Hard Rock Preparation for ICP: User Guide

Manual Information

Author(s):	L. Brandt
Reviewer(s): Management Approval:	H. Barnes, K. Bronk D. Houpt (Supervisor of Analytical Systems) 7 May 2014
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In This User Guide

Introduction

Hard rock analysis is used to determine basalt whole-rock geochemistry by ICP-AES. Fused glass beads are dissolved in 10% HNO_3. This solution is further diluted and the resultant analytes are subsequently introduced into the ICP-AES. The complete process from sample table to ICP-AES analyses takes approximately 3 days.

Rock samples are prepared for ICP analysis by crushing to a fine, talc-like powder using multiple crushing and grinding steps. After the sample is ground, Loss on Ignition (LOI) is determined on the ground sample, the ICP sample bead is formed from the material left after ignition, and the sample bead is solubilized and diluted for instrumental analysis.

Safety Information

Chemical Hazards

- Lithium bromide (LiBr) – hazardous when ingested or inhaled; irritant for skin or eye contact
- 0.0172 mM LiBr wetting agent – mildly toxic
- Concentrated HNO_3 – **strong acid!**
  - Very hazardous in case of skin or eye contact or ingestion.
  - Strong oxidizer.
  - Use caution when handling!
  - May produce hazardous fumes!
- 10% HNO_3 – **strong acid!**
  - Very hazardous in case of skin or eye contact or ingestion.
  - May produce hazardous fumes.
- Methanol – Highly flammable liquid.
  - Hazardous in case of skin or eye contact, ingestion, or inhalation.
  - Severe overexposure can result in retinal damage or death.
- Isopropyl alcohol – Highly flammable liquid.
  - May spark upon pouring substantial quantities.
  - Hazardous in case of eye contact, ingestion, or inhalation.
  - Slightly hazardous in case of skin contact.
- Acetone – Highly flammable liquid.
  - Hazardous in case of skin or eye contact, ingestion, or inhalation.
- DI water – Hazardous if ingested
- Lithium metaborate ($LiBO_2$) – Very hazardous in case of ingestion!
  - Hazardous in case of eye contact or inhalation.
  - Slightly hazardous in case of skin contact.

Inhalation Hazards

- Rock samples ground to a talc-like consistency (which is the objective) can be an inhalation hazard. Take care not to inhale the dust – breath masks are recommended if the powder flies easily.

Apparatus, Reagents, & Materials

Laboratory Apparatus

- Compensated Dual Analytical Balance System (***Figure 1***)
- Drying ovens at 110°C and 60°C

Rock Grinding

- Splitting room saw
- Buehler grinder/polisher with 70 µm grit diamond grinding wheel
- Sonicator (with small water bath)
- X-Press crusher (***Figure 2***)
- Spex Shatterbox with tungsten carbide (WC) grinding vessel (***Figure 3***)
- Spex Mixer Mill (***Figure 4***)

LOI/Bead-Making

- Fisher Ashing Furnace (***Figure 5***)
- Sample Bead Maker (***Figure 6***)

Dissolution/Dilution

- Wrist-action shaker (***Figure 7***)
- Acid baths (***Figure 8***)

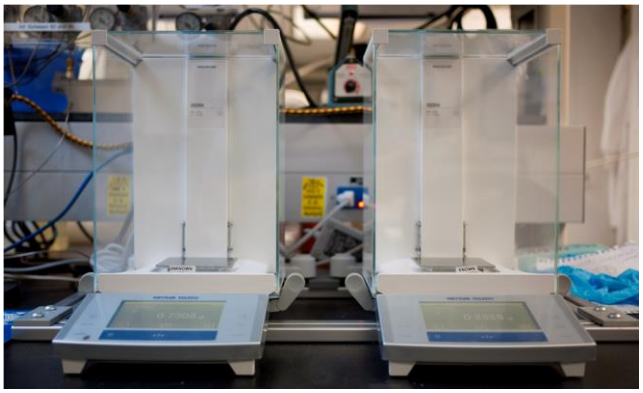

Figure 1. Mettler-Toledo Dual Analytical Balance System.

Figure 2. X-Press Sample Crusher.

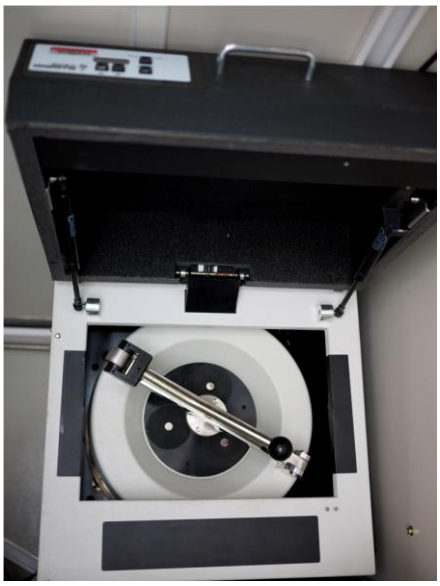

Figure 3. Shatterbox.

Figure 4. Mixer Mill.

Figure 5. Furnace.

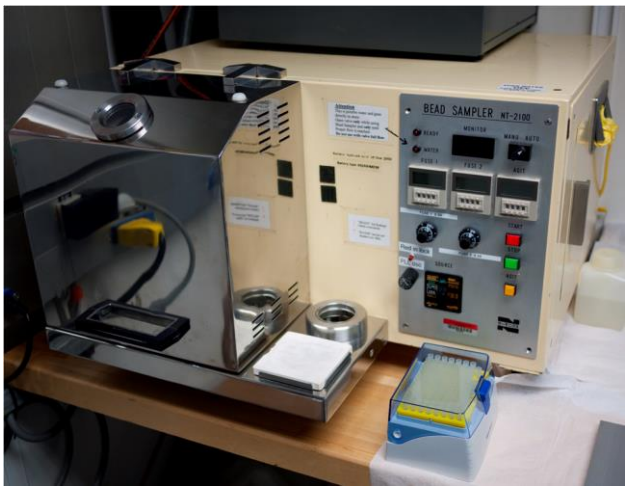

Figure 6. Bead Maker.

Figure 7. Burrell Wrist-Action Shaker.

Figure 8. Acid Baths.

Reagents

- 0.0172 mM LiBr wetting agent (0.15 mg ultrapure LiBr in 10 mL DI water)
- 10% nitric acid (143 mL concentrated nitric acid/L of solution). Caution! Always add acid to water.
- Isopropyl alcohol, laboratory grade
- Methanol, laboratory grade
- Acetone, laboratory grade
- DI water (18.2 MΩ laboratory water)

Materials

Grinding Samples

- Beakers
- Glass cleaner
- Tweezers
- Teflon spatula
- X-Press aluminum die
- Core liner pieces and clear endcaps
- Delrin plugs
- Acid-washed 1-oz glass bottles
- Weighing paper, 6 x 6
- Kimwipes

LOI/Sample Bead

- Quartz crucibles
- Tongs
- Vials containing 400 mg lithium metaborate flux (preweighed on shore) (Figure 9)
- Milligram calibration weighing set
- Weighing paper, 2 x 2
- Acid-washed vials for excess ignited powder
- Agate mortar and pestle
- Pt-Au crucibles

Figure 9. Preweighed Vial Containing Lithium Metaborate Flux.

Preparing Rock Samples

Rock samples are prepared for ICP analysis by crushing to a fine, talc-like powder, using the following procedures on each sample:

1 Cut to size (see ***Cutting Samples to Size***).

2 Polish (see ***Polishing Samples on Diamond Wheel***).

3 Clean (see ***Cleaning Samples***).

4 Dry (see ***Drying Samples***).

5 Crush (see ***Crushing Samples in the X-Press***).

6 Grind (see ***Grinding Samples in the Shatterbox***).

Cutting Samples to Size

Cut samples small enough to fit into the X-Press crusher using the core lab splitting room saw, according to the following guidelines:

- Cut samples to ~1–2 cm in length and width.
- Avoid veins, infilled vugs, etc., in the cut samples.
- Remove as much contaminated material as possible.
- Avoid cutting pieces too small or to irregular shape.
- Cut the first samples smaller to get a feel for the relative rock hardness.
- Contact the petrologist(s) if cutting reveals unexpected features.

Notes about altered samples:

- It may be desirable to crush the sample into 1 mm size pieces and hand-pick/separate the vesicles/vein material from the whole-rock basalt to obtain enough unaltered basalt for analysis.
- Speak to the scientist about this method if alteration is visible within the sample.

Polishing Samples on Diamond Wheel

Clean the surfaces of the cut samples by grinding on a high-speed, diamond-impregnated disc (70 µm grit) to remove rock that may have been contaminated by the drill bit, saw blade, or other unwanted material.

Note: Use the Buehler grinder/polisher in the Thin Section Lab with an 8 inch grinding wheel replacing the thin section grinding lapwheel. A spare 8 inch grinding wheel and discs are stored in the cupboard under the lapwheel. If the diamond disk does not have self-adhesive, use an aluminium ring to hold the disk in place.

1. Turn the water on with enough flow to cool the sample and clean the wheel, but not full force.
2. Start the wheel on low speed and then increase the speed to a higher setting.
3. Hold the sample against the wheel and grind off all surfaces that could have come into contact with a saw blade, bit, core barrel, or any altered surface. Round the corners of the sample to prevent cutting and splitting the Delrin discs during the X-press crushing process (see Crushing Samples in the X-Press).
4. Move the sample back and forth across the wheel. Do not hold the sample in one place and wear out a single groove of the diamond disk.

Cleaning Samples

After polishing the sample surfaces, wash the rock chips in lab grade methanol (if available) or lab grade acetone to remove contaminants such as oil from the skin and residue accumulated while using the diamond wheel. From this point onward, wear gloves when handling the samples to avoid reintroduction of skin oil contamination. While the samples are wetted with solvent, keep them under the hood if possible.

Caution: Vapor inhalation of methanol or acetone should be kept to a minimum – work under the canopy hood, and ensure that it is operating while using these chemicals to avoid concentration of the fumes. Care should be taken to avoid sparks or other conditions that may ignite the fumes.

1. Place each sample in a clean beaker and write the number/name of the beaker on the corresponding sample bag.
2. Pour enough methanol, acetone, or isopropyl alcohol, per scientists request, into the beaker to cover the sample and to keep the beaker from floating in the sonic bath.
3. Sonicate for 15 min using the small clean sonic bath. (The large sonicator requires too much water for this procedure.)
4. Dispose of the solvent by placing it into a fume hood and allow the solvent to evaporate.
5. Add DI water to the beaker to cover the sample and rinse/sonicate the sample for 10 min.
6. Repeat the DI rinse until the water is clear after sonicating. If the samples are very soft and/or clay rich, they will not reach a clear DI water state. Continuing to sonicate will only dissolve the sample.
    - If after 3–4 washings, the water still isn't clear, go to the next step.
    - If a sediment sample appears to be dissolving, rinse with DI only once or twice; typically 3 times is adequate for basalts.
7. After the final rinse, decant as much water from the beaker as possible.

Drying Samples

Clean the oven at the beginning of the expedition, before it is turned on. The top inside the oven in particular must be clean because the samples are open in the oven. The oven used for ICP sample prep should be used only for ICP samples.

Caution: Do no place samples still wet from flammable solvents in the oven!

1. Place the beakers on a tray and insert the tray into a sample prep oven at 110°C for 12 hr.
2. Note the time each set of samples goes into the oven to dry.
3. After 12 hr, remove the samples from the oven to cool.

Crushing Samples in the X-Press

Crush the cleaned and dried samples into small pieces with the X-Press to improve ease of grinding with the Spex Shatterbox (or Spex Mixer Mill for very small samples). To determine which shatterbox vessel size is appropriate, use the following guidelines:

- Large vessel:
  - Samples are crushed in the X-Press into 10 mm long pieces
  - ~31 g of sample is required (using less sample may crack the vessel)
- Small vessel (most commonly used):
  - Samples are crushed into 3 mm long pieces
  - ~10–15 g of sample is required

To crush the samples with the X-Press, proceed as follows:

1. Clean the area around the X-Press with glass cleaner and then with isopropyl alcohol.
2. Clean the X-Press inside and out with glass cleaner followed by isopropyl alcohol.
3. Put on powderless nitrile gloves and "wash" your gloved hands with isopropyl alcohol. Periodically repeat the washing.
4. Place Kimwipe sheets on all working surfaces.
5. Clean the supplies needed during the crushing procedure with isopropyl alcohol and arrange them on the Kimwipes: tweezers, X-Press aluminium die, core liner pieces, clear end caps, and Delrin plugs.
6. Apply the appropriate sample label for each sample on acid-washed 1-oz glass bottles. It may be helpful to place a small label on the lid of each bottle for quick identification.
7. Put a piece of 6 cm x 6 cm weighing paper in the X-press. Collect clean stainless steel base and two clean Delrin discs. Clean the Delrin discs before use with isopropyl alcohol.
8. Remove a sample from the oven. Working with a hot sample facilitates crushing.
9. Put one of the Delrin discs in the stainless steel base:
   - Arrange one or more sample pieces on the disc.
   - Place the other Delrin disc on top of the sample pieces.
   - Carefully slip the piece of core liner onto the stainless steel base. The liner prevents scattering crushed pieces.
   - Stack the aluminium die on top of the Delrin disc.
10. Open the X-Press door and carefully place the sample crushing setup (as described in previous step) in the center of the platen.
11. Keep pressure on the Al die and carefully tighten the jackscrew (snug, not tight) until the shaft is secure.
12. Close the plastic door and tighten the release valve on the right side of the X-Press. The X-Press will not operate unless the door is closed. Stand off to the side of the X-Press, so your body is not in line with the hydraulic gauge, which is the most likely place for the Shatterbox to give. **Wear eye protection!**
13. Push the switch down to activate the piston.
14. Bring the pressure up to 5 or 6 tons to start. Sometimes it takes a few minutes for a sample to crack. Leave the machine on for up to 5 min before rotating the sample 90° to change stress.
15. Watch the gauge and the Delrin discs, as sharp sample corners can sometimes split the discs forcefully. If the discs blow, the sample may shatter inside the press. Take your time and go slowly.
16. If the sample does not crack, take the assembly apart and rearrange the sample pieces with clean tweezers (this is a common occurrence). Only rearrange large sample pieces. Crushed samples can be placed in a labeled bottle.
17. If the sample still does not crack, try letting off the pressure and then running it back up multiple times. The crack sometimes happens during the decompression. This will have to be done half a dozen times or so for an average sample. If the material is relatively soft, one or two crushings will be enough. A large, very hard sample may take many tries. If the Delrin disc cracks, replace with new one(s). Also, watch for pieces of Delrin plug that sometimes scrape off. Separate small pieces of Delrin disc from the crushed sample.

18. Wash and scrub the beakers and rinse them thoroughly with DI water after all of the samples have been crushed. If time allows, place the beakers in the acid bath for 15 hr after cleaning with DI water.
19. Put the beakers in an available oven in the chemistry lab to dry.

Grinding Samples in the Shatterbox

After the samples are crushed, they are ground into a very fine powder. If possible, grind the entire sample together in a single grinding vessel to achieve complete homogenization. One method of grinding is to use the Shatterbox equipped with a tungsten carbide (WC) grinding vessel.

Note: Only use the WC vessels, as the steel vessels provide numerous contaminating elements. Handle carefully. The grinding vessels are heavy and brittle, making them subject to chipping or cracking if dropped.

1. Transfer the sample pieces to the grinding vessel; if any pieces are on top of the puck or ring, use plastic gloves, tongs, or clean paper to move the sample into the cavity.
   - Small vessels require a minimum of 5 mL and maximum of 20 mL; 10 mL usually works best.
   - The largest vessel requires a minimum of 20 mL and maximum of 60 mL; 40 mL is usually optimal.
2. Replace the lid to the vessel and carefully place the whole assembly in the Shatterbox.
   - The large vessel sits directly in the Shatterbox
   - Small vessels sit on a plate with pins in it.
3. If only one vessel is used, place it on the center pin of the mounting plate. If two vessels are used, add the third vessel with no puck in it. You cannot run either one or two vessels on the outer pins or the Shatterbox will be out of balance and this can cause much damage.
4. Put the rubber pad and the top plate over the grinding vessel(s).
5. Bring the safety arm over the top of the vessel (centering the plate beneath the arm's centering bracket) and fasten it securely.
6. Set the timer to 2–5 min, which may be necessary to grind the sample into a talc-like powder.
7. Turn the switch to **On** and press **Start**.
8. If the Shatterbox makes any ominous noises (metal-to-metal sounds), shut it off and find out what is wrong.
9. When finished, remove the grinding vessel(s) and place them on a Kimwipe or a piece of white paper.
10. Open the grinding vessel and feel the ground powder.
    - If it feels like talcum powder on the skin (i.e., no grit; apply some on the forearm with tweezers; if the tweezers touch the skin, they need to be washed before using them in the powder again), it is fine enough.
    - If it feels gritty, the sample needs to be to ground a little longer.
11. Use a clean piece of weighing paper or the Teflon spatula to clean the powder from the top surface of the puck and ring.
12. Disassemble the vessel carefully using plastic gloves or clean paper towels. Put the puck, ring, and O-ring on the inverted lid.
13. Carefully pour the powder from the vessel onto clean weighing paper. If any powder remains behind (in the bottom corner), use a clean plastic spatula to dislodge it.
14. Transfer the powder to the correct pre-labeled sample bottle. If multiple batches of the same sample are ground (i.e., coarse-grained rocks or inter-laboratory standards), transfer the powder to a clean-labeled Ziploc bag and homogenize it as needed.

Cleaning the Grinding Vessels between Samples

1. Wearing nitrile gloves, wash the individual pieces of the grinding vessels with DI water and a small piece of a scouring pad (no soap).
2. Spray the washed pieces with isopropyl alcohol and wipe with a Kimwipe.
3. Lay the pieces to dry on a Kimwipe.
4. Wash the O-ring on the large grinding vessel with water and dry with a paper towel (do not use acetone).

Note: Do not blow pieces dry with compressed air, as ship's air is dirty. The lid is particularly vulnerable to surface rust if it is not dried quickly and thoroughly.

Note: Some parts of the small grinding vessels are a set and the lids and pucks should not be intermixed.

Determining LOI and Making Sample Bead

ICP analyses are completed on ignited samples used to determine LOI. Petrologists use LOI as an indication of degree of alteration. Low LOI values suggest relatively fresh, unaltered basalt. High LOI numbers suggest alteration, i.e., possibly a lot of clay or other alteration minerals within the sample.

The LOI of a sample is determined by weighing a small amount of the sample (~5 g) before and after ignition (i.e., in ashing furnace for 12 hr cycle with maximum temperature at 1020°C for 4 hr). Usually the sample loses weight as water is driven off, though an iron-rich, water-poor sample may gain weight.

Loss on Ignition

Determining a sample LOI comprises three procedures:

- Pre-ignition weighing
- Igniting samples
- Post-ignition weighing

Pre-ignition Weighing

Pre-ignition weighing is performed on the Mettler-Toledo Dual Balance (for a more in-depth guide to using the balance refer to the Balance User Guide on Cumulus). Ensure you have enough acid-cleaned quartz crucibles before starting to weigh the crucibles. Number each crucible with a diamond tipped pen.

Weighing Crucibles

1. To start the balance program, click on the balance icon located on the desktop.
2. Enter LIMS user name and password to access the application. The default is last name for login and password. Click **Accept** for LIMS authentication.
3. The balance verifies connections and then asks for a number of measurements. 1000 is generally a good number (but this value depends on the state of the sea – larger waves/rougher seas may require more counts and vice versa). Make sure both pans are empty and click **OK** to initialize the balances.
4. Tare the balances:
   - Enter 0 g into the ***Reference field***.
   - Click **Tare**.
   - Click **Start Measurement**.
   - Click **Get Mass**. The new Tare value will appear in the *Tare* field on the left side of the screen.
5. Place an empty quartz crucible on the *Unknown* balance.
6. Place an appropriate reference mass (close to the crucible's weight: ~15–20 g) on the *Known* balance.
7. The large crucibles are too heavy for the balance. Use them only as "holders" for the small ones so they do not fall over in the desiccator's rack.
8. Enter the reference mass in the *Reference Mass Field* on the software.
9. Click **Weigh** and then **Start Measurement** to weigh the crucible.
10. Once the measurement is complete click **Get Mass**. Record this value in the Excel spreadsheet.

Weighing Sample

1. Collect ~5 g of sample powder using a spatula and carefully transfer the powder into the quartz crucible.
2. If sample is scarce, a smaller amount can be used; however, error will be larger.
3. Click **Weigh** then **Start Measurement.** Once measurement is complete click **Get Mass.** Add or remove powder and reweigh until a sample of ~5 g is attained (±0.05 g is acceptable). Record the exact sample weight in the Excel spreadsheet.

Use this step until the sample weights saved into LIMS feature is implemented: Save the weight values in the LOI Excel spreadsheet and email it to the chemistry scientists and technicians. At the end of the expedition, give the spreadsheet to the programmers to upload into LIMS.

To be implemented: Save the following sample weights into LIMS:

- pre-ignition mass_container
- pre-ignition mass container + sample
- post-ignition mass_container

4. Put the small crucible in one of the big ones, place a lid on it, and leave it in the desiccator until a set of samples is ready to ignite.

Igniting Samples

Samples are ignited in the Fisher ashing furnace located in the chemistry lab. The entire ignition cycle takes ~12 hr

Material	Ignition Time at °C
Basalts	4 or 5 hr at 1025°C
Si-rich sediments	4 or 6 hr at ~900°C
Samples with: • Muscovite • Biotite • Amphibole • Carbonates	6 hr or more at **XXX**°C

To ignite the samples, follow these steps:

1. Set up the Fisher furnace temperature programming as follows for basalt samples:
   - Ramp at 3°C/min to 900°C and hold at 900°C for 1 hr.
   - Ramp to 1025°C and hold for 4 hr.
2. Put the samples in the furnace before the program starts.
3. Press **Run** twice to start the program.
4. After the program finishes and the crucibles are cool enough (50°–200°C), remove the crucibles with the special handle to pick up the rack. Store the rack in the desiccator until it is time for post-ignition weighing and to make the beads. This should be done as soon as practical so the sample does not rehydrate.

Caution! Crucibles and samples at high temperatures can be very dangerous! They can cause severe burns upon immediate contact with skin, and can ignite combustible and flammable materials.

Note: The Fisher furnace cannot ramp at the default rate of 25°C/min. When the program determines it is at the desired soak temperature, it begins the countdown regardless of whether the programmed maximum temperature was reached.

Post-Ignition Weighing

This step should be done as soon as possible after the samples come out of the furnace and have reached room temperature. Store the samples in a properly-charged dessicator until they are cool enough to weigh. (Hot samples cause inaccurate weighing on any analytical balance.)

1. Reweigh the crucible with the ignited sample in it to determine how much weight the sample gained or lost. Follow the same weighing procedure as in Pre-ignition Weighing.
2. Calculate the LOI values and enter them into LIMS. The formula used to calculate LOI is:

$$\%LOI = 100 \times (\text{weight change during ignition})/(\text{fresh sample weight}).$$

Note: By convention, weight lost during ignition is typically recorded as a positive LOI value, whereas weight gained is recorded as a negative LOI value. Report the results to 2 decimal places. A negative LOI is possible if the sample oxidizes, but this is not the usual case.

Cleaning the Quartz Crucibles

1. Wash the crucibles with DI water and a small piece of a scouring pad (**no soap**).
2. Rinse several times with DI water.
3. Place crucibles in a 10% HNO_3 bath for 12 hr.
4. Rinse 3 times with DI water after the acid bath.
5. Dry the crucibles in the oven at a maximum temperature of 60°C.

Making the Sample Bead

Two processes are required to make a sample bead:

- Weigh ignited sample powder from the LOI and add to a vial containing 400 mg of lithium metaborate flux (typically preweighed onshore). (This step is often completed by the chemistry technicians.)
- Fuse sample powder and flux combination to make a glass bead, which is then dissolved in nitric acid.

The solution created by the second process is diluted and analyzed by ICP.

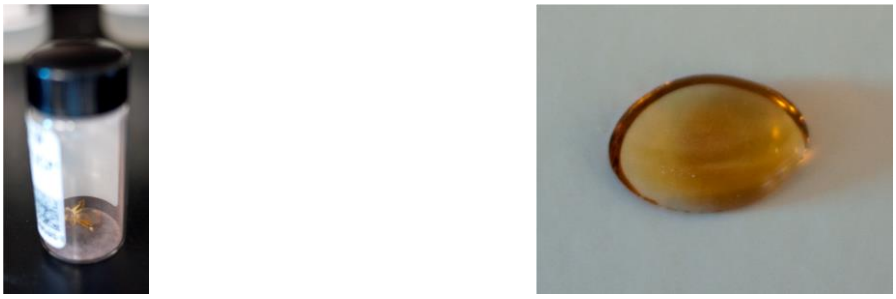

Figure 10. Sample Bead.

Weighing the Sample Bead

Weighing the ignited sample is a fairly critical step. The sample weight should be as close to 100 mg as possible. Inaccuracies in the weight will show up in the analytical results.

1. Clean the countertop around the balance and the balance pans. Cover working surfaces with sheets of white paper.
2. Arrange supplies on the white paper: tweezers, mg calibration weighing set, and weighing paper (2 x 2).
3. Ensure the following items are available and labeled for each sample.
   - 1 bottle of preweighed flux
   - 1 new, empty, acid-washed vial for the remaining ignited powder
4. Pre-label the bottles before weighing (one label each on the cap and the bottle).
5. Fold a small (2 x 2) weighing paper into 4 quadrants and smooth it out again to make a little “cup.” Place it on the *unknown* pan in a stable position and place a 100 mg weight on the paper.
6. Close the door of the balance and tare, then trim after 50–75 counts.
7. Remove a crucible of ignited powder from the desiccator and transfer the sample from the crucible to a clean agate mortar by dumping the powder out gently.
8. Grind the sample in the agate mortar to a fine powder.
9. When the taring and grinding are done, remove the 100 mg weight from the paper on the unknown pan.
10. Transfer a bit of the ground ignited powder onto the weighing paper with a clean spatula (rinsed with isopropyl alcohol).
11. The goal is to replace the 100 mg weight with 100 mg of sample, so the ideal final weight will be zero.
12. Close the door and weigh the sample, putting more sample on or taking it off until you achieve a reproducible weight that is within ±0.00050 g of 0 (half a milligram).

13. When the sample weight is as close to 100 mg as you can get it (i.e., 0.0995–0.1005 g), open the labeled bottle with the preweighed flux in it and **carefully** pick up the paper with the sample powder on it and transfer all of the powder into the bottle containing the flux. Snap the paper a few times with a flick of your index finger to make sure everything goes in.
    a. IMPORTANT! If any of the powder is spilled, the sample must be discarded and reweighed!
14. Homogenize the sample/flux mixture by holding the vial slightly off of vertical and rotating it. Tap it from time to time on the bench top as you rotate it to clear any powder from the sides of the vial. Avoid getting the sample/flux powder stuck around the cap.

Fusing the Sample into a Bead

The most critical aspect making the bead is maintaining a constant sample:flux ratio. A ratio of 1:4 (sample:flux) suffices in most situations. If samples are small (e.g., volcanic glasses), a sample mass <0.1 g may be used, but the same sample:flux ratio must be maintained for the samples and the calibration standards (otherwise the matrix will not match). For example, 0.05 g of sample requires 0.2 g flux.

1. Pour the powder mix into a Pt-Au crucible, once the sample is weighed and homogenized.
2. Pipette 10 µL of 0.172 mM LiBr wetting agent into the sample powder.
3. Fuse at 1050°C for 10–12 min. Heat the sample in two stages:
    - Stage 1 at 700°C for 2 min
    - Stage 2 at 1050°C for 5 min, followed by agitation at 1050°C for 5 min.
4. Sample Bead Maker settings: Fuse 1 = 120 s, Fuse 2 = 300 s, Agit = 300 s.
5. Remove the Pt-Au crucible as soon as the monitor shows no current. With the Pt-tipped tongs, lift out the crucible and swirl the contents to get the entire sample into one bead. **Wear eye and hand protection!**
6. Place the crucible on its cooling seat for at least 3 min. Let the bead cool and solidify in the bottom of the crucible.
7. When cool, pop the bead off the crucible. A sharp "whack" squarely on the hard desktop facilitates release. Do not use a sharp or hard tool to remove bits of the bead stuck to the Pt-Au crucible. Scraping the bottom of the crucible will ruin the crucible and cause more beads to stick.
    - If beads stick, gently use the Teflon/plastic spatula to nudge the bead off.
    - If it still remains, make a note of the sample and let the chemists know which sample may have errors due to the loss of sample material a stuck bead causes.

Cleaning Platinum Crucibles

1. Rinse the Pt-Au crucibles with DI water.
2. If beads are stuck to the bottom, sonicate with DI water for 30 min or more.
3. Place crucibles in HNO_3 10% bath for 12 hr.
4. Rinse with DI water 3 times and place in oven to dry. If the crucibles require polishing, see ***Polishing the Platinum Crucibles***

Polishing the Platinum Crucibles

It may be necessary to polish the crucibles to remove scratches. Do this no more than once per expedition because polishing thins the platinum and in time the crucible will crack. A polishing machine is located in the ICP prep area.

1. Wrap a silk cloth (like the cloth used to clean eyeglasses) around the polishing nozzle.
2. Apply a diamond paste (Grade 30, found in Thin Section Lab) to the front of the silk-covered nozzle and place the crucible over the nozzle.
3. Turn polisher on and polish the crucible bottom for ~30 s (the bottom will be shiny). Be careful because the crucible will get hot. Do not try to remove any deep scratches – the crucibles are not that thick. The least amount of polishing the better.
4. Clean the crucibles with isopropyl alcohol and put in 10% HNO_3 bath for 12 hr.

Using the LOI Furnace to Make Sample Beads

If the bead maker breaks, use the LOI furnace to make beads.

Caution! Safety is a major issue with this procedure; use proper personal protection equipment and note where the nearest fire extinguisher is located.

1. Obtain the following safety equipment:
   - Welder's jacket (orange leather) and welder's gloves
   - Face shield
   - Long pants
   - Steel toed boots
2. Have a designated spotter in the room with you when placing samples in and taking samples out of the oven.
3. Heat ashing furnace to 1020°C.
4. Pour the sample powder mix into a Pt-Au crucible.
5. Pipette 10 µL of 0.172 mM LiBr wetting agent into the sample powder.
6. Place the entire rack of crucibles in the black rack. Six per rack makes for easier access with the tongs when removing samples from the oven.
7. Carefully open the oven door, place the rack of crucibles into the oven, and close the door.
8. Wait until the temperature returns to 1020°C, then soak the samples for 5 min.
9. Remove each sample crucible with the long tongs. When the sample is removed, turn the crucible 45° to one side and then back to the other side to mix the sample as much as possible.
10. Place the sample crucible in a second black rack on the counter to cool. These crucibles are very hot and may burn anything near or under them. Take appropriate precautions.

Cleaning the Plasticware

- Wash the bottles and vials used for rock analyses in 10% reagent grade HNO_3.
- Soak plasticware a minimum of 12 hr in each of two baths.
- Triple-rinse the bottles/vials in DI.
- Oven dry at 40°C.

Standards

ICP-AES is a comparative analytical technique, in that the machine's response (measured in "counts") needs to be calibrated against standards in which the concentrations of the various elements are known. For rock analysis, calibration is best achieved through comparison to internationally recognized and approved Standard Reference Materials (SRMs).

Note: Every ICP-AES run must have a set of standards run along with the unknowns.

Types of Standards

For basalts, robust and linear calibrations can be achieved with a blank and five SRMs:

- DNC-1
- BIR-1
- BHVO-2
- W-2
- BCR-2

Additional well-characterized rocks (although not formal SRMs) to check the results include K1919 and BAS140.

Note: Because SRMs are required for each run, they tend to get consumed rapidly. In the interest of conservation, if enough calibration solutions are left over from a previous run, and if these solutions have been prepared identically, then it is appropriate to reuse those SRM solutions until they are depleted.