HYDROXIDE PREPARATION

It is extremely important that during this step only one test tube at a time is opened. This is to ensure that caps are not switched.

Make sure all the beakers containing Al and Be fractions have been returned to the hot plate. Dry down at a setting of 275 (sub-boiling). This will take several hours, perhaps overnight.

FOR BE ONLY --- After samples are dry, add 1 ml perchloric and fume to dryness at full on hotplate setting. This step effectively reduces boron in targets and should not be omitted. Do this step as close as possible to the final precip step.

Remove the beakers one at a time from the hot plate and set in the hood. Place a small disposable pipette in the beaker. Use the Eppendorf to add 1 ml 1.2N HCl to the beaker to dissolve the cake. Swirl.

Put the box of ACID RINSED test tubes and caps on the counter. Place a test tube rack in the hood. Put a clean tube in the rack and use the disposable pipette to transfer the sample to the 15 ml test tube. Transfer the label from the beaker to the test tube. Wash the beaker with several ml of DI water and transfer the wash to the test tube. Do this two times. Aim for a total of 5-7 ml.

To precipitate the sample, use the precision pipette to add approximately 0.20 ml of 15% NH_4OH that should be made fresh every several weeks. Do not put the pipette tip inside the tube. Cap the tube. **Vortex for 20-30 seconds**. Check the pH of each sample and make sure it is between pH 8.1 and 8.9; if not, use NH_4OH or HCl to adjust it. **Sit aside for at least several hours.**

After samples have sat, vortex again and **check all** of the **pH's** again. They tend to change even after only sitting for the minimum time.

Do the same for all the remaining fractions of Al and Be.

Centrifuge each sample for four minutes at the highest speed. Decant the liquid into the waste container. It should be treated as Be waste and added to the acid waste bottle.

Add about 0.5 ml DI water. Vortex. **Let the Be fractions sit at least 1/2 hour before centrifuging again.** Centrifuge. Decant. At this point the samples are in a gel form and are ready to be dried down in quartz crucibles on a drying block.

DRY DOWN and BURNING

WORK IN THE COLUMN HOOD

Use clean gloves to remove quartz crucibles and put them in the 16 outer holes on the two drying blocks. PUT BE CRUCIBLES IN THE BE HOLES AND AL CRUCIBLE IN THE AL HOLES. The Be crucibles have seen a second HF etch. The Be quartz crucibles are thinner and more etched.

Line up the samples in BATCH ORDER. DOUBLE CHECK.

You will add each Be and Al sample in sequence to its quartz crucible.

Take the first Be sample, the blank. Use a dropper bottle filled with DI water and add approx. 1-2 drops of DI water to the sample. Use a clean disposable pipette to liquefy the gel and transfer to the proper quartz crucible. Be careful not to drip the sample at all. Move the pipette so it does not come over any other crucibles. Add 1-2 more drops DI water to the centrifuge tube and transfer to the crucible. Try to use the least amount of DI water necessary to transfer the sample. DO NOT FILL THE CRUCIBLE TO THE VERY TOP. If there is too much sample to fit in the crucible at one time, leave remainder in the centrifuge tube, re-cap and wait until a significant amount of the sample has dried down and then transfer remainder to the right quartz crucible. REPEAT FOR ALL OTHER SAMPLES. Cover the crucible temporarily with the blue cap after it is filled to protect the sample and to mark your place in the block.

Carefully place the blocks in the block heaters, turn on the heaters and dry off overnight. Using the VWR Heat Block on the "high" setting, set to ~4 =87 C. (Presently this is our righthand side heating block) Using the VWR Standard Heatblock on "high" setting adjusted to ~90-95 C. (This is in the left hand side position at present.)

When samples are dry, turn off the heat blocks and let them cool fully. After the samples are cool, remove the block to the hood counter.

INSPECT THE TONGS. If there are any chrome chips, remove them with sandpaper and acid wash tongs before use.

Light Bunsen burner.

Start at one side of block. Pick up crucible with tongs. Carefully glide the sample over the cooler part of the flame to burn off the water portion of the sample. This is indicated by white plumes of smoke and some spitting sounds from the sample in the vial. When all signs of fuming have stopped, hold in center of flame for one minute, using the timer to measure the time. When the timer alarm sounds, set the sample down in block to cool. Repeat for all samples, working systematically across the block.

When all samples have been burned and are in the block, cover the block with a plastic cover (we use an inverted tupperware container) to prevent any foreign material from contaminating the samples.