Standard Operating Procedure for the Preparation of Oxides

A: Cooking

1. Clean an alumina mortar for major elements, and a silica mortar for trace elements *(See additional material: I)*
2. Obtain sample of interest, sticky note, and small labeling sticker.
3. For sticky note:
    - Write name, which Oxide, and Lot #

   For labeling sticker:
    - Write which oxide and Lot #
4. View pertaining information for the oxide on the table below:

Material	T°C	t (hours in furnace)	Notes
SiO_2	800	18	½ full (to make sure to have 7g)
TiO_2	800	4	
Al_2O_3	800	18	
Cr_2O_3	800	6	
Fe_2O_3	800	1	
Mn_2O_3 / MnO_2	700	4	Very long baking time if it's the first one
MgO	1000	24	1/3-1/2 full
$CaCO_3$	400	4	
Na_2CO_3	300	0.5	Consult Sarah Small crucible
Orthoclase (verre)	400	4	Consult Sarah
Apatite	110	4	Consult Sarah Vacuum Oven
K_2CO_3	110	4	
NiO	800	4-5	Dangerous chemical
ZnO	700	4	Up to 24h
Sc_2O_3	700	4-5	Not more than 5h !
Cu_2O	700	4-5	Dangerous chemical
Co	700	4-5	Dangerous chemical
V_2O_5	300	4-5	Dangerous chemical Melts at 690 C
Nd_2O_3	700	4-5	
La_2O_3	700	4-5	
Yb_2O_3	700	4-5	
Sm_2O_3	700	4-5	

5. Fill a crucible with the pertaining oxide powder.
    - **Make sure to view the Safety data sheet of the oxide before handling it! (See *Additional material: III*)**
    - Check *Additional material: II* for oxides that require further preparation procedures
    - **NOTE:** If $T < 800^oC$ use *ceramic* crucible. If $T \geq 800^oC$ use *alumina* crucible.

- Fill the crucible between about 1/3 or 1/2 of the way full for Major elements, and less full for minor or trace elements (Experiment dependent).

6. Set furnace to between 300-500 ºC.
7. Open furnace. Use clean tongs to take off crucible lid, then place crucible in furnace (it is best to pinch the side of the crucible with the tongs but avoid touching the sample in doing so) followed by placing the lid back on top of the crucible in the furnace. Place the crucible near the center of the furnace, avoiding the walls. Close furnace.
8. Place sticky note in front of furnace door, save labeling sticker for later. If multiple crucibles are in the furnace, draw a map. On sticky not write:
    - Time and day in, Time and day at ___ ºC (wanted temp), Time and day expected to be out, Time and day out. Fill in as necessary
9. Slowly raise temperature of furnace to specified temperature. Around 100 ºC / 15 min is generally good.

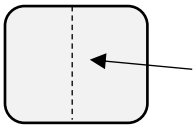
B: Vacuum

1. Once the allotted time is finished, prepare vacuum. Turn glass nozzle at the top of the vacuum jar lid so it is aligned with the small hole at the top of the container. Make sure tubing is connected. Remove vacuum lid and place upside down on counter next to vacuum. Turn pump on. you should hear air being sucked out of vacuum lid.
2. Obtain a pair of clean platinum tongs and oven mittens (In my experience, if $T \geq 700$ºC, you will need oven mittens to remove the crucible without burning your hand). Remove anything from your arms that could burn: watches, arm bands etc.
3. Open the furnace. With the tongs carefully remove the crucible lid and place it on the brick in front of the furnace. Next, carefully remove the crucible and place into open vacuum jar. Add crucible lid. Put lid on vacuum jar making sure that the little hole is still aligned with the nozzle. Close the furnace.
4. Let vacuum run for about 20 min. Once the pressure gauge has hit at least 20, you are generally fine.
5. After letting the vacuum run, turn the nozzle away (at least 90º) from the small hole in the vacuum lid. This will seal the vacuum. Turn off the pump and carefully remove tubing. Let sample cool in vacuum for another 30-45 min depending on initial temperature. In mean time, start preparing weighing procedure (steps *C:1-3*).
6. Once cooled, turn nozzle so that hole is aligned. You should hear air rushing into nozzle as the vacuum is released. Carefully (Don't tip jar!) slide the vacuum lid off of the vacuum jar. If lid does not move, it is best to wait several more minutes for the air to re-equilibrate, and then try again. Once lid is off, remove crucible and lid (with gloves) and place in safe location next to scale.

C: Weighing

Perform steps 1-3 when sample is in vacuum:

1. Clean area around analytical scale. With gloves, obtain a piece of weighing paper. Fold in half, being careful not to touch the inside side, cut the corners of the folded in half paper so that once unfolded, the weighing paper is this shape:



This is to help grab the paper once sample has been added, and to ensure maximum amount of sample is being weighed.

2. Turn on scale and place weighing paper on scale. Do not tare! (yet) Let weighing paper sit on scale until sample has been removed from vacuum.
3. Clean a spatula, and keep the cleaned mortar nearby were weighed sample will be placed.
4. **NOTE:** Go to "For MgO" section if weighing MgO.
5. Once step *B: 6* is completed place crucible next to scale. Tare scale. Over the weighing paper, with one hand carefully scoop out oxide powder using the spatula while holding the crucible with the other.
6. Once target weight is obtained, grab weighing paper being careful not to spill any of the oxide. Add oxide to alumina crucible. Rinse weighing paper with ethanol into crucible in order to make sure all of the oxide has been transferred.
7. Cover the mortar loosely with aluminum foil to prevent particle contamination, but still allowing evaporation of the ethanol. Place sticky note next to the mortar. Place small sticker on crucible lid and store in safe place.

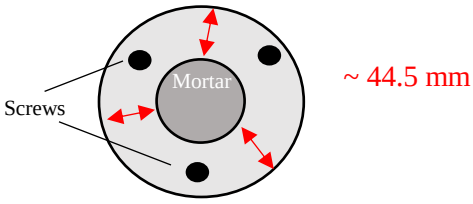
For MgO:

- Put needed weight of MgO on the weighing paper as fast as possible. Ex) if 0.58g of MgO is needed, it is ok to have somewhere between 0.55-0.64 g.
- Start a stopwatch on your phone, record the exact weight of the MgO, every time it changes (~30s):
    - Watch the weight.
    - Add a lap to the stopwatch (records time).
    - Do this for 5 minutes.
- Add time (x axis) vs. weight (y axis) to a plot in excel. The plot should look roughly linear or square-root shaped. Use an appropriate trendline and determine the y-intercept. This gives a proper estimation of the true (anhydrous) weight of MgO. Record this weight.
- Proceed back to step *C:6.*

D: Grinding and Mixing

For major elements:

1. Once all oxides of interest are in the mortar, lightly crush (don't mix) the larger grains within the mortar. Then wait for the ethanol to evaporate. Scrape any remaining sample from the pestle back to the mortar using a small (~1 inch) plastic sheet.
2. If powder still looks poorly sorted (i.e. there are larger pieces within a finer power) then manually and lightly grind the sample slowly for up to one hour before adding it to the automatic grinder. Make sure nothing falls out in the process.
3. Cover sample in the mortar with aluminum foil and move next to the automatic grinder.
4. Hold up the mechanic arm of the grinder and remove the pestle. Clean it thoroughly with DI water and ethanol. Unscrew the mushroom-shaped screws that hold the mortar in place making sure that they are also cleaned. Wipe down all additional parts of the automatic grinder that could contaminate your sample. Once the grinder is clean, remove the aluminum foil from your sample and place it in the center of the grinder inside the three screws.
5. Make sure the mortar is perfectly centered. To do this, use a ruler and measure the distance from the mortar to the edge of the grinder at the three different location between the screws. Depending on the mortar use, the distance should be approximately 44.5mm (See illustration below).



6. Once centered, screw the mushroom shaped holder back into the screws, making sure not to move the mortar from its centered position.
7. Add the clean pestle back into its holder, tightening using the two screws on the left side. The metal part should be flush with its holder, but still barely visible.
8. Add alcohol to transform the oxide powder into a viscous paste. Turn on the grinder (The time on the nob is not accurate, so just turn it to a high time)
9. Close the plastic box lid but keep is slightly open from the bottom with a small piece of wood to allow the evaporated alcohol to escape.
10. Check on the grinder often (at least every 15 min) making sure there is enough ethanol to keep the sample wetted and contained in the mortar. If the grinder makes a noise that sounds like the pestle is bouncing and hitting the mortar or squealing, stop the machine and tighten the 2 screws that hold the pestle in place.
11. After about 30 min, stop the grinder and let the sample dry. Use the plastic piece describer earlier to scrape back the sample that is stuck on the pestle. You can also use this plastic piece to scrape the sample of the edges of the mortar back into the center. Once dried, repeat steps *D: 8-11* two more times.

For trace elements:

1. Lightly and manually grind the batch of trace element oxides for 3x30 min in the silica mortar.
2. Use the little piece of plastic to scrape back any remaining sample from the pestle into the mortar.

E: Decarbonation

1. After the 3rd round of grinding, place the powder back into a clean alumina crucible using the piece of plastic. Place the crucible into the furnace at 300ºC using the technique describe in (*A*).
2. Raise the temperature of the furnace up to 800ºC at a maximum rate of 100ºC/hour! It is best to increase the temperature 50ºC every half hour.
3. Once decarbonation is complete, follow the steps in (*B*) removing the sample and letting it cool in the vacuum.
4. Transfer the sample onto a folded weighing paper and slide it into a small clean vile. Do not use ethanol at this point, as we want to keep the sample dry. Once all of the sample is transferred, close the vile and label it.

Additional Material:

I : Cleaning Mortar

Major elements:

1. Obtain and Alumina Mortar, rinse once with ethanol.
2. Make sure grinder is clean as explained in *D: 4*.
3. Position mortar perfectly in center of automatic grinder (*see step D: 5, illustration*).
4. Add silica and ethanol to the mortar so that a slurry develops.
5. Make sure pestle is held tight in place (*D: 7*).
6. Let grinder run for 2x15 minutes, making sure enough alcohol remains within the slurry.
7. (Optional) Use piece of plastic to scrape leftover silica from the pestle back into the mortar.
8. Remove mortar and pestle and rinse with DI water. Use ethanol to clean mortar again before samples are added. If stored for a long time after cleaning, add aluminum foil to cover mortar.

Trace elements:

1. Obtain a silica mortar, rinse with ethanol.
2. Add Silica powder and ethanol to the mortar, so that a slurry-like consistency develops.
3. Manually grind the powder with the mortar and pestle for ~ 30 min.
4. Give mortar a final rinse with ethanol and cover in aluminum foil.

II: Material Specifics

$CaCO_3$ / Al_2O_3

- Do not keep in oven over the allotted time. $CaCO_3$ will lose CO_2 and Al_2O_3 can become corundum.
- For $CaCO_3$, note on sticky note and in lab notebook whether it is the first time this batch of powder has been cooked. $CaCO_3$ should not be cooked over three times.

K_2CO_3 (?- ask Sarah for specifics)

- Tends to absorb CO_2. Only use small amount in bulk composition, otherwise use orthoclase or microcline.

Apatite (?- ask Sarah for specifics)

- Crush apatite crystal in mortar with ethanol to obtain a fine powder. Put the mortar on top of the furnace with aluminum foil to let dry. Once dry, put the powder in a crucible and place inside a vacuum oven at temperature 110-120 ºC.

Orthoclase (?- ask Sarah for specifics)

- Saw small thin sections. Using steel pestle, place both the thin section and pestle near the base. With a hammer, hit pestle 2 or 3 times. Crush into powder. If needed, use weighing paper to crush bigger pieces and prevent them from flying around. Use a magnet to collect steel shavings. Place in clean mortar and grind to powder.

III: Safety Information

NiO, Co, V_2O_5, or Cu_2O:

- These elements / oxides are dangerous and can be extremely harmful to your health and well-being.
- Always wear gloves, safety goggles, lab coat, and respirator any time you are handling these materials.
- Handel these samples with care. Transfer powder out of containers slowly to minimize to possibility of inhaling fine powder or allowing spontaneous combustion.
- Read the lab safety sheet in detail before handling these materials.
- Immediately call poison center if exposed **(800-222-1222)**
- Thoroughly rinse all body parts that encountered material for at least 15 minutes.
- If inhaled, do not induce vomiting, move to well ventilated area in position comfortable for breathing.

$CaCO_3$, La, MnO_2, Nd, K_2CO_3, SiO_2, V, TiO_2, or Y

- These materials can be extremely harmful, if exposed repeatedly.
- Always wear gloves, safety, glasses, and lab coat. Respirator recommended if fine powder becomes suspended in air.

- Skim safety data sheet for specifics of each material
- Rinse thoroughly with water if body contact with material occurs.
- Call poison center if symptoms of irritation persist.

Al_2O_3, Cr_2O_3, Glassy C powder, Fe_2O_3, La_2O_3, MgO, Nd_2O_3, Sm, Sc_2O_3, Yb_2O_3, or ZnO, Sm_2O_3

- These materials are generally safe
- Always were gloves. Safety goggles recommended.
- Refer to lab safety data sheet for specifics, or if irritation occurs.