm
Start Date: mm/dd/yyyy
Click to submit.

Once logged in, we must run a program called Data Scan on desktop.
Press Scan.

File ID:
Set Up: 27 market lab (starting and ending angle, step size, time of data acquisition)
Scan ID: make notes of sample

Press **start scan**- Takes about 1hr (time indicated in the screen)
The file is saved after 1 hr and sent to another computer.

Press **close** from control panel.
Open the cover (turn small knobs counter clock wise) and remove the sample.
Leave it open

To get the data go to room 9.120
In that room there is a computer. Go to **Xdata**
Sort by date modified (our file should be the last text file)
Select our file and the double click to **Look at It**

The document record the data in rows.
Columns mean nothing, start from left and move to the nest slot
Start at top left 10.00 deg increase 10.05 degrees for the next number.

#10.00	*ϖ 10.05º*	*æ 10.10 º*
#$_1$10.15	*ϖ$_1$10.20º*	*æ$_1$10.25 º*
#$_2$	*ϖ$_2$*	*æ$_2$*

X-Ray diffraction analysis

In the computer we will see a bunch of peaks in the program.
To see this file we just have to double click on the name of file in Xdata
Ask Dr Swinnea about any question that we can have in order to do X-ray diffraction. He is nice to ask.

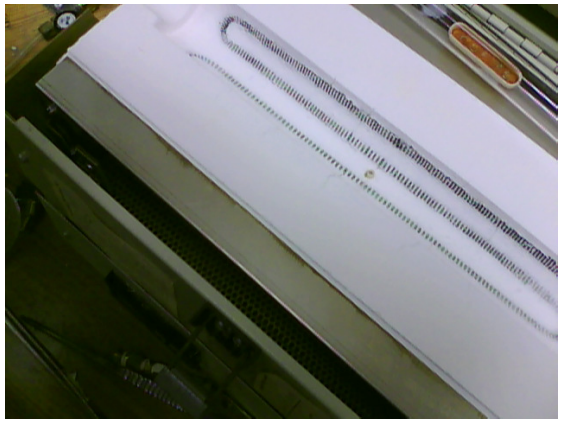
In this experiment we are looking at $Ca_{2.5}Y_{1.5}Cu_5O_{10}$
Start by pushing **Chemistry Recall**
We will see a periodic table of the chemical elements. If we press one it turns green. It means AND if we press a second time it turns blue it means OR
We selected Ca, Y, Cu, O in blue and we pressed **ok**, getting yellow peaks.

Go down the list of the different elements until it makes same structure as Calcium Copper Yttrium Oxide. So for one case since it matches pretty well is $Ca_{2.5}$

We can then find the lattice parameters a, b, and c, in order to do that, ask to Swinnea (9.114) it will help us to draw the lattice.

Low Pressure Oven Oxygen
Tube Furnace

Put the sample (pellet shape) in an alumina crucible. Use metal rod to place the crucible in the center of the gas tube.



The thermometer resistance is at the center of the white furnace. Sample should be exactly on top of the thermometer.

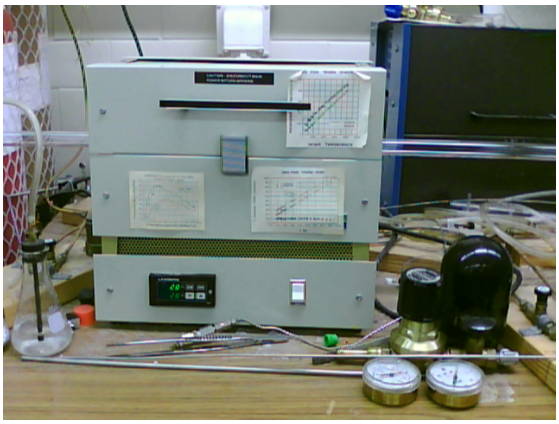
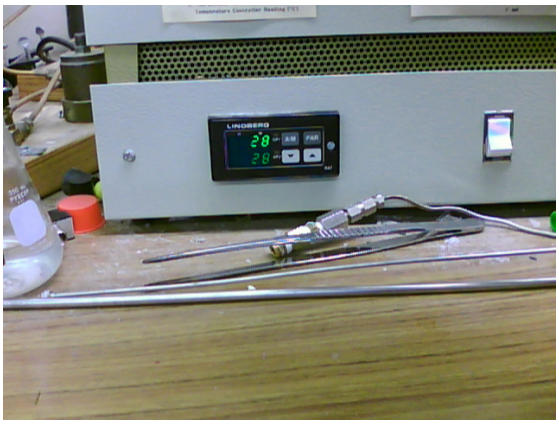
The position of the tube is such that the air will flow from the clean side of the tube to the black side of the tube. Consecutively close the furnace and lock it.
Use oxygen from cylinder this is low pressure oxygen 1Atm
Place the Rubber Stopper in the flow end of the tube and the other end should be connected to a Stopper that has a tube that flows into water to monitor the flow (looking at the bubbles)

Turning on the oxygen flow by opening the silver valve all the way

The black valve determines the pressure we want it to go to psi (But this valve is not the most important. The most important valve is small oxygen valve connected to the glass tube


We want a flux that will produce 1 bubble every 13 seconds (lowest pressure possible)

The oven has a controller:

Instruction manual model 55035

It has 3 points same system as the high pressure oven.
R1=ramp rate
L1=target temp
D1=dwelling time/duration time

This oven has only 2 phases LC=2

Press **par**

Idle, run, hold can be selected by using Δ *or* ∇

s.p. = after program ends, the temperature at which this program goes to 27
ture = ignore

LC=1
R1=10.00 deg/min
L1=<1000

D1=1440 min
R2=0.35 deg/min
L2=450
These is the same as the high Pressure Oxygen Furnace
To test just set r1=1 deg/min
L1=35

To begin the program, press par until you see idle then press Δ to run
Lc=1 is a loop

To turn off: press par and set from run to hold or idle

Tube furnace with super doper gas mixer

The cylinders are argon, mixture of argon and oxygen 0.1999%, hydrogen, and oxygen.
Normally we want 1 atm air 20% oxygen
For less % of oxygen we must use more argon
Example:
To get a 10% oxygen we use a mixture of 9 parts argon and 1 part oxygen.
We connect the metal cylinder from the gasses to the end of the glass tubing. The connector is fragile, be carefully not to tighten.

Multi gas controller

To turn on, turn the key to the position On
The screen is broken.
The manual of the multi gas controller indicates that we should be seeing
Push 1 goes to p 11 menu.
Ch1

Units SCCM (Standard Cubic Centimeter/Min)
Set everything first before to turn on the flow on.
To turn Ch1 on press