

XRD Sample Preparation Hard Rock

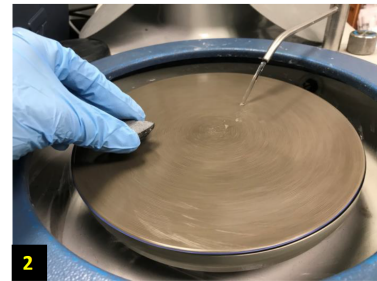
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Before being crushed in the X-Press, hard rock samples for ICP analysis are polished (*Figure 1.1 and 1.2*) and cleaned in a sonic bath at least three times (for 10-15 minutes each): the first round of cleaning is with isopropyl alcohol and the following rounds are with DI water (*Figure 1.3*, see [ICP Sample Preparation](#) for further details). Hard rock samples for XRD analysis do not usually need to be polished before being crushed.



Buehler Grinder Polisher

1. Clean the diamond disc with a dressing stone



2. Polish all sample faces and edges



Branson Sonic Bath



3. Sonicate the samples with isopropyl alcohol inside or DI water for 15-20 minutes



4. Cover the beakers with Parafilm after drying oven at 110°C for about 12 hours

Figure 1. Polishing and cleaning hard rock samples

Crushing Samples in the X-Press

The X-Press is a motorized hydraulic press that crushes hard rock and highly compacted sediment samples into smaller pieces.

Preparing the X-Press area

1. First, clean the X-Press and counter with simple green and isopropyl alcohol. Lay down a Kimwipe or sheet of paper on the counter.
2. Next collect the following supplies that make up the 'crushing unit' of the X-press (*Figure 2*). Materials are located in the drawer labeled 'X-PRESS SUPPLIES' in the X-Ray Prep Area in the Thin Section Lab.

- Weigh Paper 6" x 6"
- Piece of core liner
- Two Delrin discs
- Stainless steel base
- Aluminum Die



Figure 2. Materials needed for assembly of 'crushing unit'. A. Weigh Paper 6"x6". B. Core Liner C. Two Delrin Discs D. Stainless Steel Base E. Aluminum Die

3. Put on gloves and clean the components with isopropyl alcohol before setting them down on the clean surface.
4. Once cool enough to handle, collect sample beakers from the desiccator located in the X-Ray Lab. Put a piece of Parafilm® over each beaker and bring them over to the X-Press (Figure 1.4).

Assembling the crushing unit

Now that the X-Press area is clean and the samples are in the Lab, assemble the crushing unit as follows:

1. Grab the stainless steel dish. This is the base for the crushing unit. Place a piece of Weigh Paper on the base (Figure 3.1).
2. Put one Delrin disc on top of the weigh paper (Figure 3.2).
3. Place the sample on top of the Delrin disc (Figure 3.3). These discs can fracture leaving Teflon flakes in the sample so arrange the sample such that the two flattest surfaces are the top and bottom.
4. Now place the second Delrin disc on top of the sample (Figure 3.4). Again make sure the disc rests flat against sample.
5. Put the aluminum die on top of the Delrin disc, holding it until you slip the core liner over the unit (Figure 3.5).
6. Now slip the piece of core liner over all the pieces and resting inside the stainless steel base (Figure 3.6). This contains the sample pieces inside the unit.

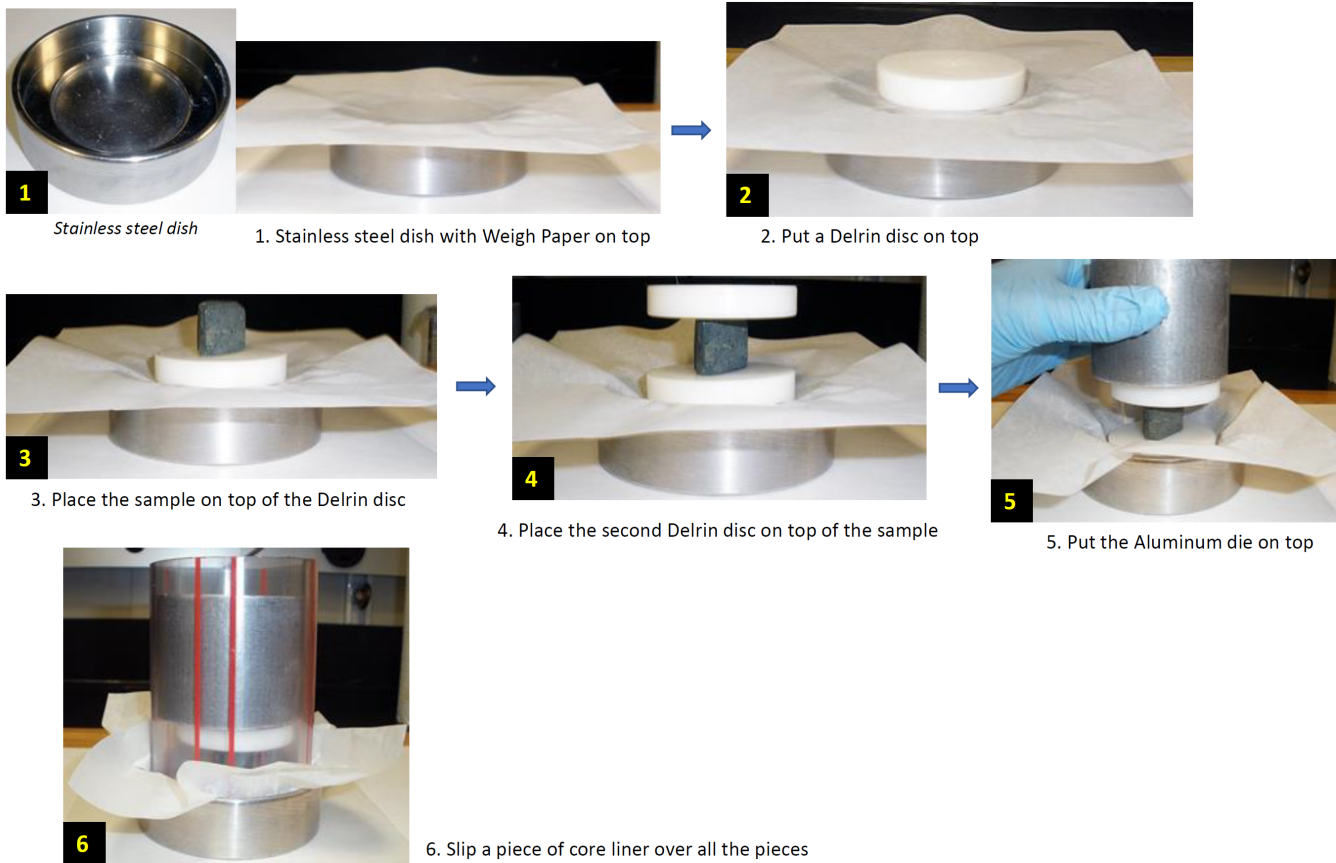


Figure 3. Assembling the crushing unit

The crushing unit is now assembled and we can start crushing samples with the X-Press (Figure 4A).

Crushing Samples

Safety Note: Always wear safety glasses and hearing protection. Do not stand directly in front of the X-Press while it is operating. Never leave the X-Press unattended while using. Do NOT take the instrument above 10 tons of pressure.

Place the crushing unit inside the X-Press (Figure 4B) in the middle of the metal platform (Figure 4A, arrow A). Slide the polycarbonate door down in the vertical indents (Figure 4C). **Note:** The polycarbonate door sits on two interlock switches that enable operation. If the door is not fully closed or pressing down on these switches the machine will not work. Tighten the jackscrew (Figure 4A, arrow B) until it rests firmly against the aluminum die. Tighten the pressure relief handle until just tight with a clockwise turn (Figure 4A, arrow C).

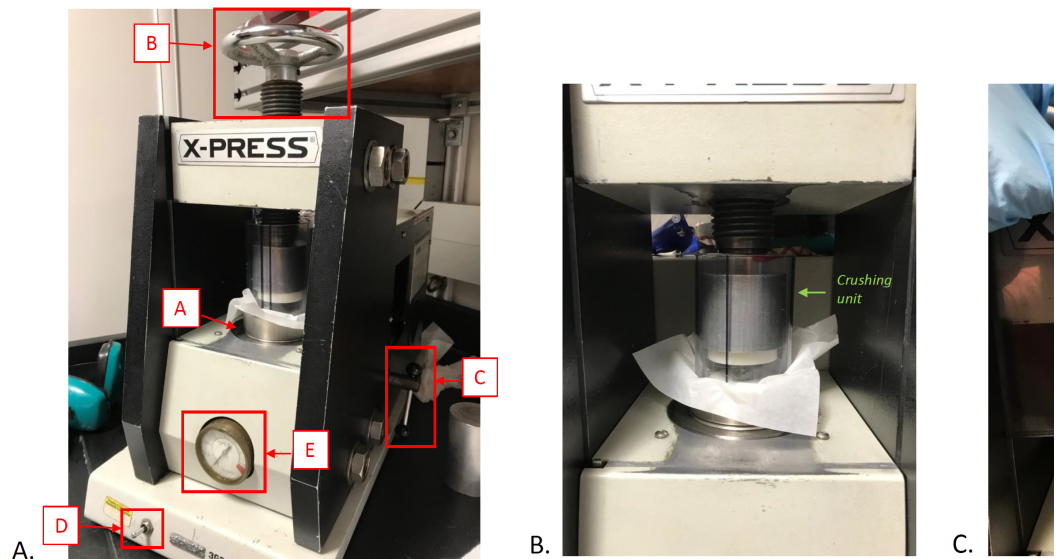


Figure 4. A. Overview of the X-Press. A. Metal platform sample rests on B. Jackscrew C. Pressure Relief Handle. D. 'On' toggle E. Pressure Gauge. B. Crushing unit inside the X-Press. C. Polycarbonate door

Crush the sample by holding down the toggle switch (Figure 4A, arrow D). The motor and pump can be heard and the pressure will rise (Figure 4A, arrow E). When the toggle is released, the sample will sit under that pressure. **Do NOT leave a sample under pressure** for any reason. For most samples ~5 tons of pressure is enough force to crack it. If you find the need to go near 10 tons, try rotating the sample onto another side and repeat the process again. Taking the pressure up greater than 10 tons can cause the discs and the core liner to shatter, as well as contaminating the samples and creating a safety hazard.

After the sample has cracked, release the toggle switch. To remove the unit, loosen the pressure relief handle (Figure 4A, arrow C) by rotating it counter clock wise and hold down the toggle switch. The pressure gauge should read zero and the metal platform will lower down. **Do not leave the platform up, always make sure to lower it back down after crushing a sample.** When the platform is level with the surface let go of the toggle switch and unscrew the jackscrew. Then slide the polycarbonate door up and remove the unit. The pieces can be poured into a labeled bottle that will eventually hold the finely ground powder. From here the pieces will then be put into the Shatterbox vessels.

If pieces are still too large then repeat the same setup and crush it again. Look out for and remove any pieces of the Delrin Discs that may have chipped off and gotten into the sample. Leaving pieces of the Delrin Discs in the sample will cause contamination.

It may happen that the metal platform does not lower down completely after the pressure is released. In that case, remove the crushing unit and replace it by the aluminum cylinder (Figure 5). Leave the pressure relief handle loose. Tighten the aluminum cylinder with the jackscrew. Slide the door in place and press down the toggle switch. Rotate the jackscrew to keep the aluminum cylinder tightened as pressure increases and the platform lowers down. When the platform is level with the surface, let go of the toggle and unscrew the jackscrew. Remove the aluminum cylinder and proceed to the next sample.

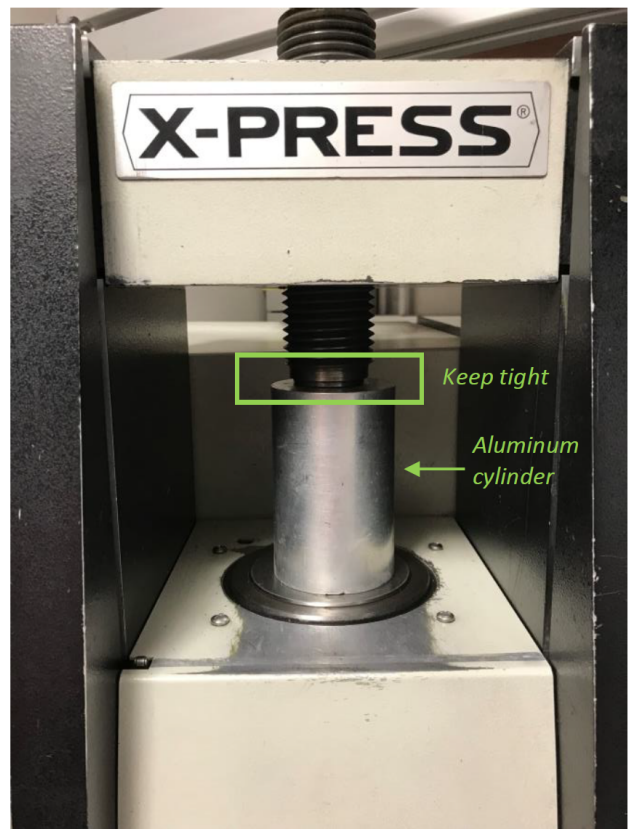
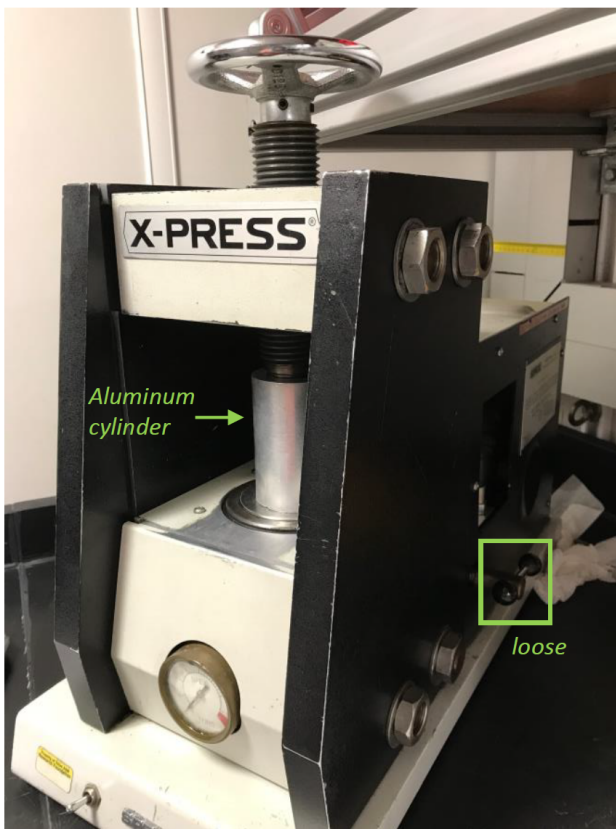


Figure 5. How to lower down the metal platform.

Grinding Samples in the Shatterbox

The shatterbox takes the crushed pieces from the X-Press and grinds them into a very fine powder. The Spex shatterbox is capable of grinding three standard size samples or one large sample. We use tungsten carbide grinding vessels.

Apparatus and Materials

- Shatterbox
- Tungsten Carbide Vessels: Vessel, Puck, and Lid
- Samples
- 1oz Sample Vials
- Sample Labels
- Teflon spatula
- Weigh paper 6" x 6"

Turn on the Shatterbox by flipping the 'On' switch located on the back panel above the power cable (Figure 6A, arrow A). The control panel (Figure 6B) is located on the front of the lid (Figure 6A, arrow B) next to the handle.

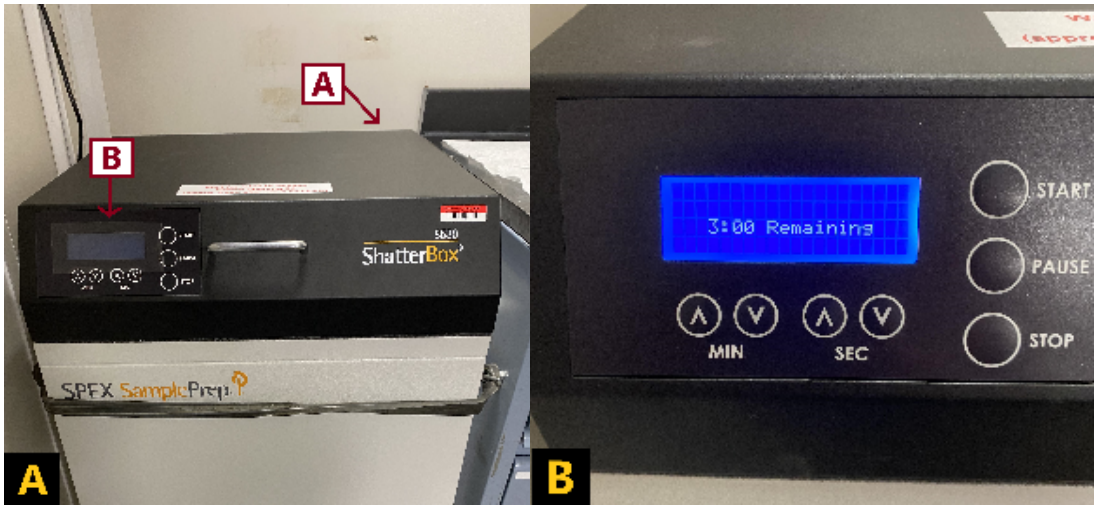


Figure 6. A. Shatterbox. A. Power switch. B. Control panel. B. Control Panel

There are two sizes of grinding vessels: small and large. Each size has different components and requires a different setup inside the Shatterbox. The small vessel holds between 5-20 mL of material and has three components: container, puck and lid (Figure 7). The small vessels are usually used for grinding samples for ICP analysis.

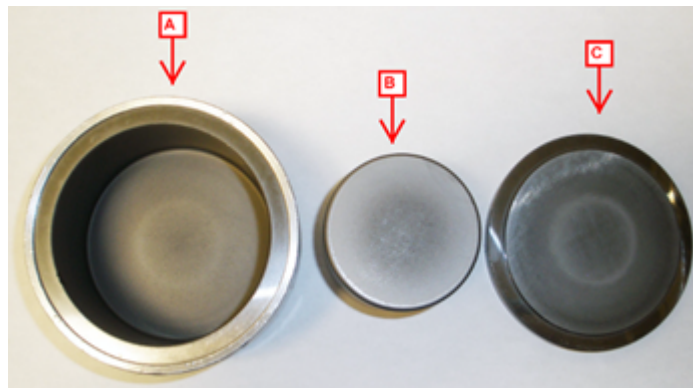


Figure 7. Small vessel components. A. Container B. Puck. C. Lid

The large grinding vessel holds between 20 – 60 mL of material and has five components: a container, puck, inner ring, O-Ring and lid (Figure 8).



Figure 8. Large Vessel components. A. Container B. Inner Ring C. Puck D. O-Ring E. Lid

The small vessels have a small indent in the bottom of the container and they will sit in the shatterbox resting on either a three-pinned rack plate (Figure 9) or a one-pinned rack plate (Figure 10). The three-pinned rack plate is usually used.



Figure 9. Three-pinned rack plate to hold three small vessels in the Shatterbox



Figure 10. One-pinned rack plate to hold one small vessel in the Shatterbox

The three-pinned rack plate will hold three grinding vessels while the one-pinned rack plate will only hold one grinding vessel.

The large grinding vessel will sit directly in the shatterbox without an additional plate below it.

Loading the Shatterbox

Transfer the sample pieces into the grinding vessel. Pour sample pieces between the puck and the wall of the vessel (Figure 11). **There can't be any material on top of the puck or on top of the walls of the container** (the area the lid sits on); otherwise the vessel will not seal properly and the sample can spill inside the shatterbox. If any pieces are on top of the puck or ring, use tweezers or a Kimwipe to move the sample into the vessel. Put the lid on and place in the shatterbox.

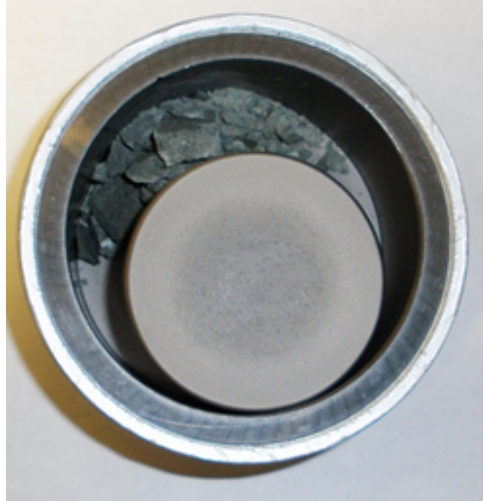


Figure 11. Small vessel filled with sample pieces. No sample material is on the top the puck or in the lid ring.

Open the lid, pull out the lever arm (Figure 12, arrow A), and pull up the clamp arm (Figure 12, arrow B). This will allow full access to the inner platform (Figure 12, arrow C).

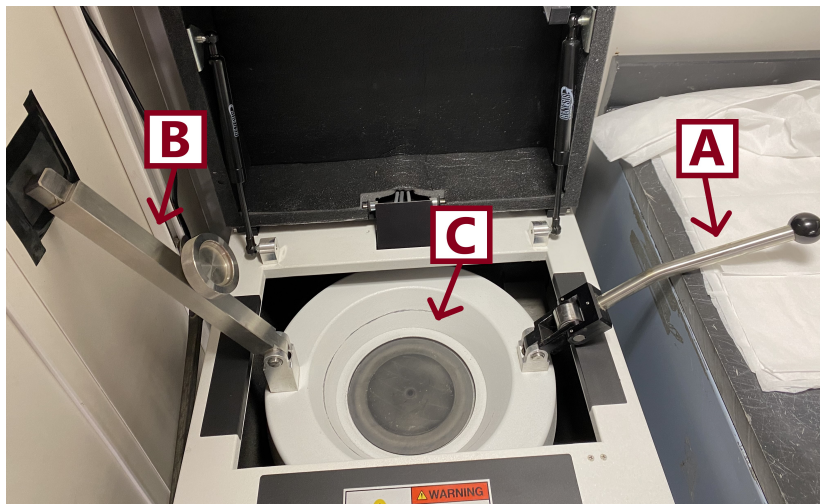


Figure 12. The inside of the Shatterbox. A. The lever arm B. The clamp arm C. The inner platform

Depending on vessel size, you will either put in the one- or three- pinned rack plate (small vessels) or the single large grinding vessel. The shatterbox setup will also vary depending on sample number. If you are crushing one small sample, use the one-pinned rack plate with one small grinding vessel, whereas for two or three samples use the three-pinned rack plate (Figure 13). For crushing two samples, two vessels will be full with sample, whereas the third container can have quartz sand added to it. It is important to maintain balance within the machine to prevent damage.

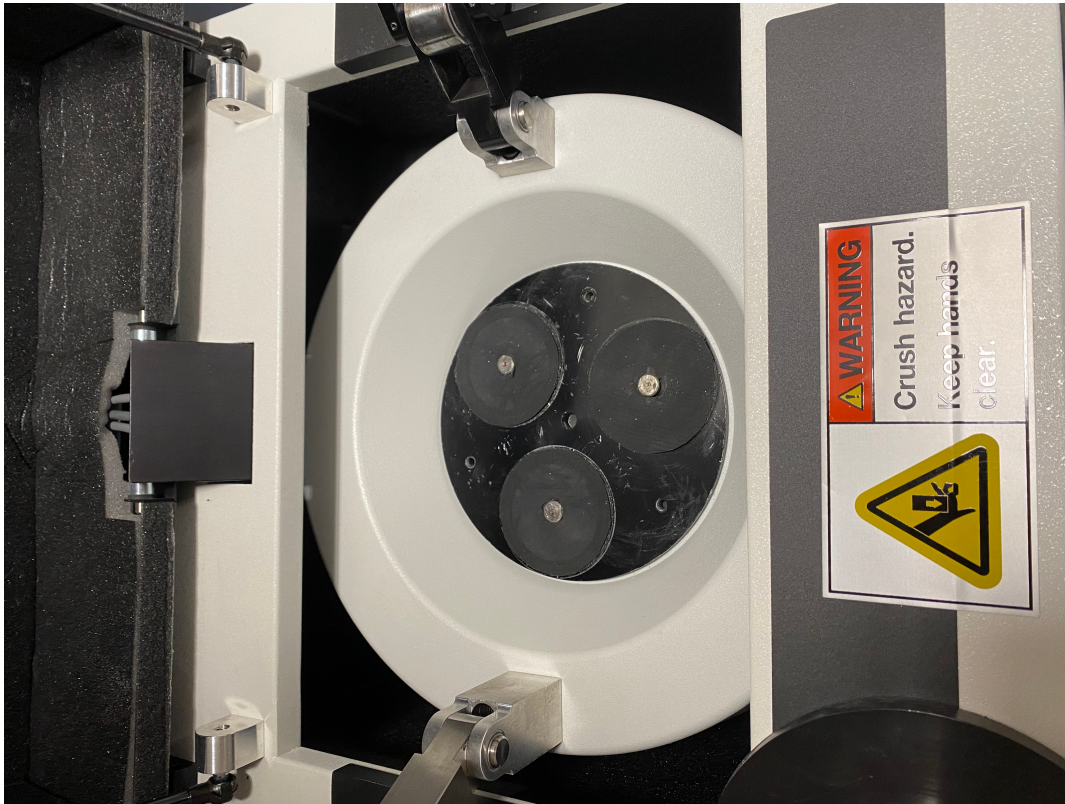


Figure 13. Inside the Shatterbox with the bottom three-pinned rack plate resting inside the inner capsule.

Now load vessels onto the rack plate (Figure 14). The divet in the bottom of the vessels will settle onto the pins and fit firmly in place.

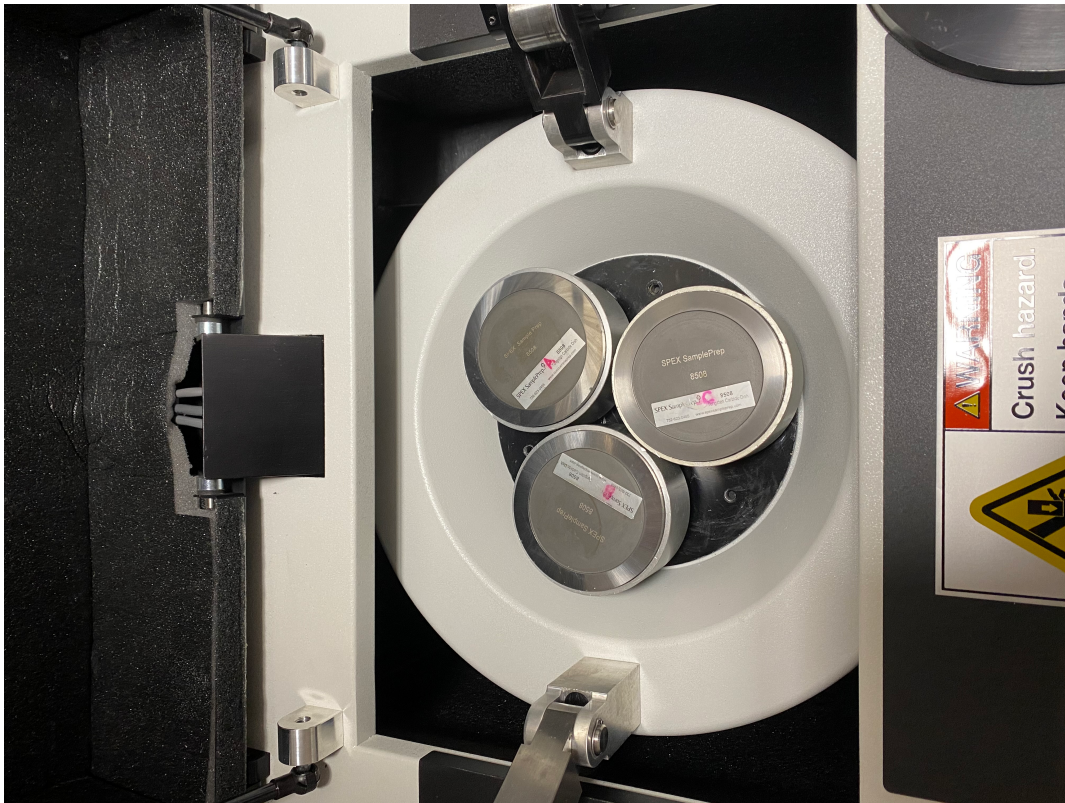


Figure 14. Three samples loaded into the Shatterbox.

Put the top plate over the vessels. Bring down the clamp arm (Figure 15, arrow A). The wheel guide on the clamp arm will settle onto the boss (Figure 15, arrow B) when centered properly.

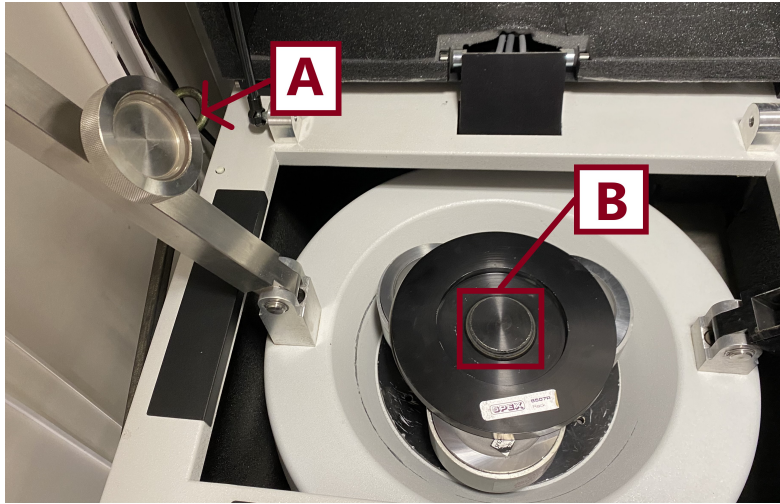


Figure 15. The top plate sitting on top of the three samples. A. Clamp arm. B. the boss of the top plate, where the clamp arm will attach.

Bring the lever arm over the end of the clamp arm (Figure 16A, arrow, 16B). Then move the lever arm from right to left over the clamp arm (Figure 16C) and all the way horizontal.

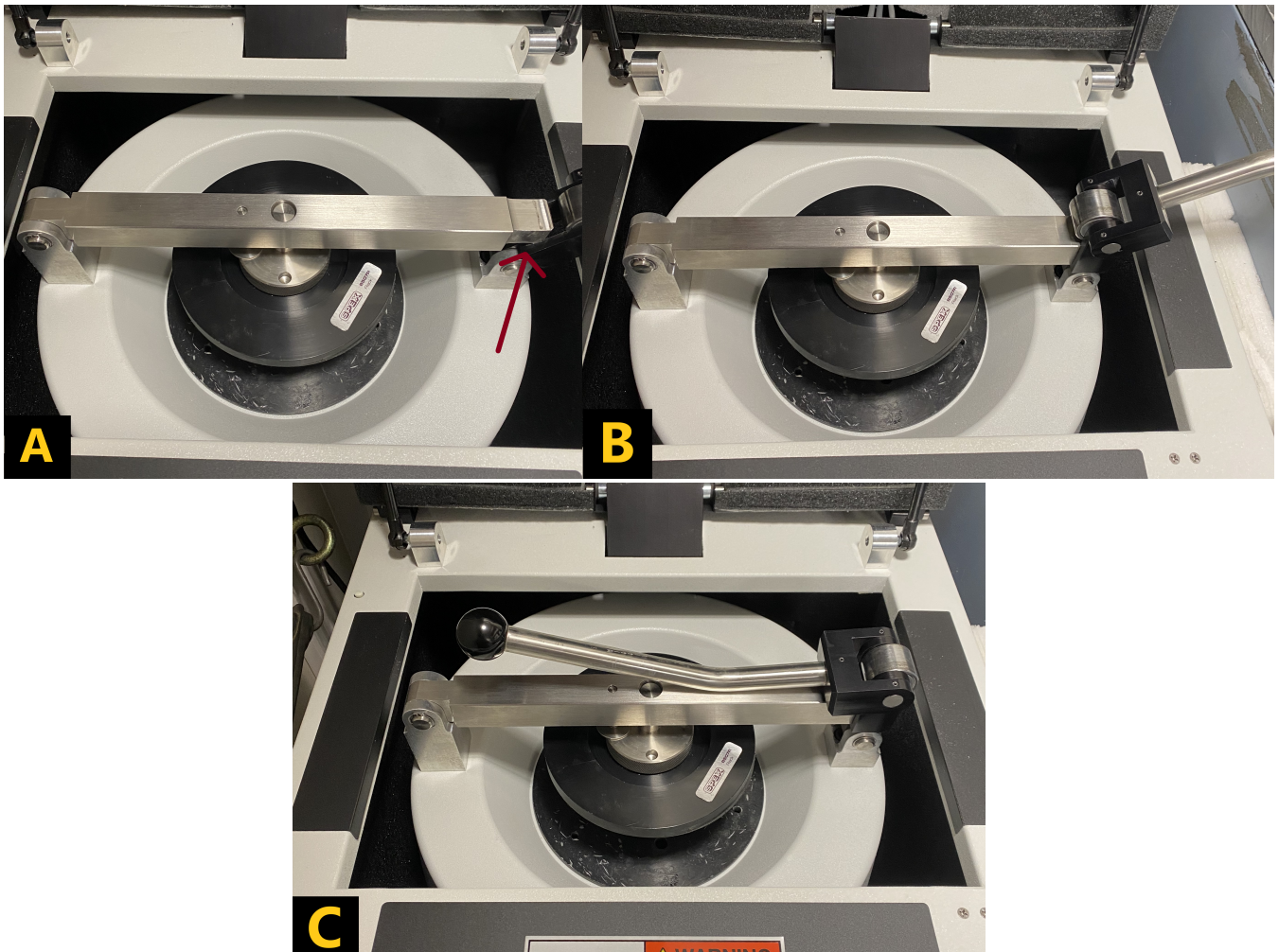


Figure 16. A. The clamp arm inserted into the end of the lever arm. B. The lever arm pushed over the end of the clamp arm. C. The lever arm in its final position over the clamp arm.

The resistance in the lever arm is very important and must be adjusted before each use. There should be moderate resistance in the lever arm while pushing it over and down onto the clamp arm. If the resistance is too low the containers can shake free; whereas, if it's too strong the clamp can break. Ideal tightness is just past the point where the vessels can be rotated while the clamp is down. Adjust the resistance by raising the clamp arm and pushing on the 'locking pin' on the clamp arm (Figure 17, box). Hold the locking pin up and turn the wheel guide (Figure 17, double headed arrow) to loosen or tighten the pressure of the clamp on the containers. Rotating the guide clockwise tightens; whereas counterclockwise loosens.

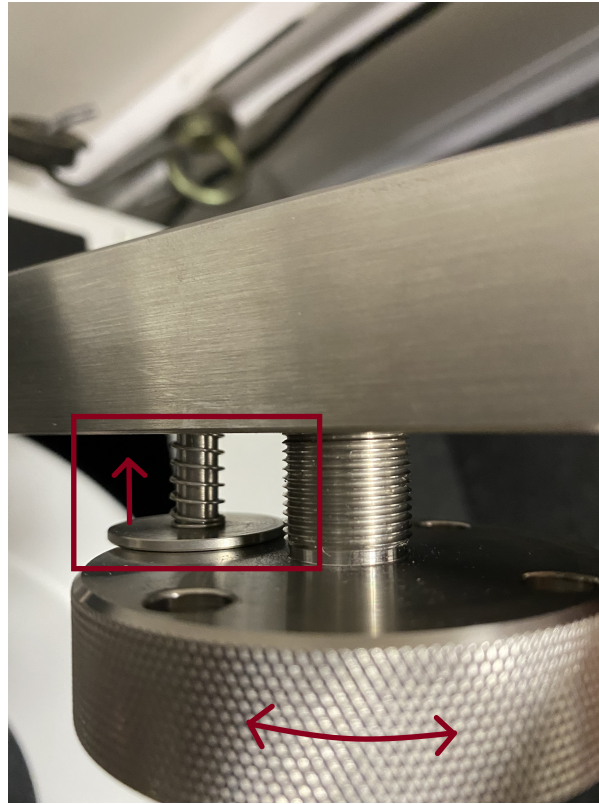


Figure 17. The lever arm and the wheel guide.

Now close the lid and set the grinding time. The LCD screen displays the current operating time (Figure 6B), the default of 3:00 will return every time the machine is turned on at the back. Adjust the time by pressing on the 'Min' and 'Sec' buttons. Seconds will change in increments of 10. The timer maximum is 10:00 and the timer counts down in increments of 5.

When the time is set press the 'Start' button (Figure 6B). To temporarily pause operation, press the 'Pause' button. The machine will stop, but the lid will not unlock. To stop the machine press the 'Stop' button. When operation has slowed enough, you will hear the click of the lid unlocking.

Normal Sounds: The shatterbox is loud, so a constant hum and the sound of the rubber on the containers is normal. The foam and strapping surrounding the shatterbox helps keep it in place and minimize some of the noise.

Abnormal Sounds. If there are any metal on metal sounds shut off the shatterbox immediately. Something inside the shatterbox has probably come loose and will damage the inside of the container. Sometimes there is a thumping noise based on the balance of the machine as it operates, but it may subside after a few seconds. If it does not stop, pause the operation and wait for the machine to stop, then start it again. The shatterbox is meant to be operated on a level surface, and this thumping is a result of the boat movement usually. Therefore, **avoid using the shatterbox during high seas or during transit.** This should help reduce unnecessary strain on the shatterbox.

When the shatterbox cycle is done, open the lid and remove the vessels, placing them on the counter. Open the grinding vessel and with clean tweezers take a bit of the powder and feel it against the inside of your wrist. The sample should feel like talc powder. If it does not, repeat the shatterbox cycle. [A 1-3 minute cycle is enough for most of rocks.](#)

Transfer powder into Vial

Disassemble the vessel carefully wearing Nitrile gloves. Clean off any powder on the lid or puck with a Kimwipe or Teflon spatula. Carefully remove the puck from the vessel. Pour the sample onto a clean piece of weigh paper. If any powder remains, use a cleaned Teflon spatula to dislodge it.

Never use metal to dislodge or scrap out sample material, as any grooves or scratches in the vessels will increase the risk of contamination.

Cleaning the Grinding Vessels

Vessels must be cleaned in between samples and after all samples have been run for the day. Vessels should never be put away wet. This alters and tarnishes the grinding vessel.

[In Between Sample Runs](#)

1. Wearing nitrile gloves, wash the individual pieces of the grinding vessels with tap water and a small piece of a scouring pad (no soap). Rinse with DI water.
2. After each piece is washed, dry it with a Kimwipe, immediately spray it with isopropyl alcohol, and wipe it down with a Kim Wipe. Do not use the ship's compressed air line to dry pieces as the air is too dirty.
3. Lay the pieces on, and cover vessels with Kimwipes.

After the last run for the day

1. Take a scoop of quartz sand and put it in your vessel(s) and run it as you would a sample for several minutes (~3 min should be adequate).
2. Remove the vessel and empty out the sand. Scrub the pieces with tap water and a scouring pad and rinse with DI water. Dry with a Kimwipe, and then spray with isopropyl alcohol and wipe down with Kimwipes. If your vessel is particularly dirty run a combination of quartz sand, a little hot water and detergent (Borax). This can be run for several minutes. A thick paste will form and then you clean it as normal.