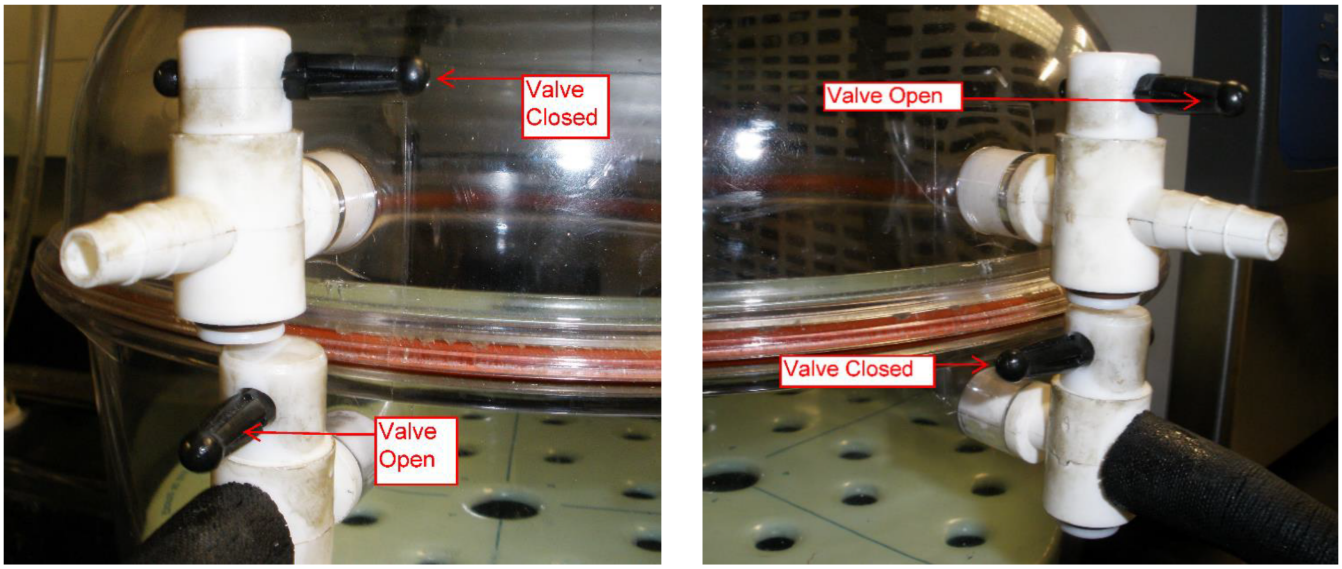
XRD Sample Preparation of Bulk Sediment

Bulk sediment samples are prepared for X-ray diffraction by grinding, which depending on the sample compaction, i.e. soft versus lithified, can be accomplished by two different methods.

Drying Samples

Freeze-dry sample(s) for at least 12 hours before grinding. If the samples still feel cold when removed from the freeze dryer, the samples still have moisture in them and need to dry longer. There is one freeze-dryer in the Thin Section Lab. If broken, use the freeze-dryer of the Chemistry Lab.

The freeze dryer is comprised of a sample "bell" chamber and a Labconco freeze-dryer. On the bell are two valves, as shown in *Figure 1*. Each valve has an "Open" and "Closed" position. The top valve controls the vacuum inside of the bell, and the bottom valve controls the air flow between the cooling coil and bell. A valve parallel with the tube is open and allows air flow; a valve perpendicular with the tube is closed. In *Figure 1*, configuration A on the left (closed) will hold a vacuum, but configuration B on the right (open) will not.



Making and Holding vacuum inside of the bell chamber *Releasing vacuum in the bell chamber*

Figure 1. (A) Freeze dryer bell valves in closed position. (B) Freeze dryer bell valves in open position.

To freeze-dry samples:

1. Cut open the sealed sample bags and fold the top edge over to keep the bag open.
2. Take the top of the bell off of the dryer and arrange samples in the bell, making sure no sample bags are pinched closed. Close the top valve of the bell chamber (*Figure 1A*).
3. Open the vacuum valve (bottom valve) slowly so that you do not cause a large rush of air to blow the samples around. *Figure 1A* shows the configuration to dry samples and *Figure 1B* shows the configuration for loading and unloading samples.
4. Flip the "On" switch located on the right side of the Freeze Dryer located in the Thin Section Lab.
5. Press the "Auto Refrigeration" button (*Figure 2, arrow A*) and then the vacuum button (*Figure 2, arrow B*). The temperature will start to drop and the vacuum pump will turn on.
   a. When the temperature drops to –40°C, the vacuum is created in the bell and the pressure drops.
   b. Expect the temperature to be between –42° and –52°C and the pressure to be ~0.350 mBar.
   c. The indicator lights (*Figure 2, arrow D*) show how the cooling and pressure reduction are progressing. When all indicator are lights are on the freeze dryer is at its peak performance.

d. If there is an error the red "Alarm" light will turn on. Press the "Menu" button (*Figure 2, arrow C*) to view it and clear it if necessary.

6. After samples are dry, close the bottom (vacuum) valve to stop the air flow between the cooling unit and the bell. Slowly open the top valve to release the vacuum in the bell. Remove the samples from the bell and store them inside the desiccator (located in the X-Ray Lab) until they are ready to be ground to prevent reabsorption of moisture.

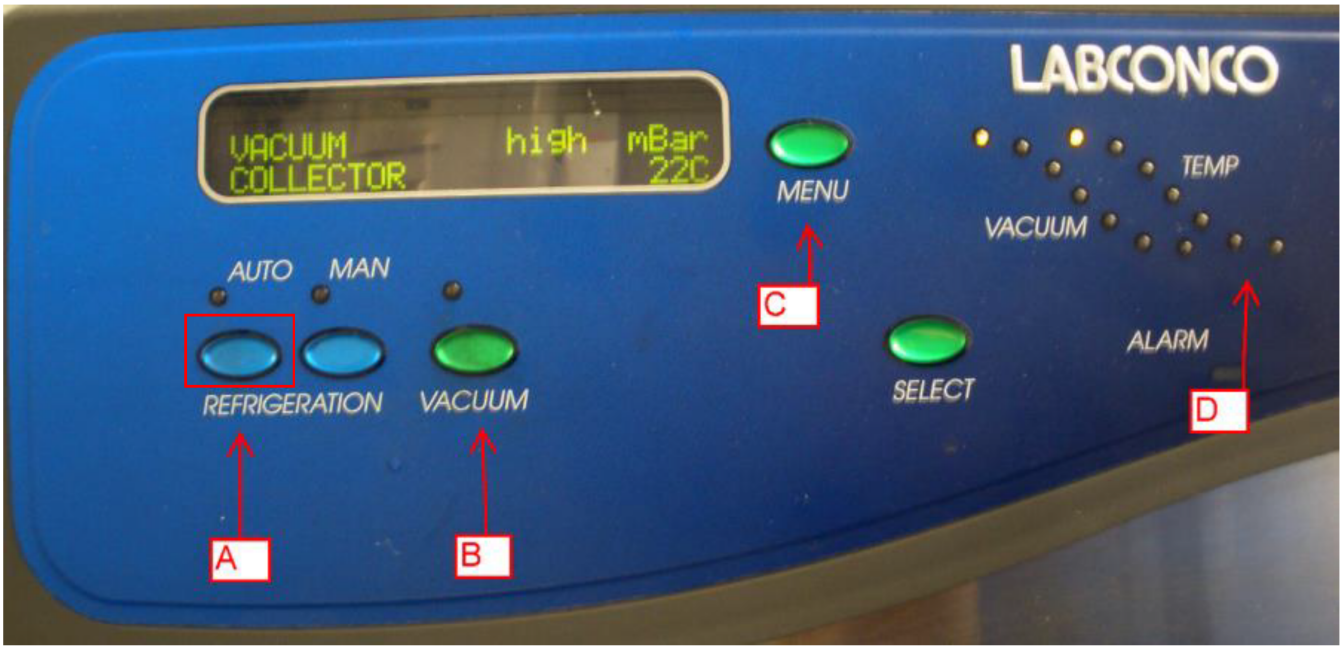


Figure 2. Freeze dryer control panel.

Mortar and Pestle

Choose the appropriate mortar and pestle size (large or small) and place it on the counter. Obtain a glass slide, a scoopula, and a sample holder (*Figure 3*). The sample holder shown in Figure 3 is a frontloading sample holder for the D4 Bruker XRD. Clean all items after each use with isopropyl alcohol and a Kimwipe to avoid cross-contamination.

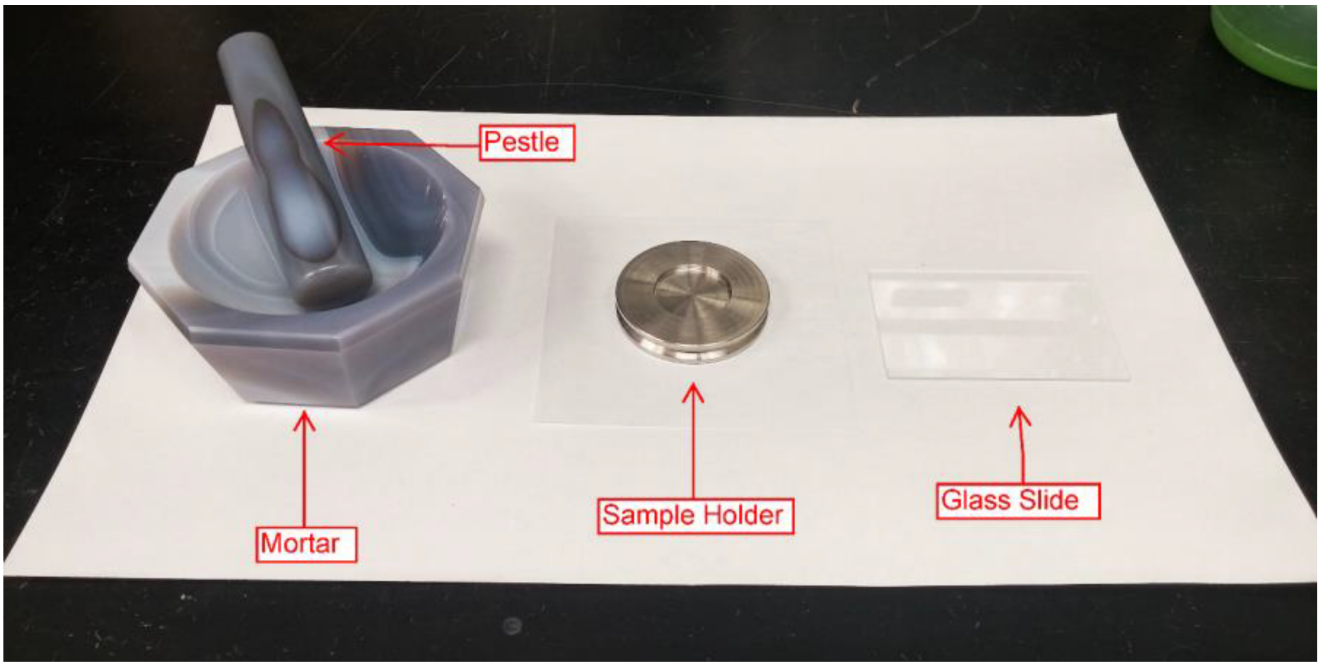


Figure 3. Mortar and pestle sample preparation set up to grind soft sediment.

Pour freeze-dried sample into the mortar and grind the sample with the pestle. When finished, the sample should be the consistency of talc powder. Test the sample by taking a pinch and rubbing it on your skin. If the sample feels gritty, it needs to be ground more. If the texture is talc powder like and the sample looks homogenous, you can pack the sample into a sample holder to be placed in the Bruker or Aeris XRD sample area.

Mixer Mills

The follow instructions are for two mixer mills, Spex 8000 and the newer Retsch MM400. Currently the Retsch MM400 is the recommended mixer mill to use and it is located in the Microbiology area of the Chemistry Lab.

Spex 8000 Mixer Mill

The 8000 mixer mill (*Figure 4*) is located in the X-Ray Preparation area of the Thin Section Lab.

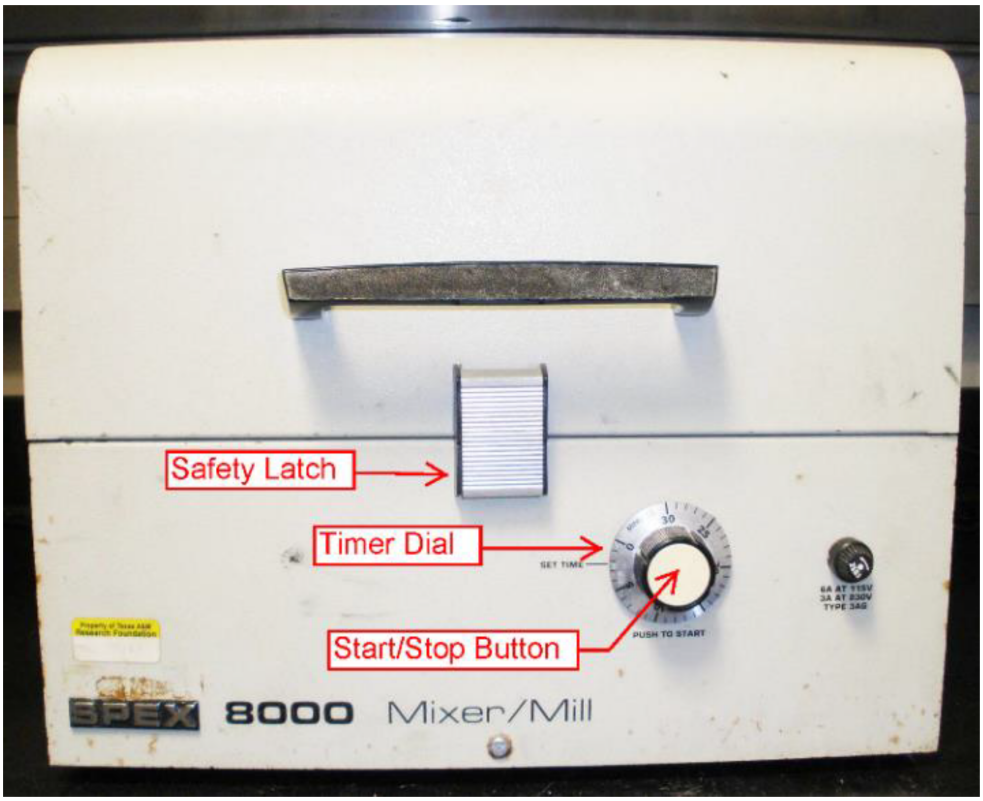


Figure 4. Spex 8000 Mixer Mill. Featured are the safety latch, timer dial, and start/stop button in the middle of the dial.

Grinding Vessels

There are three types of grinding vessels available for the 8000 mixer mill: alumina ceramic, tungsten carbide, and hardened steel (*Figure 5*). Tungsten carbide and steel vessels are better for more robust grinding, and alumina ceramic is better for minimizing contamination. Check with the Science Party to see which vessel type is preferred.

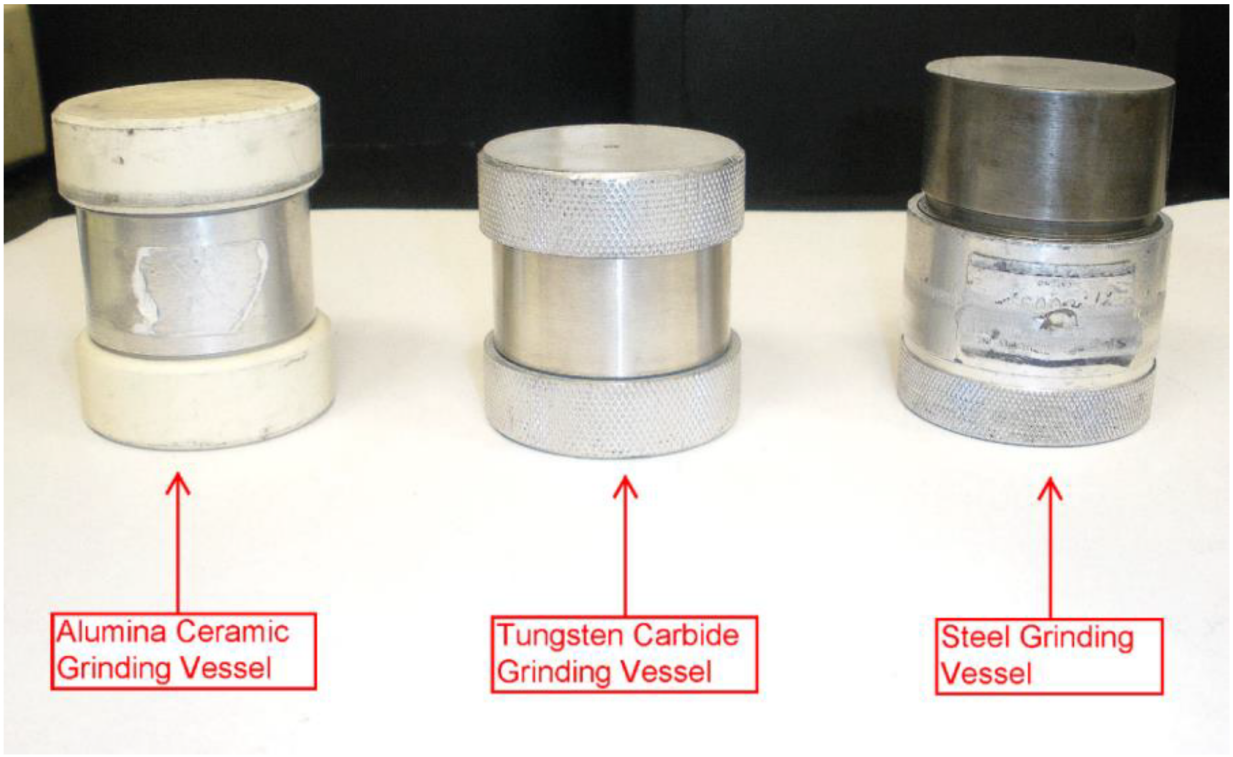


Figure 5. Three types of mixer mill grinding vessels.

Each grinding vessel has its own components. Parts should not be shared among the different types of vessels. **Combining pieces made of different materials can cause severe damage** to the pieces involved.

Below are the components for each type of vessel. The steel (*Figure 6*) and tungsten carbide (*Figure 7*) vessels both have a vessel body with attached lid and one separate lid that is screwed on. Cross-threading is very easy with these containers, so be very careful when screwing on the lid. Also note that the steel container has an O-ring, whereas the tungsten carbide does not. The alumina ceramic vessel (*Figure 8*) is assembled differently than the other two vessels: two cork rings are placed inside each lid, and the lids slip onto either side of the vessel body.

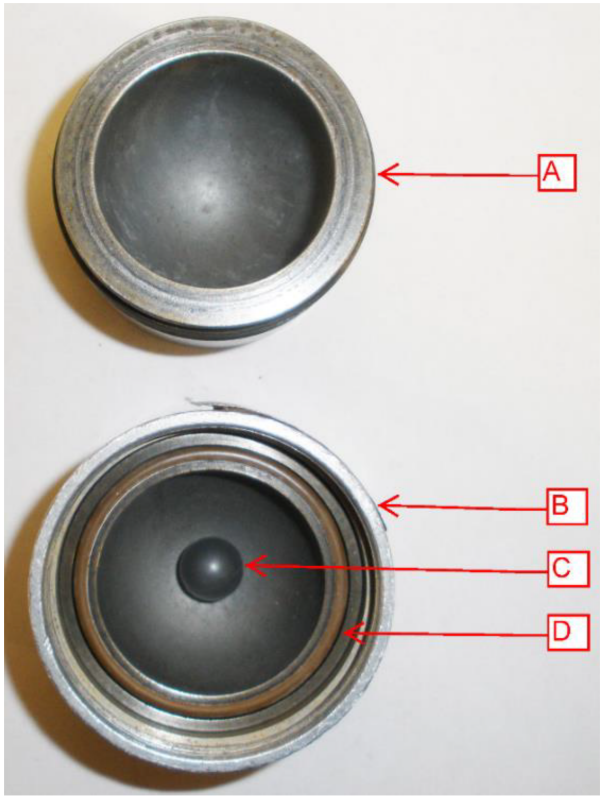


Figure 6. Steel vessel components. (A) Lid (B) Container (C) Steel Grinding Ball (D) O-Ring

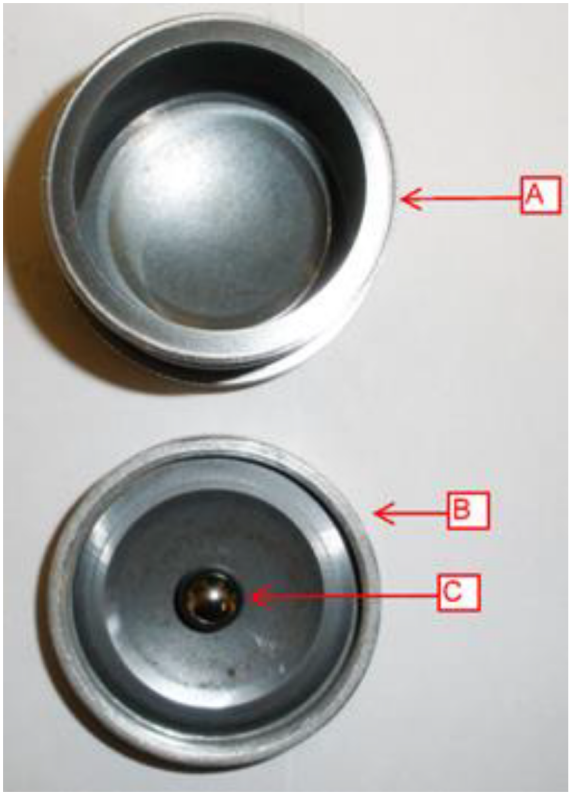


Figure 7. Tungsten carbide vessel. (A) Vessel container (B) Lid (C) Tungsten carbide grinding ball

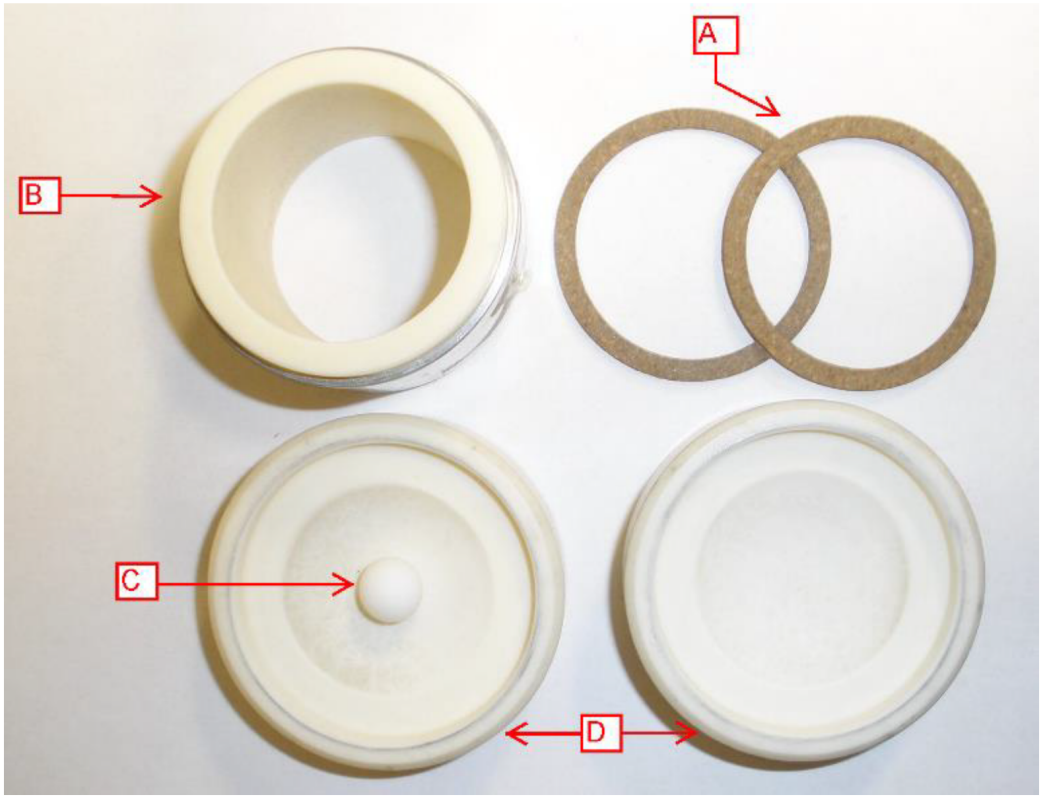


Figure 8. Alumina ceramic vessel components. (A) 2 cork rings (B) Vessel body (C) Alumina ceramic grinding ball (D) 2 lids

Label the vessel with a small printed label of the sample it holds. After the sample is ground, transfer that label to an 8 or 16 mL snap cap bottle that will hold the powder.

Loading the Grinding Vessel into the Mill

Put your sample inside the vessel. The material should be approximately the size of a pea to prevent any jamming and to ensure all pieces are ground up. Place 1 to 2 grinding balls inside the container. Tungsten carbide and steel vessels can take up to 2 balls. The alumina ceramic vessel is more brittle and 1 ball is recommended. Finish assembling the grinding vessel and open the lid to the mixer mill.

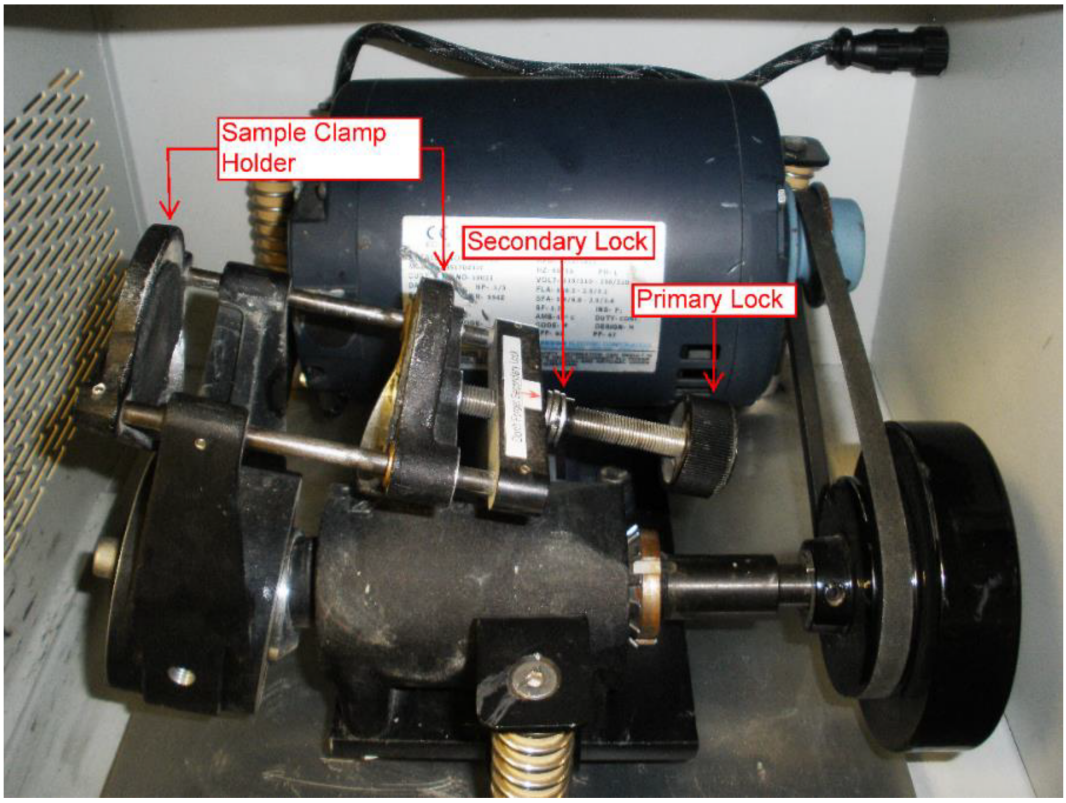


Figure 9. Inside the 8000M mixer mill. Highlighted is the sample holder clamp that holds the grinding vessels, the primary lock, and the secondary lock.

Hold the grinding vessel between the sample holder clamps (*Figure 9*), flush against one side. Still holding the vessel, start turning the primary lock. The holder will begin to clamp down on the sample. When the vessel is secured, you can remove your hand and continue tightening the primary lock until the sample is firmly gripped. Then tighten the secondary lock until it feels firm. Check the vessel to make sure the lids are resting flat on the clamps. If the vessel is ajar inside the clamp, when the motor starts the vessel can start grinding away at itself or fly loose into the machine.

Starting the Mill

To operate the 8000 mixer mill (*Figure 4*), turn the timer knob to the desired grinding time. The time will vary depending on sample material. Start with a conservative time, 2 to 3 minutes, check the material, and add more time if the sample still feels gritty. Harder rocks can go up to 10 minutes or higher to grind down to the right sample consistency. Press the "Start" button in the middle. That button will also stop the mixer mill. The dial does not move automatically, so if it is turned to 5 minutes it will stay there. The dial does not need be set back to 0 to work.
When the mill finishes grinding, remove the vessel the same way as it was put into the clamp: hold the vessel firmly against one clamp while unscrewing the primary lock. Keep the vessel straight and hold it firmly. If one of the lids starts to crack open, sample material will spill into the machine. If this happens, clean up the powder with a damp towel. Any leftover powder can get into the motor and damage it.

Transferring Powder

Collect a metal tray, weigh paper (large or small), 8 or 16 mL clear snap cap bottles, a scoopula, Kim Wipes, and isopropyl alcohol. Clean all materials with isopropyl alcohol. Place a piece of weigh paper on the metal tray. Pour the sample material from the grinding vessel onto the weighing paper. Use the scoopula to scrape the sides and lids of the vessel to remove extra material. Transfer the sample from the weighing paper into the sample vial. Label the vial with a small printed label and label the cap with a permanent marker.

Cleaning the Vessels

Clean the grinding vessels with a toothbrush and DI water. In some cases the vials are still dirty or have sample stains on them. If so, take a small amount, approximately 5 mL, of silica sand and grind it in the mixer mill for ~3 minutes. Then pour out the sand (can be trashed in burnables or collected and thrown overboard) and clean the vessel with DI water and a toothbrush or green scrub pad. Lay out cleaned vessel parts on a Kim Towel and dry with a Kim Wipe. The cork rings take a longer to dry, so collect fresh, dry rings before grinding the next sample.

Retsch MM400 Mixer Mill

The manufacturer's manual for the Retsch MM400 can be found here (in Retsch MM400 XRD Lab Notebook). *Figure 10* shows the Retsch Mixer Mill.

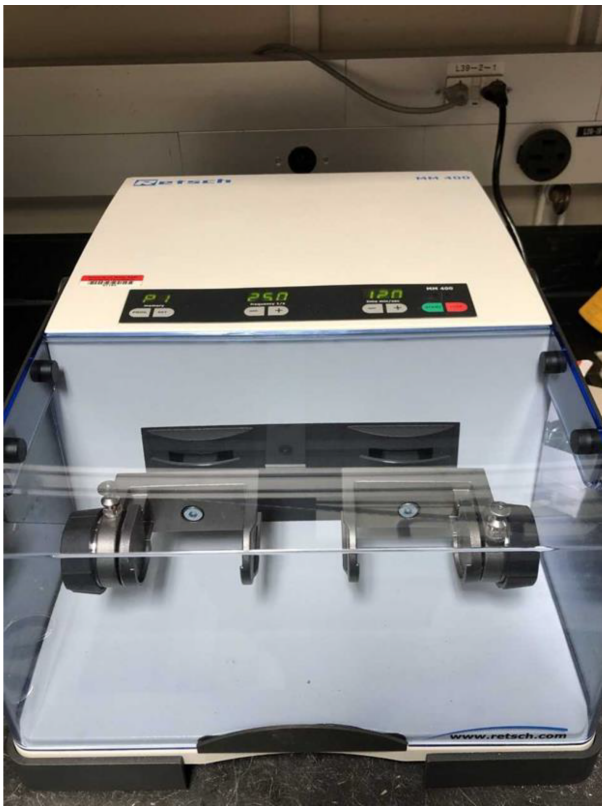


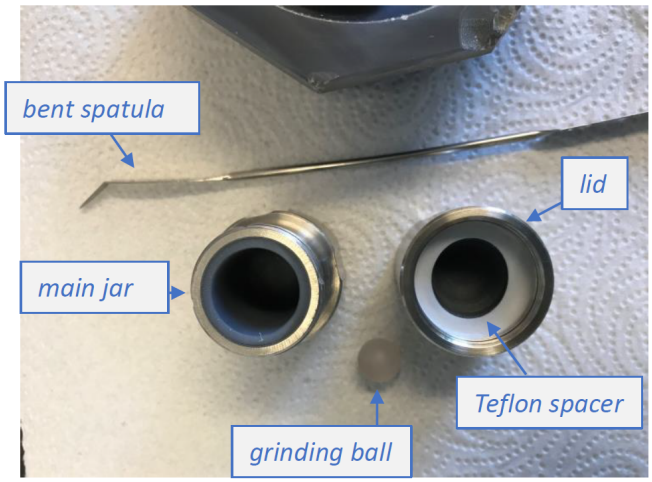
Figure 10. Retsch MM400 Mixer Mill

Grinding Vessels

Each grinding jar for the Retsch Mixer Mill (Fig. 11) has a main jar, a lid, a ball, and a white teflon spacer. **Use the Teflon spacer to avoid damaging the agate** (*Figure 12*). The inside of the jar and lid are lined with agate and the ball is of made of agate.

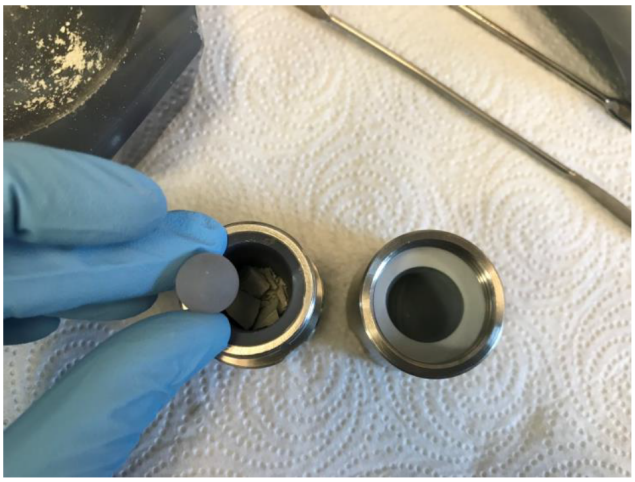
Figure 11. Agate grinding jars and ball.

Pour the freeze dried soft sediment (not hard rock) grains into the jar (*Figure 12, step #1*). Fill the jar half way to three-fourths with sample. Place one grinding ball in the jar (*Figure 12, step #2*). Clean off any of the sediment sample with a Kimwipe that has gotten onto the jars threads and screw the lid on. Be mindful not to cross thread the lid to the jar.



Sample holder of the Retsch Mixer Mill

1. Fill the jar half way to three-fourths with sa



2. Place one grinding ball in the jar

3. Screw the lid on

Figure 12. Agate grinding jars with teflon spacer and the bent metal spatula for removing the teflon spacer.

Loading the Jar into the Grinding Mill

Place closed grinding jar horizontally in the clamps with the pin in open position. Verify the pin is in closed position (*Figure 13*) and tighten the clamps by turning the handle until the jar is securely tighten (hand tight is good). After the grinding jars are secured in the clamps, the pin is in the closed position and the clear cover is closed it is time to select grinding settings (*Figure 13*). Close lid and select desired program to run.

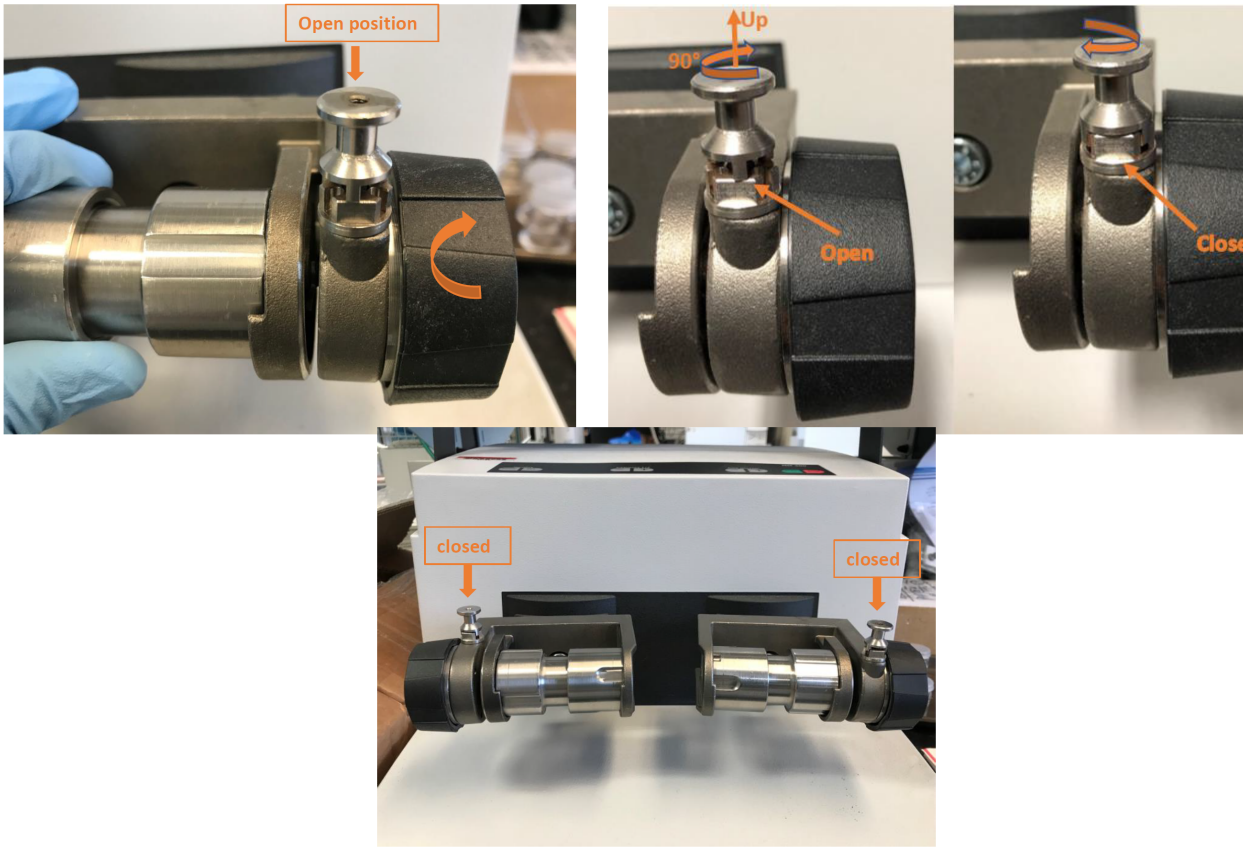


Figure 13. Retsch jar clamp

Starting the Mill

Press "PROG" to go through the list of programs already created (Figure 14, arrow A). Program 1 (A) is currently set for 12 min at 25 1/s. This setting is good for clay rich material. Softer sediments such as $CaCO_3$ typically need less time. *It is important to only grind soft sediments in the agate jars and hard material should be ground in the shatter box (see XRD Sample Preparation Hard Rock).* Press "START" to start a program (*Figure 14, arrow D*). After grinding cycle has completed turn knob counter clockwise and pull pin up and into its open position (*Figure 13*). Unscrew clamp and open jar to verify grind size. If sediment is still too coarse regrind.

Figure 14. A. Preset programs. B. The shaker frequency. C. The time grinder is set for. D. The start and stop.

To create a program, press "PROG" and choose a program number (you can create up to 9 different programs) (Figure 14, arrow A). When ready, press "SET" (Figure 14, arrow A), and numbers on screen start flashing. Select a frequency (Figure 14, arrow B) and a time (Figure 14, arrow C) by pressing "-" or "+". Press "SET" to save your program, flashing stops. Then press "START" to run the program just created (Figure 14, arrow D).

Transferring Powder

After grinding is complete, rotate clamp counterclockwise and pull the pin up into the open position (*Figure 13*). When the pin is in the open position, unscrew the black knob until grinding jar is loose enough to remove from clamps. Pour powder onto weigh paper or directly into clean and labeled sample bag or sample vial.

Cleaning the Jars

To clean the jars and grinding ball rinse with DI water and Kimwipes until the Kimwipe comes out clean. Finish cleaning by rinsing with isopropyl alcohol and drying with Kimwipes. **Use the bent metal spatula to pry out the white Teflon spacer, we have spares but those can be reused many times.**

Shatterbox

Shatterbox vessels are not commonly used for sediment XRD samples. Please refer to the XRD Sample prep for hard rock samples for instructions for using the shatterbox.