Procedure for Disk Fusion Using M4 Fluxer,
LOI, and 12.5% v/v HCl

Materials:

-sample and standard powders
-porcelain crucibles (= to number of samples)
-metal basket for transporting porcelain crucibles
-1 large pair of tongs
-1 small pair of tongs
-desicator jars
-electronic balance
-metal spatulas
-camel hair brushes
-Li-borate flux (49.75 % Li-metaborate, 49.75% Li-tetraborate, 0.50% LiBr)
-LiI solution
-large porcelain crucible with pouring spout
-6 platinum crucibles
-20 µl pipetter with pipet tips
-weighing paper
-Citranox

Procedure:

1. LOI all samples and standards you are going to use:

    a. Turn on the muffle furnace and set the temperature to **1000 ºC** (marked on dial).

    b. Wipe the porcelain crucibles clean with a kimwipe and place them in the metal basket (max six at a time). Use the large tongs to place the metal basket, using the large tongs, into the pre-heated muffle furnace for **30 minutes**.

    c. Remove the metal basket from the muffle furnace, using the large tongs, and set the basket onto the brick next to the desicator jars. Use the small tongs to remove one crucible at a time from the metal basket, placing each in the desicator jar until cool (about **15 minutes**). Keep the lid of the desicator very slightly open to allow heat to escape as the crucibles cool.

    d. Make sure the balance is level. Turn it on and calibrate it.

    e. Remove one crucible from desicator jar, using tongs, and place on balance. Weigh crucible and record weight on LOI sheet. Do not tare the balance. If the weight varies excessively, put the crucible back in the desicator to fully cool. Add approximately **3-4 grams** of powdered sample, record weight of crucible +

sample, and return crucible to desicator jar, using the small tongs. Repeat with six crucibles and samples.

f. When all samples have been weighed, use the small tongs to place the crucibles on the metal basket. Put the metal basket into muffle furnace, using the larger tongs. Leave the crucibles + sample in the muffle furnace for **30 minutes.** Note the position each crucible is in (scratched faintly on the side of the metal basket.

g. Remove the metal basket from the muffle furnace, using the large tongs, and set the basket onto the brick next to the desicator jars. Remove crucibles, using tongs, from metal basket and place in desicator jar until cool **(20 minutes).**

h. Remove one crucible at a time from desicator jar, using tongs, and place it on the balance. Weigh the crucibles with dry sample and record the weight. Once satisfied that you have reliable weights, dump the sample into the trashcan and wipe the crucible with a kimwipe.

i. Calculate the LOI with the following formula:

$$\frac{\textbf{(Crucible \& Wet Sample – Crucible \& Dry Sample) *100}}{\textbf{(Crucible \& Wet Sample – Crucible)}}$$

j. Enter your weights into the LOI spreadsheet and save it on the Room 206 Data Reduction computer under the file path ICPRaw>LOIs. Save it with the filename "*YYMMDD_PROJECT NAME_Initials"* where YYMMDD is the date, PROJECT NAME is the name of your project, and Initials are your initials.

2. Weigh sample and flux mixture for fusion:

a. Make sure the balance is level, turn it on, and calibrate it.

b. Place porcelain crucible (the large one with the pouring spout) and tare the balance. Weigh out **1.0000 ± 0.0010 grams 49.75% Li-metaborate/49.75% Li-tetraborate/0.50% LiBr flux** (be careful, there are two similar-looking bottles) and pour into platinum crucible. Record the weight and crucible number (not 0130) on your Data Sheet. Brush remaining flux into platinum crucible and cover it with a sheet of weighing paper. Close the lithium metaborate as soon as you are done.

c. Return the empty porcelain crucible to the balance. Tare the balance.

d. Weigh out **1.0000 ± 0.0010 grams sample**. Pour to the side of the crucible. Record the weight and tare the balance.

e. Add another **4.0000 ± 0.0010 grams 49.75% Li-metaborate/49.75% Li-tetraborate/0.50% LiBr flux** flux to the porcelain crucible**.** Pour the flux toward

the side of the crucible opposite the sample, so that if you go over the target weight you can remove some flux without removing any sample. Record the weight.

f. Remove the porcelain crucible from the balance and place the crucible on a piece of weighing paper. Mix the sample and flux with the flat end of a metal spatula until homogeneous. Pour the mixture into the platinum crucible on top of the flux that is there already. Brush off the metal spatula and the porcelain crucible into the platinum crucible. Do not set down the metal spatula before brushing it off into the platinum crucible.

g. Set the filled platinum crucible aside and cover it with the sheet of weighing paper.

h. Repeat from (3b) for all three samples, using a different platinum crucible each time, until you have a batch of three. When you are finished, throw away used Kimwipes, turn off the scale, and replace the cover. Never leave anything on the scale that you are not actively weighing.

3. Set up the M4 Fluxer for running:

a. Replace the magnetic stirring attachment with the disk mold tray if necessary. Place a platinum disk mold onto the mold tray for each sample you are running.

b. Pipet **40 µl of LiI non-wetting agent** into each platinum crucible. Do not let the lithium iodide touch the crucible, as this may cause discoloration of the platinum. Use the same pipet tip for all three samples but eject it into the trashcan after use. Clip the crucible into the green hooks of the M4 one side at a time, bending the hooks as little as possible.

c. Turn on the vents with the switch located behind the DI water tanks.

d. Turn on the gas. Be sure to open both valves.

e. Turn on the fluxer by flipping the red switch in the front to on. Also flip the switches corresponding to each crucible.

f. Run the fluxer disk program (**P7**). The program is also called "Chantal's Disc" and can be found in the "Data Bank" folder of the M4PCLINK software on the room 208 computer. Turn off the gas and vent when you are finished.

4. Put each disk in a labeled, clear, plastic petri dish with a piece of round filter paper in the bottom.

Cleaning:

1. Platinum crucibles:

a. Fill the plastic container marked, “Citranox for Pt crucibles” with 10% Citranox + DI water solution.

b. Place the plastic container with the platinum crucibles in the ultrasonic for at least 30 minutes.

c. Rinse each platinum crucible 5 times with DI water and 3 times with 18 mega ohm water. Dry in ICP-MS drying oven (about 15 minutes).

Note: If the crucible(s) contains abundant beads or is oxidized after the ultrasonic bath:

i. Fill the platinum crucible(s) with 5 grams of Li-metaborate/Li-tetraborate flux. You can use an already made cleaning disk (flux only) if one is available, but crush it with the agate mortar and pestle before placing it into the crucible.

ii. Place the disks and crucible(s) on the fluxer.

iii. Run the disk fusion procedure (P7).