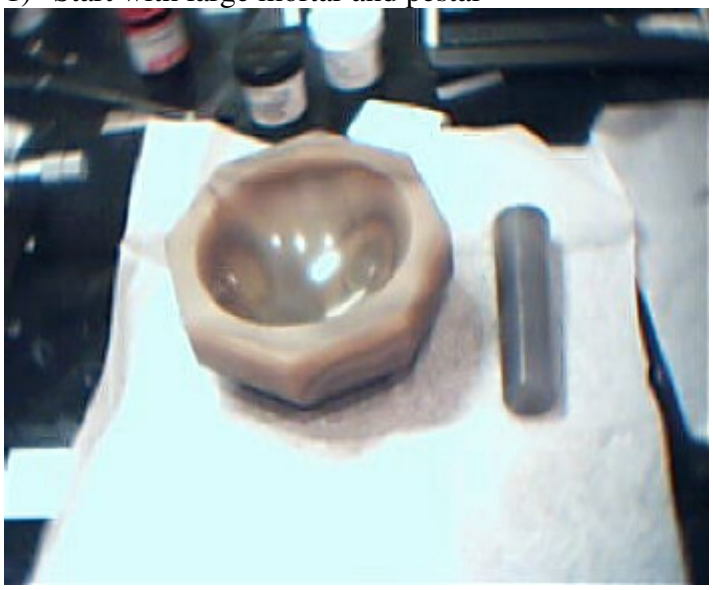
# How to make crystal samples

1) Start with large mortar and pestal



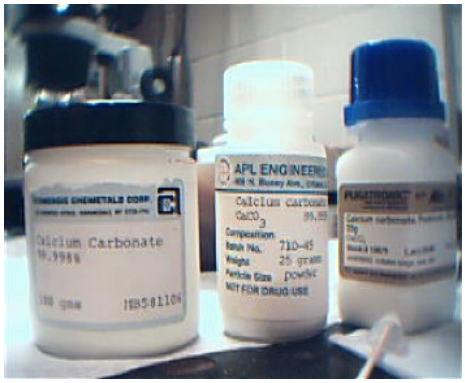
2) Clean the mortar with acetone

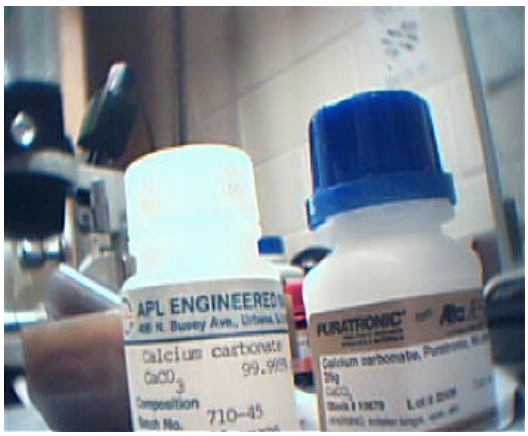
Our sample is $x = 0$ $Ca_{2+x}La_{2-x}Cu_5O_{10}$

We want to make 2g of it.

Located in the cabinet of the samples room we will use three compounds.

Calcium carbonate (3 different purities) 99.99%, 99.995% and 99.998%

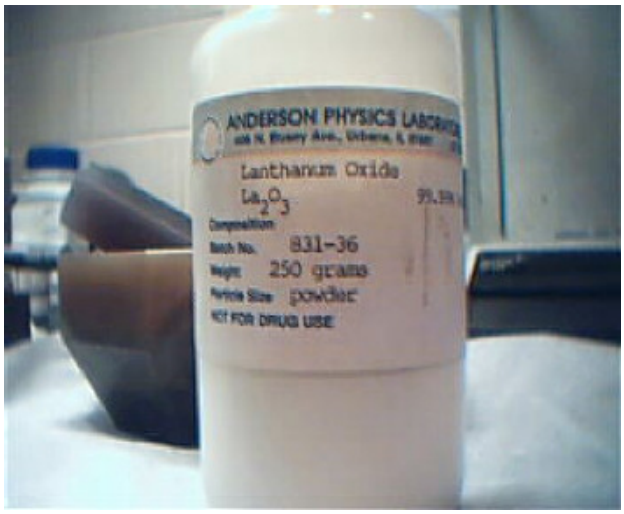




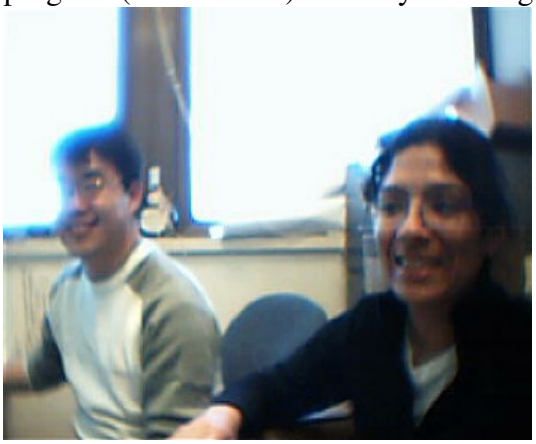
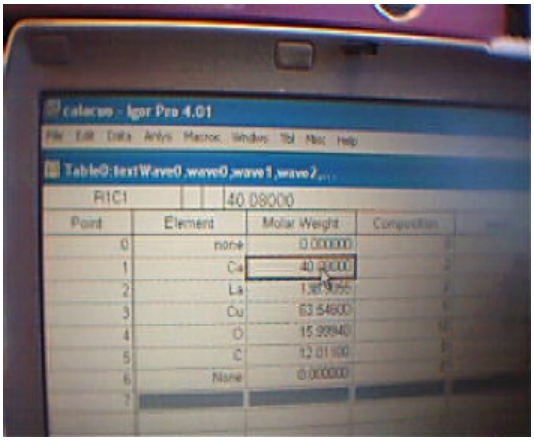
We will start by the least pure.

The other compound is Lanthanum Oxide $La_2O_3$ 99.99%




Finally we will use Copper oxide $CuO$ 99.995%


To calculate the quantities that we need to make this compound we will use a computer program (CALACUO) made by Keesong Park the one runs in IGOR PRO


It is located on the lab homepage under manuals. In sample making manual.
How does it work?
We introuce at the column “req amount” the quantity that we want to produce.
Press enter
Type in the command windows amount()
Press enter
And we get the quantities that we need.

To get the details of the program
Select window
Procedure window.
The results obtained for our sample are

$CaCO_3$ 0.47907g

$La_2O_3$ 0.779732g

$CuO$ 0.951847g

The next thing is to dry the sample in the oven for 12 hours. Because of the reduction of weight due to the evaporation we must include more of 10% of the initial quantities calculated.
Each compound requires a different temperature for it to be dried.

Calcium carbonate $CaCO_3$ $100^0$ (we will use the oven at $135^0$

Lantinum oxide $La_2O_3$ $900^0$ outside the sample room

Copper oxide $CuO$ $450^0$ Little oven outside the sample room next to the blue one.

In order to select the $450^0$ hold the black button and turn the knob to set the temperature


Set it to room temperature $30^0$ when finished

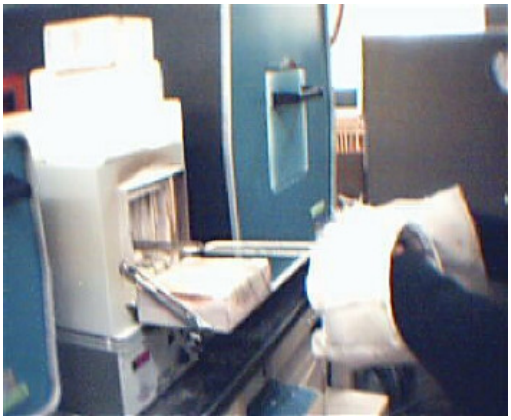
In order to set the sample into the oven, we need crucibles for drying
Drying crucibles are in a drawer in the sample room


Use the particular crucible that is labeled for a particular compound.
For $La_2O_3$ $900^0$ we can use alumina (like white ceramic)
Use a beaker (like glass) to dry the Calcium Carbonate $CaCO_3$ $100^0$
Always use acetone to clean Beakers and Crucibles.

We put the samples in each oven at 3:50 pm
To put in the oven the samples we must use gloves and tongs and wait 12h


To open the $450^0$ oven we have to pull first the handle up



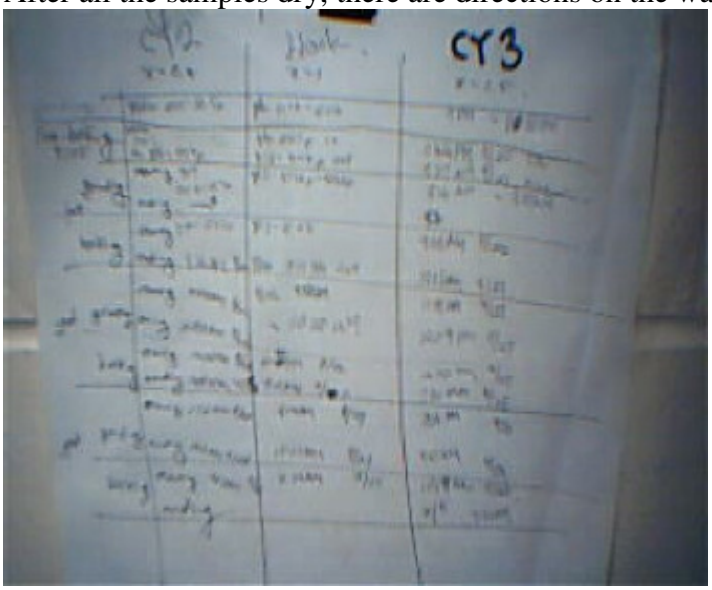


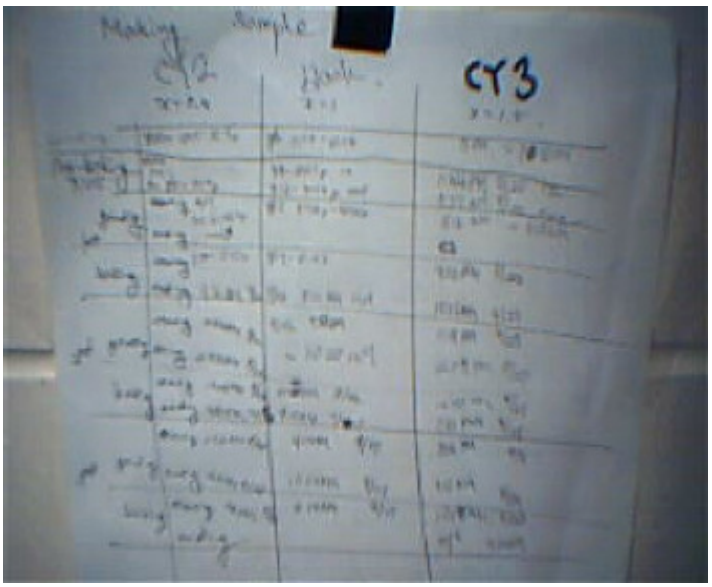
In the cabinet there are some smaller containers of samples closed with a black lid.



Those are samples already dried, so if we dry extra quantity we will add this to the containers and if we don't have enough quantity we can take some from the samples.

After all the samples dry, there are directions on the wall inside of the sample room



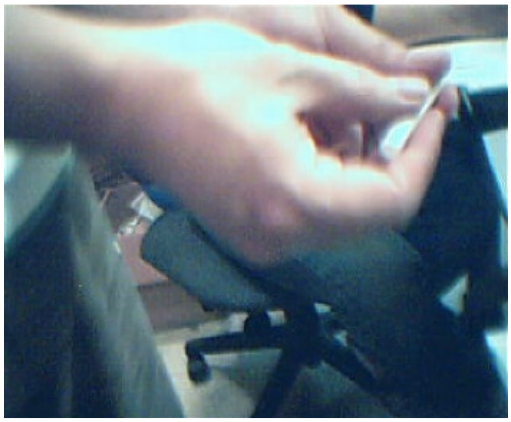
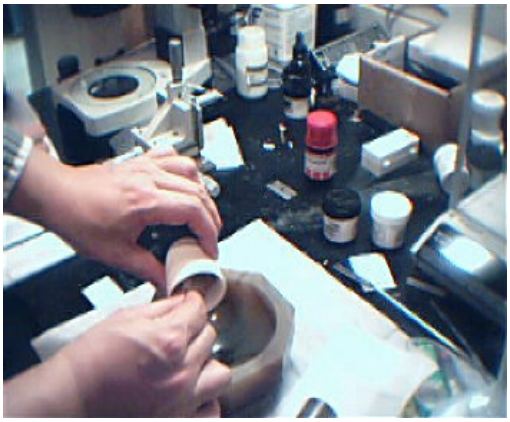
These directions indicate the steps to prepare $x = 0$ $Ca_{2+x}La_{2-x}Cu_5O_{10}$

Once the samples dry, measure exactly the weight for each compound, put all on a large mortar (cleaned with acetone)

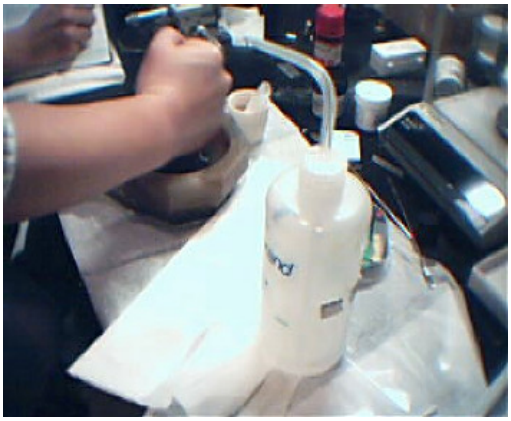
Note the sample impurities 99.99%. 99.99% and 99.995% the total sample impurity is equal to the higher of the impurities.

## Pre-grinding

Grind more than 30 min (Make sure to take out as much of the sample as possible by rolling up a weigh paper and scraping the crucible)
During the grinding put acetone in the mortar and continue grinding (rotations for 30 min) Acetone helps for thorough mixture.
After the 30 min. take the acetone mixture and place in hood to evaporate.






## Pre-bake

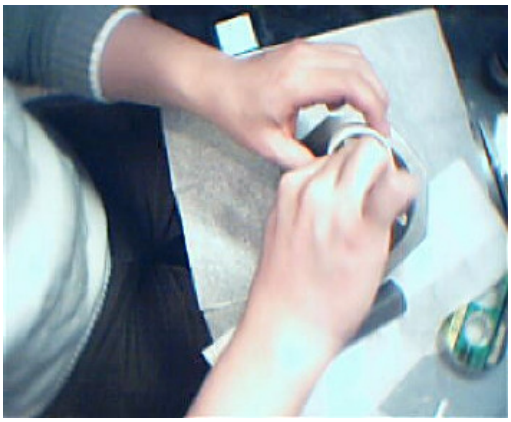
We choose $900^0$ to take the $CO_2$ out of $CO_3$ because we need $Ca_5O_{10}$ and no C. This is called Calcination process.

After the acetone is evaporated we take as the entire compound into a crucible for more than 12 hours


Using a paper we will take as much material as possible.








Pre-baking is the first baking of the compound. After pre-baking

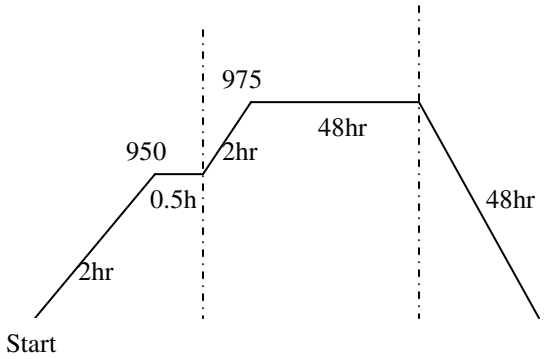
## First grinding

Again take all the quantity as possible from the crucible and grind for 30 minutes with acetone.
Again let the acetone to evaporate and put again all the material in the crucible.

## First Baking

Each compound will have a different procedure. So we try to find the most efficient method in order to produce the compound that we are interested in.
In our case, we don't know what will be the best procedure. Consequently we will start with $950^0$
We will use the oven outside of the sample room.


This is a programmable oven. We are going to use Keesong's thesis fig 31 p 33.
This is a 3 step process




Start: Press ⊃ to go through the menus until you find Pent then press ↓ (= enter)
Pn (= program number) there are 8 saved numbers

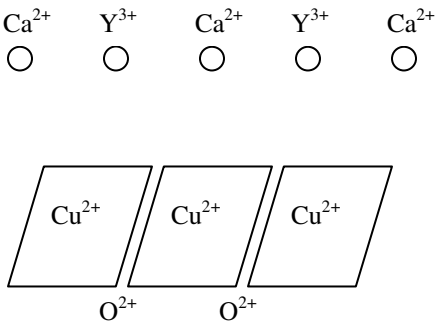
Press ⊃ again and see 1
Press ⊃ again to see n5
Press ⊃ to see 3 (it means that this program has 3 steps as diagram above)
Press ⊃ 2.00 hr
SP = set point = target temperature $950^0$C
ST = set time = 0.3 (0 hours, 30 min)
RT = ramping rate = 2.00 (2 hr)

Start with target temperature (SP)

To run the program
Pent
Press ⊃ until you see P1 then ↓ to run
Press hold and then Press ⊃ until off and then press ↓

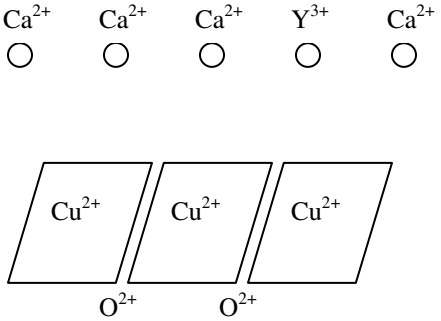
When the process is done we will repeat the process grinding and later baking two times more.

This process is called **Conventional Solid State Reactim Method** it is done at normal temperature.
Some times oxygen is needed to be added in order to perform the Making sample process.
Example
$Ca_{2+x}Y_{2-x}Cu_5O_{10}$
For x = 0 $Ca_2Y_2Cu_5O_{10}$



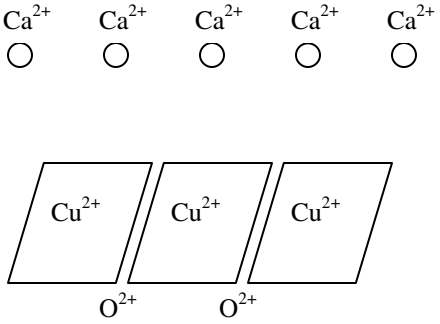

The charge is 0.

If x=1 $Ca_3Y_1Cu_5O_{10}$ it means:




Because the charge should be again 0 it means that one positron is free. This positron will go to the oxygen $O^{2-}$ getting $O^{+}$.
Because the interaction between Cu and O is an ionic bond, the bond now will be weaker.

If x=2 $Ca_4Cu_5O_{10}$ consequently




Two protons will be free and the $O^{2+}$ becomes O. It means that the ionic bond between Cu and O disappear.
Oxygen is needed during the baking. We must add extra oxygen in order to keep the oxygen atoms in the structure of the crystal.