Procedure for Solution 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
-2 2000 ml graduated cylinders
-1 100 ml graduated cylinders
-1 500 ml beaker
-repipet dispenser
-Li-metaborate flux (98.5% Li metaborate, 1.5% Li bromide)
-LiI solution
-large porcelain crucible with pouring spout
-6 platinum crucibles
-3 Teflon beakers with magnetic stirring bars
-20 µl pipetter with pipet tips
-10 ml pipetter with pipet tips
-weighing paper
-Citranox

Procedure:
1. LOI all samples 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. Prepare 12.5 % volume/volume HCl

a. Pour **~1500 ml of deionized water** into a 2000 ml graduated cylinder.

b. Add **250 ml** of **38% HCl** to the graduated cylinder with the water in it. Using the 100 ml graduated cylinder, add 100 ml of HCl two times and then add another 50 ml.

c. Fill the 100 ml graduated cylinder, which was used to measure the HCl, with deionized water and pour into the 2000 ml graduated cylinder. Repeat as necessary to make **2000 ml of HCl** solution.

d. Pour the HCl solution into the other 2000 ml graduated cylinder to mix the solution.

e. Pour the HCl solution into a 500 ml beaker and fill the repipet dispenser (~1000 ml) that is labeled '**12.5 % v/v HCl.**'

f. Store the remaining HCl solution in 500 ml narrow mouth bottles and label with '12.5 % v/v HCl', your initials, and the date. Store these bottles under the hood until you need them.

3. 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 **0.6000 ± 0.0006 grams 98.5% Li metaborate/1.5% Li bromide** (be careful, there are two similar-looking bottles) flux 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 **0.20000 ± 0.0002 grams sample**. Pour to the side of the crucible. Record the weight and tare the balance.

e. Add another **0.6000 ± 0.0006 grams of lithium metaborate** 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 each sample, using a different platinum crucible each time, until you have a batch of three. Throw away used kimwipes, turn off the scale, and replace the cover. Never leave anything on the scale that you are not actively weighing.

4. Set up the M4 Fluxer for running:

a. Fill the number of Teflon beakers needed (up to three) with **80 mL** of **12.5 %v/v HCl solution** using the repipet dispenser. Add a magnet to each beaker. The repipeter is set to distribute 40 mL at a time, so do it twice. Place the filled beakers onto the fluxer. Note: if you see bubbles in the tube of the repipeter, tilt the repipeter gently and empty a small amount of HCl into a spare glass beaker.

b. Pipet **20 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. Put away the LiI as soon as you are finished to prevent deterioration by sunlight.

c. Clip the crucible into the green hooks of the M4 one side at a time, bending the hooks as little as possible.

d. Turn on the vents with the switch located behind the DI water tanks. Also turn on the gas. Be sure to open both valves.

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

g. Run the fluxer solution program (**P6**). The program is also called "Chantal's SolnB" and can be found in the "Data Bank" folder of the M4PCLINK software on the room 208 computer.

HINTS: a) If a glass bead(s) sticks to the lip, bottom, or side of the crucible, wait until the program stops and gently brush the bead into the solution with a kimwipe. Generally, samples with high SiO_2 contents (e.g. granites) are the stickiest. Platinum scratches easily and is expensive to replace, so **do not scrape the crucible to get a bead**; if it does not brush off easily, you will have to redo the fusion. The bead should come off in the ultrasonic bath, but should not then go into your solution.

b) After the molten sample is poured into the beakers make sure that all of it gets mixed by the magnetic stirrers. If not, nudge the beaker over with a metal spatula to ensure all of the glass dissolves.

c) If some glass remains undissolved in the beaker (as in after nudging a bead in), run the stirring function (**F10**) for as long as it takes to dissolve the remaining glass. Press Stop to end the stirring.

6. After the program is finished, remove each beaker from the fluxer and pour the solution into a clean **125 ml wide-mouth bottle** using a larger magnet on the outside of the beaker to keep the magnetic stirring bar from falling out of the beaker. Label the bottle with the sample name, the date, and the preparer's initials as so:

Initials
MM/DD/YY
Sample Name

7. Dilute samples to be analyzed for major elements:

a. Fill an additional **125 ml wide-mouth bottle** with **40 ml** of 12.5 % v/v HCl solution with the repipet dispenser for each sample.

b. Pipet **20 ml of sample** solution into the new bottle (pipet twice with the **teal 10 ml pipetter**). Label the new bottle the same as the other bottle but put "1/3" underneath the sample name.

Cleaning

1. Platinum crucibles:

i. fill the plastic container marked, "Citranox for Pt crucibles" with 10% Citranox + DI water solution.

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

iii. 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. Replace the magnetic tray with the disk holder. Place the disks and crucible(s) on the fluxer.

iii. Run the disk fusion procedure (P7).

2. Teflon beakers and magnets:

a. Scrub Teflon beakers and magnets with the green scouring pad (in the labeled Ziplock baggie) with DI water.

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

3. Pipet tips for dilutions:

a. Rinse three times with nitric acid from red nitric acid bath. **Do not let the pipet tips sit in the red nitric bath.**

b. Rinse the pipet tips 3 times with DI water and 3 times with 18 mega ohm water. Dry in ICP-MS drying oven.