



Environmental Analysis Program

Pomona College
Biogeochemistry Laboratory

SOP-L205

X-Ray Fluorescence (XRF) Sample Preparation and Analysis

Standard Operating Procedure

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1. Purpose

This SOP describes the complete workflow for preparing geological rock samples and analyzing their major-element oxide composition by wavelength-dispersive X-ray fluorescence (WD-XRF). The procedure covers sample powdering, flux fusion bead preparation, bead polishing, and instrument operation on the PANalytical XRF spectrometer using the SuperQ software with the “Geology Beads” application (Claisse, 2003; Norrish & Hutton, 1969; PANalytical, 2012).

2. Scope

- **Applies to:** Rock, mineral, and geological samples analyzed as lithium tetraborate fusion beads for major-element oxide determination (e.g., SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , MgO , Na_2O , K_2O , TiO_2 , P_2O_5 , MnO)
- **Does not apply to:** Pressed-pellet trace-element analysis, handheld (portable) XRF measurements, or non-geological materials
- **Locations:** Rock preparation lab (powdering and polishing); analytical lab / wet lab (weighing, fusion); XRF lab (instrument operation)

3. Responsibilities

Role	Responsibility
Lab PI	Approve methods, ensure training compliance
Lab Technician	Maintain instrument, assist with troubleshooting
Trained Personnel	Follow this SOP, maintain sample tracking, clean up
New Students	Complete training before independent use

Contact the lab technician, Jade Star, or Bob with any questions.

4. Definitions

- **Certified reference material (CRM):** A geological standard with certified major-element oxide concentrations from an accredited producer (e.g., NIST, USGS, BCR); used to verify XRF accuracy during each analytical session (Norrish & Hutton, 1969).

- **Detection limit (MDL):** The lowest concentration of an element reliably detected above background; varies by element, matrix, and instrument settings; typically 1–50 mg/kg for common elements by WD-XRF.
- **Flux (dilithium tetraborate, $\text{Li}_2\text{B}_4\text{O}_7$):** A reagent mixed with the rock powder in a 2:1 (flux:sample) ratio; lowers the melting point and produces a homogeneous glass bead that eliminates mineralogical matrix effects during analysis (Claisse, 2003).
- **Fluorescence (X-ray):** The emission of characteristic X-rays from a sample excited by primary X-rays; each element emits at unique energies that identify and quantify it (Norrish & Hutton, 1969).
- **Fundamental parameters (FP) method:** An XRF calibration approach that uses known physical constants to calculate element concentrations from measured fluorescence intensities without matrix-matched standards for every sample type.
- **Fusion bead:** A glass disc produced by melting the powdered rock sample with lithium tetraborate flux at 1000 °C; the homogeneous glass eliminates mineralogical matrix effects (Claisse, 2003).
- **Geology Beads:** The SuperQ application/archive configured for major-element oxide analysis of fusion beads on the PANalytical XRF spectrometer (PANalytical, 2012).
- **K-alpha and K-beta lines:** The most commonly measured characteristic X-ray emission lines for elements; produced when an electron is ejected from the K shell and replaced from the L or M shell, respectively.
- **Loss on ignition (LOI):** The mass percentage lost when a pre-weighed rock powder is heated to 1000 °C; reflects H_2O , CO_2 , and organic matter content; used as a correction in major-element summation (Norrish & Hutton, 1969).
- **Matrix effects:** Absorption and enhancement of X-ray signals by other elements in the sample; minimized by the flux fusion method (Claisse, 2003).
- **Shatterbox:** A vibratory ring-and-puck mill used to grind rock chips to a fine powder suitable for fusion.
- **SuperQ:** The PANalytical software used to control the XRF spectrometer, define measurement sequences, and retrieve results (PANalytical, 2012).
- **WD-XRF (wavelength-dispersive XRF):** An XRF configuration in which characteristic X-rays are separated by a crystal monochromator before detection; provides higher spectral resolution and lower detection limits than energy-dispersive (ED-XRF) systems.
- **X-ray fluorescence (XRF):** A non-destructive elemental analysis technique in which a sample excited by a primary X-ray beam emits characteristic fluorescent X-rays that are measured to determine elemental composition (Norrish & Hutton, 1969).

5. Safety and Hazards



Hazards

- **High-temperature furnace:** The muffle furnace operates at 1000 °C. Crucibles and tiles removed from the furnace are extremely hot. Do not touch them, even with gloves, until fully cooled.
- **Compressed air:** Loud; wear ear protection when blowing out equipment.
- **Rock chipper:** Flying rock fragments are common. Always wear safety glasses. Never look into the chipper while it is running.

- **Shatterbox:** The unit vibrates intensely and can travel across the floor. Get help repositioning it if needed.
- **Chemical reagents:** Dilithium tetraborate flux is a fine powder; avoid inhalation. Ethanol is flammable.
- **X-ray radiation:** The XRF instrument contains an X-ray source. Follow all posted radiation safety procedures. Do not open the instrument lid while the arm is moving or the flap is up.

5.1. Required PPE

- ☐ Safety glasses (required in the rock prep lab at all times)
- ☐ Laboratory coat
- ☐ Nitrile gloves (powder handling and polishing)
- ☐ Ear protection (when using the shatterbox or compressed air)
- ☐ Heat-resistant gloves (when handling crucibles or the tile from the furnace)

5.2. Emergency Procedures

Do not work in the lab without letting someone know your plans and estimated return time. Phones and safety showers are in all labs. Refer to [SOP-L002 Laboratory Safety](#) for all emergency procedures.

6. Prerequisites and Required Reading

- [SOP-L002 Laboratory Safety](#)
- Furnace operating instructions (posted above the furnace)
- This SOP in full

! Important

Minimize contamination at every step. Wear gloves, clean equipment thoroughly between samples, and assume any shared equipment is dirty unless you just cleaned it. Leave the lab and equipment cleaner than you found it.

7. Required Materials

7.1. Equipment

Item	Location / Notes
Small rock chipper	Rock prep lab
Rocklabs shatterbox (tungsten carbide head)	Rock prep lab

Item	Location / Notes
Analytical balance	Analytical lab, corner by XRD room
Vortex mixer	Counter left of weigh station
Deionizing bar	Right of the Vortex mixer
Muffle furnace (1000 °C)	Far left corner, wet lab
Grinding/polishing laps (4 stages)	Darkroom / grinding lab
Ultrasonic cleaner (sonicator)	Grinding lab or XRF lab
Diamond-tipped pen or rotary tool	Darkroom
PANalytical XRF spectrometer	XRF lab (PANalytical, 2012)

7.2. Consumables

Item	Notes
Dilithium tetraborate flux	Stored in desiccator, left of weigh station
Graphite crucibles (9 per batch)	Drawer under the furnace
Plastic cups with lids	Cabinet left of weigh station
Brown butcher paper	Shelves, back wall, rock prep lab
White printer paper	For transferring powder
Kimwipes	General use
Ethanol	Bottle on shatterbox table; bulk jug on side counter
Sand (for cleaning shatterbox)	Bowl in center of table, rock prep lab
Green Brillo pads	Drawer, rock prep lab
Sample bags and labels	Drawer under counter, XRF lab
XRF sample sheets	Drawer left of weigh station
SiC sandpaper	For removing saw marks

8. Procedure

8.1. Step 1: Powdering the Sample

A complete fusion batch is 9 samples. If you plan fusions, turn on the furnace first (see §6.3) since it takes about 1 hour to reach 1000 °C.

8.1.1. Chipping

1. Use the rock chipper to reduce each sample to pieces smaller than approximately 1 cm. Wear safety glasses. Clean the chipper jaws thoroughly between samples with compressed air and an ethanol wipe.
2. Weigh out approximately 20–30 g of chipped material into a labeled sample bag.

8.1.2. Shatterbox Milling

3. Place a piece of brown butcher paper under the shatterbox head assembly to catch any spillage.
4. Wipe all shatterbox bowl components (ring, puck, and bowl) with an ethanol-dampened Kimwipe. Allow to dry completely.
5. Load the chipped sample into the tungsten carbide bowl. Do not overfill (maximum ~two-thirds full).
6. Lock the lid and run the shatterbox for 60–90 seconds. Longer milling times reduce particle size further.
7. Unlock the lid and carefully transfer the powder onto white printer paper, keeping all components on the paper edge so exterior metal surfaces do not contact the sample. Funnel the powder into the labeled container. Use a fresh sheet of paper for every sample.

8.1.3. Cleaning the Shatterbox

8. If powder is stuck, scrub with a green Brillo pad. If that is insufficient, run a scoop of sand for 15 seconds and discard.
9. Detail all pieces with compressed air (hold pieces in the airflow; scrub with the Brillo pad). **Do not let the metal hose tip touch any bowl surface.** Use ear protection.
10. Finish with an ethanol wipe on a Kimwipe. After the last sample, clean up, recycle paper, replace the bowl, and turn off the shatterbox.

8.2. Step 2: Weighing and Mixing

1. Bring your powdered samples to the analytical lab. Get an XRF sample sheet from the drawer left of the weigh station, fill out the top portion, and keep it on a wooden tray next to the balance.
2. Retrieve a small cup of flux from the desiccator.
3. For each sample:
 - a. Label a plastic cup lid with the sample number.
 - b. Place the cup (no lid) in the cardboard holder on the balance, close the sliding door, and zero.
 - c. Weigh 3.5000 g (± 0.0004 g) of sample powder. Record the exact mass. Zero the balance.
 - d. Add 7.0000 g (± 0.0004 g) of flux (2:1 flux-to-sample ratio) (Claisse, 2003). Record the mass. If you add too much flux, remove the excess but **do not return it to the reagent container.**
 - e. Seal the lid tightly.
4. Mix each cup on the Vortex mixer for about one minute. If powder clings to the sides, pass the deionizing bar over the cup.

8.3. Step 3: First Fusion

8.3.1. Furnace Startup

1. Flip the black switch on the front of the furnace.

2. Press the T1 temperature button and confirm $T = 1000\text{ }^{\circ}\text{C}$.
3. Set a duration (e.g., 24, 48, or 72 hours). Press Enter and Start. It takes roughly 1 hour to reach $1000\text{ }^{\circ}\text{C}$.

Caution

Do not spill glass in the furnace or introduce liquids. Do not use this furnace for temperatures below $500\text{ }^{\circ}\text{C}$ – use a regular drying oven instead. Do not shut off the furnace mid-cycle with the power button; let it run out its programmed time and shut off automatically.

8.3.2. Loading Crucibles

4. Select 9 graphite crucibles from the drawer under the furnace. Clean each inside by gently wiping with a Kimwipe and blowing out with compressed air.
5. Back at the analytical lab, carefully empty each cup into the corresponding crucible. **Maintaining the correct order is critical – once the powder is in the crucible, there is no way to tell samples apart.**
6. Place the empty cups (lids on) in the box labeled “Dirty Cups for Blowing Out.”

8.3.3. Fusing

7. In the wet lab, place a tile on the brick stand (small hole facing toward you) and crank the stand up until the tile is about halfway up the side of the furnace.
8. Put on both long heat-resistant gloves. Using the tongs, slide the tile into the furnace. Close the door as quickly as possible.
9. Wait 20–30 minutes for the furnace to recover to $1000\text{ }^{\circ}\text{C}$, then leave the samples for 10–15 minutes. Longer fusion times help carbon-rich samples degas fully.

The tile and crucibles are extremely hot when removed from the furnace. Do not touch them, even with gloves, until fully cooled.

10. Wearing gloves, open the furnace and carefully pull the tile out using the tongs and the tile hole. Move the tile to the hot plate and close the furnace.

8.4. Step 4: Regrinding and Second Fusion

1. Once the crucibles are cool enough to handle, pour the first-fusion glass back into the shatterbox bowl. Re-grind for 60 seconds.
2. Return the powder to the crucible and re-fuse at $1000\text{ }^{\circ}\text{C}$ for 15–20 minutes to remove any remaining bubbles and produce a homogeneous bead.
3. Once cooled, inspect each bead visually. Beads should be clear, bubble-free glass covering the full crucible bottom. If a bead has a dark carbon pellet at the bottom, fuse for an additional 15 minutes.

8.5. Step 5: Polishing the Bead

Polish the flat (analytical) surface of each bead on the 4-stage grinding/polishing laps to remove surface scratches and contamination that would scatter X-rays.

1. Use the coarsest lap first (e.g., 600 grit SiC), working through progressively finer stages.
2. After each stage, clean the bead in the ultrasonic cleaner with distilled water (3 minutes).
3. After the final lap, inspect the flat surface under the lab light; it must be mirror-smooth with no visible scratches.
4. Label each bead on the curved (non-analytical) side using a diamond-tipped pen or rotary tool.

8.6. Step 6: XRF Analysis

1. Open a “Measure and Analyze” window from the SuperQ Manager. Wear gloves and avoid touching the flat (analytical) surface of the bead (PANalytical, 2012).
2. Click the multi-cup icon to open the Sample Changer. Double-click position A1. Enter the sample name; set type to “Routine,” application to “Geology Beads,” priority to “Normal,” and verify weights are correct. Click **Add**. Available positions are A1–A8 and B5–B8.
3. Once all samples are entered, click **Measure**.

Note

If the XRF has not been running recently, the instrument needs to warm up until the front-panel readouts display 60 and 50. Run the first bead until those values are reached, then click Abort. Wait for the arm to return the sample, then restart. Running samples before warm-up can compromise the analysis (PANalytical, 2012).

8.7. Step 7: Retrieving Results

1. Open a “Results Evaluation” window. Set the archive to “Geology Beads” and adjust the date range. Use the “Name Mask” field to filter results. Click on a sample and then click “View.”
2. To export data: open a “Results Quantitative” window. Adjust date and name mask. Click the Print icon, then “Delimited Values Report” to clipboard. Paste into a spreadsheet.

9. Quality Control

- Verify the 2:1 flux-to-sample ratio for every sample; record exact masses on the XRF sample sheet (Claisse, 2003).
- Visually inspect beads before loading; reject cracked, broken, or bubble-compromised beads.
- Run at least one CRM alongside unknowns to monitor instrument drift and accuracy (Norrish & Hutton, 1969).
- If a bead required a third fusion, note this on the sample sheet.

10. Cleanup Checklist

- ☐ All paper recycled (butcher paper, white paper)
- ☐ Shatterbox bowl cleaned and replaced; shatterbox off
- ☐ Chipper cleaned (ethanol wipe on all contact surfaces)
- ☐ Crucibles returned to the drawer (or discarded if degraded)
- ☐ Plastic cups blown out and restocked (or discarded after 6 uses)
- ☐ Polishing laps spun dry
- ☐ Sonicator drained if not in regular use
- ☐ XRF sample sheet completed and filed
- ☐ All personal items removed from the lab
- ☐ Work surfaces wiped down

11. Troubleshooting

Symptom	Possible Cause / Action
Glass does not cover crucible bottom	Insufficient fusion time; re-fuse longer
Bead cracked or has deep air bubbles	Air trapped during fusion; re-fuse briefly
Carbon pellet at bottom of bead	Carbon not fully degassed; fuse longer next time
Powder clings to plastic cup walls	Pass the deionizing bar over the cup
XRF front-panel numbers not at 60/50	Instrument not warmed up; run a bead until ready
XRF data appear anomalous	Sample run before warm-up; re-run

12. References

- Norrish & Hutton (1969)
- Claisse (2003)
- PANalytical (2012)

Claisse, F. (2003). *Fusion and fluxes for XRF sample preparation*. Corporation Scientifique Claisse.

Norrish, K., & Hutton, J. T. (1969). An accurate X-ray spectrographic method for the analysis of a wide range of geological samples. *Geochimica Et Cosmochimica Acta*, 33(4), 431–453. [https://doi.org/10.1016/0016-7037\(69\)90126-2](https://doi.org/10.1016/0016-7037(69)90126-2)

PANalytical. (2012). *SuperQ software: User manual*.

13. Related SOPs

SOP	Title	Relationship
L002	Laboratory Safety	Required safety training
L111	Muffle Furnace	Furnace operation details

14. Training Record

Before performing XRF sample preparation and analysis independently, personnel must:

1. Read this SOP in full
2. Complete [SOP-L002 Laboratory Safety](#) training
3. Review furnace safety instructions posted above the furnace
4. Observe a trained operator perform a complete batch (powdering through analysis)
5. Prepare and analyze a batch under supervision with acceptable CRM recovery
6. Be signed off by the lab technician or PI