Procedures for Preparing Finely Ground Powders Using the Retch/Brinkmann Micro Rapid Mill

(modified from "Operating Instructions" supplied by Brinkmann Instrument Company)

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Introduction

IMPORTANT NOTE BEFORE PROCEEDING: The Brinkmann Micro-Rapid Mill was originally purchased to prepare rock samples for quantitative XRD analysis on a contract funded by Sandia National Laboratories. When the contract was completed, Sandia donated the equipment to the Department of Earth and Planetary Sciences. Careless use of this equipment can result in very expensive damage and we have no funds to replace broken parts. For this reason, NO ONE IS ALLOWED TO USE THIS EQUIPMENT WITHOUT THE EXPRESS PERMISSION OF THE XRD LAB MANAGER WHO WILL INSTRUCT ALL AUTHORIZED USERS ON ITS OPERATION.

The Retch/Brinkmann Micro Rapid Mill (MRM) is used to quickly grind, mix and homogenize materials. The MRM is essentially an automated, finely machined agate mortar and pestle. Unlike some other grinding equipment, the MRM grinds using a fundamentally non-percussive technique that tends to produce a minimal amount of very fine (< 1 μm) material. For this reason, the primary use of the MRM for YMP rock samples or standards is to prepare homogeneous, finely ground (less than 5 to 10 μm) powders for X-ray diffraction (XRD) analysis. It may also be used for preparation of powders to be made into pressed powder disks for trace element X-ray fluorescence (XRF) analysis, or for any other task in which a uniformly ground fine powder is required.

This procedure is a modification of the "Operating Instructions" supplied by the Brinkmann Instrument Company with the equipment.

These procedures are used for relatively small volume (3 to 5 mL or less) samples where fine, uniform size of the resultant powder is of prime importance. The larger clearance settings described in the "Operating Instructions" supplied by the Brinkmann Instrument Company may be used to prepare coarser powders from larger volume (up to 50 mL) samples.

Description of the Micro Rapid Mill

In the description of the instrument that follows, the numbers (in parentheses) refer to the numbers on the accompanying drawings ***located at the end of this document*** labeling various parts of the MRM. Figure 1 is a front view of the instrument (with the Plexiglas mortar cover installed) and Figure 2 is a side view (without the Plexiglas cover). The numbers refer to the parts, and not all parts appear on both drawings.

The MRM uses a pair of separately regulated motors to independently control the rotation of the mortar and pestle. The mortar rotation motor is located in the base of the unit, and drives the mortar holder (1) by means of a rotating key pin that fits in the base of holder.

The motor controls, from left to right on the front panel, set mortar rotation speed (2), pestle rotation speed (3) and grinding time (4). A green indicator (5) lights on the front panel when the motors are in operation.

A scaled micrometer screw (6) on the right side of the table controls the left-right positioning of the mortar table. The mortar table is spring-loaded to maintain pressure on the sample being

ground while allowing for movement to prevent excessive abrasion or equipment damage. A lock pin (7) may be removed to initiate eccentric operation of the mortar. ***Eccentric operation is not used for grinding fine powders, and this lock pin should always be in place.*** The position of the mortar table is shown on the scale (8)

From the mortar table upward, is the mortar holder (1), mortar (9), and pestle (10). The mortar is precisely machined to fit snugly into the mortar holder and engage with it by means of a driving pin in the holder (11). To facilitate the addition of a liquid grinding medium to the powder during grinding, the Plexiglas mortar cover sleeve (12), mortar cover (13) and rubber mortar cover gasket (14) as shown on Figure 2 are normally ***not*** installed.

Caution: Acetone will melt Plexiglas. ***Never install the Plexiglas items when grinding under acetone.*** Wet-grinding under deionized water may be done with the Plexiglas parts in place, but any other liquids should be tested for reactivity with Plexiglas on an external area of the Plexiglas prior to use as a grinding medium. Proper operation of the mill for large-volume samples requires a smooth inner surface be maintained for the Plexiglas containers.

The pestle is fixed in place by means of a locking Allen screw (15) in the pestle chuck (16). Pestle plunge adjustments are accomplished by use of the three-arm vertical adjustment lever (17) that moves the pestle motor (18) up and down. Maximum pestle plunge may be fixed by use of the precision set ring (19) and locked in place by the use the locking knob (20).

Equipment and Supplies Needed for Grinding

The following should be on-hand for use:

- ✓ Disposable powder-free rubber gloves
- ✓ Kim-wipes laboratory wipers
- ✓ White bond paper
- ✓ Medium-size weighing paper
- ✓ Squeeze bottle with liquid grinding medium (deionized water, alcohol or acetone)
- ✓ A clean double-edge razor blade
- ✓ Deionized water for rinse of mortar and pestle after cleaning

Setup of MRM for Preparation of Fine Powders

Before grinding, rock samples should be no larger than coarse sand size (particle size ≅1 mm). Powders will usually be splits of powders prepared using the SPEX 8510 Shatterbox, or by some other manual grinding technique described elsewhere.

NOTE: For samples to be used for quantitative or semi-quantitative analysis, percussive grinding (Shatterbox or ball-mill) should be kept to a minimum. Excessive percussive grinding can create a large fraction of extremely small size particles and induce strain in crystalline materials. These effects can cause broadening of the XRD peaks resulting in poor intensity data. Limiting the time in the Shatterbox can minimize these effects. In

general, the shatterbox should be used only to reduce material sufficiently so that it can be processed in the MRM.

In some cases, percussive grinding may be avoided entirely by grinding in the MRM in two stages starting with a coarse sand-sized sample prepared with the Bico jaw crusher or Plattner hardened steel mortar and pestle (discussed elsewhere). In this case, the first setting would be to grind to approximately 1 mm and the second setting to minimum tolerance. When using the mill in this way, it is extremely important that a wet grinding medium is used and tolerances be carefully set to insure that the equipment is not damaged.

- If necessary, clean the agate mortar and pestle. First wash in warm soapy tap water, rinse with deionized water, and blot dry using a lint free (Kimwipe or equivalent) laboratory wiper. ***Wear gloves while handling the mortar and pestle to avoid contamination by oils from the skin.*** All washing of equipment should be done in the Geochemistry Lab (Rm. 213) or other location where deionized water is available for the final rinse.
- Mount the mortar holder (1) being sure that the key pin is engaged with the brass keyhole in the base of the mortar holder, and the mortar holder is properly seated. Insert a clean agate mortar (9) in the holder so that the driving pin engages the hole in the mortar. Set the mortar table to the zero position (centered) as shown on the scale (8) by adjusting the micrometer screw (6). Make sure that the lock pin (6) is in place so that eccentric operation is disabled.
- Using the 3 arm vertical adjustment lever (17), raise the pestle motor arm assembly as high as it will go, locking it in place, if necessary, with the locking knob (20). Loosen the Allen screw in the pestle chuck (15), slide the pestle fully into the chuck, and tighten the Allen screw firmly. Do not over tighten.

Adjust pestle plunge for minimal clearance as follows:

a. Turn both motor controls (2, 3) fully counterclockwise to the clicked off (zero) position.

(**Note:** Mortar and Pestle motor controls can slip out of position so that scale readings are not accurate. If the knob points to a non-zero position or is off scale when clicked off, adjust the knobs so that they point to the zero position when clicked off.)

b. Turn motor control on by setting the timer (4) to some non-zero value (up to 12 minutes), and pressing the timer setting dial. The green light will come on.

c. Turn pestle motor on by turning the pestle rotation speed (3) up to about 20.

d. Turn mortar motor on by turning the mortar rotation speed (2) up to about 30.

e. Slowly lower the pestle using the 3 arm vertical adjustment lever (17) until the pestle just contacts the bottom of the mortar (you will hear the contact) and then raise it very slightly so there is no contact. The set ring (19) may have to be lowered by turning it clockwise. The pestle should be positioned approximately in the center of the mortar.

f. Use the micrometer screw (6) to shift the mortar to the left, bring the rotating pestle almost into contact with inside of the rotating mortar.

g. Lower the pestle into the mortar until the pestle and mortar just begin to click, then back off just enough to stop the clicking. When dry, the pestle will bind with the mortar when too tight causing a shaking of the table; be careful not to let this continue for more than a few seconds while making adjustments. The ideal position is just before the mortar and pestle bind, with a click or squeak indicating minimal clearance. Lock the pestle in position (20). Turn the set ring (19) counter-clockwise to fix this low pestle position. Slight movement caused by adjustment of the set ring and lock can cause the pestle to be backed off, and correct settings usually will require a few iterative adjustments for correct setting in the locked position.

NOTE: If grinding a coarse sample, back the set ring off by about one or full division (1 mm) to set for the initial grinding. The purpose of the initial grinding is to reduce large fragments to a more even size for final grinding, so precise setting of tolerances is not required.

h. Unlock the pestle unit and raise it slightly so that there is no contact between the mortar and pestle.

i. With the mortar and pestle motors still running, move the mortar to the left with the micrometer screw (6) until the pestle just begins to contact the mortar wall. When the mortar is in the correct position, a click should be heard with each rotation of the pestle. Adjust if necessary.

NOTE: If grinding a coarse sample, back off the micrometer screw ½-turn or a full turn for the initial grinding. This will give about ½ to 1 mm sidewall clearance for the initial grind.

j. With both motors running, lower the pestle to the previously set stop position. A faint chirping sound in addition to the sidewall clicking should now be heard. If it is not, lower the pestle, and readjust the set ring as explained above. (NOTE: If grinding a coarse sample, this final adjustment is not necessary.) When completed, raise the pestle clear of the mortar sleeve. Motors may be turned off, or left running if sample is ready to be ground.

It is important that the clearance be set correctly so that a fine, evenly ground powder can be obtained. It is also important that the mortar and pestle NOT be run for more than 20 seconds (or so) at a time in a dry condition to avoid damage to the mortar and pestle. It is MOST important that the plunge and sidewall adjustments not be too tight or damage will occur. The plunge adjustment is most critical, since the spring action of the mortar table helps to prevent sidewall damage. ***Do not set this equipment up without assistance from someone who is experienced in using it, or you may find yourself purchasing some expensive hardware to replace what has been damaged.***

Grinding of Samples

The procedure below describes wet grinding of fine powder under Acetone. Acetone is an excellent grinding medium for most materials because it evaporates extremely rapidly and leaves

no residue in the specimen. Acetone should always be used in a well-ventilated area and the breathing of vapors should be minimized. Laboratory-grade alcohol is also a good grinding medium, though it does not evaporate as quickly as acetone. Distilled or deionized water may also be used, but tends to cause some materials to flocculate; this can create problems in materials that tend to develop preferred orientation.

1. Measure no more than 3 to 5 mL of fine powder sample (or standard material) onto a clean piece of weighing paper. A smaller volume of sample will minimize chances of overflow of material from the mortar during grinding. If a larger volume of material needs to be prepared, grinding should be done in a few stages.
2. With the mortar and pestle motors on, lower the pestle to the position set previously for fine grinding. The bottom of the pestle should squeak slightly in contact with the bottom of the mortar, and click on the sidewall of the mortar with little or no movement of the mortar table. If necessary, readjust mortar and pestle position as described in the previous section.
3. Turn the motors off and add the sample powder to the open side of the mortar. Add a sufficient quantity of acetone from a squeeze bottle to moisten the powder; the powder should be moist without a lot of free liquid acetone notable. Excess liquid can cause splashing and loss of sample. If too much liquid is present, allow it to evaporate before turning motors on.
4. Set timer to 6 minutes (maximum time setting), set pestle rotation dial to about 20 (between 15 and 30 is acceptable), and mortar rotation dial to about 30 (between 20 and 40 is acceptable. Press the timer switch to start grinding. With the sample wet, the sidewall clearance may be reduced until a clicking and slight vibration of the mortar table is noted. (NOTE: Do not make this adjustment if grinding a coarse sample.) Motor speeds may be adjusted during grinding, and a periodic brief increase in pestle speed is useful to remove powder adhering to the base and sidewall surfaces of the pestle. If it is necessary to turn the motors off during grinding this may be done either by turning the motor controls fully counterclockwise, or by turning the timer to zero. If the latter method is used, resume grinding by resetting the timer and pressing the timer switch.
5. While grinding, periodically add a small amount of acetone to the open (left) side of the mortar. The sample should be kept moist throughout grinding. As the sample dries out, the shaking of the mortar table will begin to notably increase. Add acetone in a fine stream to quiet this shaking using a Nalgene laboratory squeeze bottle.
6. After grinding stops for first 6 minutes, reset the timer and start grinding again for another 2 to 6 minutes. Add acetone as required while grinding. When complete, unlock and raise the pestle slightly (a few mm), then use the micrometer screw (2) to shift the mortar to the right away from the pestle; set it at approximately the zero position so the pestle is centered in the mortar.
7. Raise the pestle so that its tip is level with or slightly below the rim of the mortar, and scrape it gently with a clean double edge razor blade, being careful that all powder falls into the mortar. Skin oil can contaminate your specimen so wear gloves at all times while working with the powder. When all powder has been removed from the pestle,

raise the pestle motor arm assembly as high as it will go, and lock it in place with the locking screw.

8. Remove the mortar from the mortar holder and allow the acetone to evaporate. Gently disaggregate the powder. A clean agate or ceramic pestle is convenient for this. Acetone will evaporate quickly in atmospheric conditions and does not require heating. After the powder is dry (i.e., shows minimal internal adhesion), transfer all powder to a sealable container, using a piece of clean weighing paper. The razor blade may be used to gently scrape the walls of the mortar to remove any adhering powder.
9. Prior to next use, clean the mortar and pestle as described in the previous section. During grinding, some powder will usually be spilled or splattered on surfaces in the lab. When you are done using the equipment, make sure everything is wiped clean and the work area left neat for the next user.

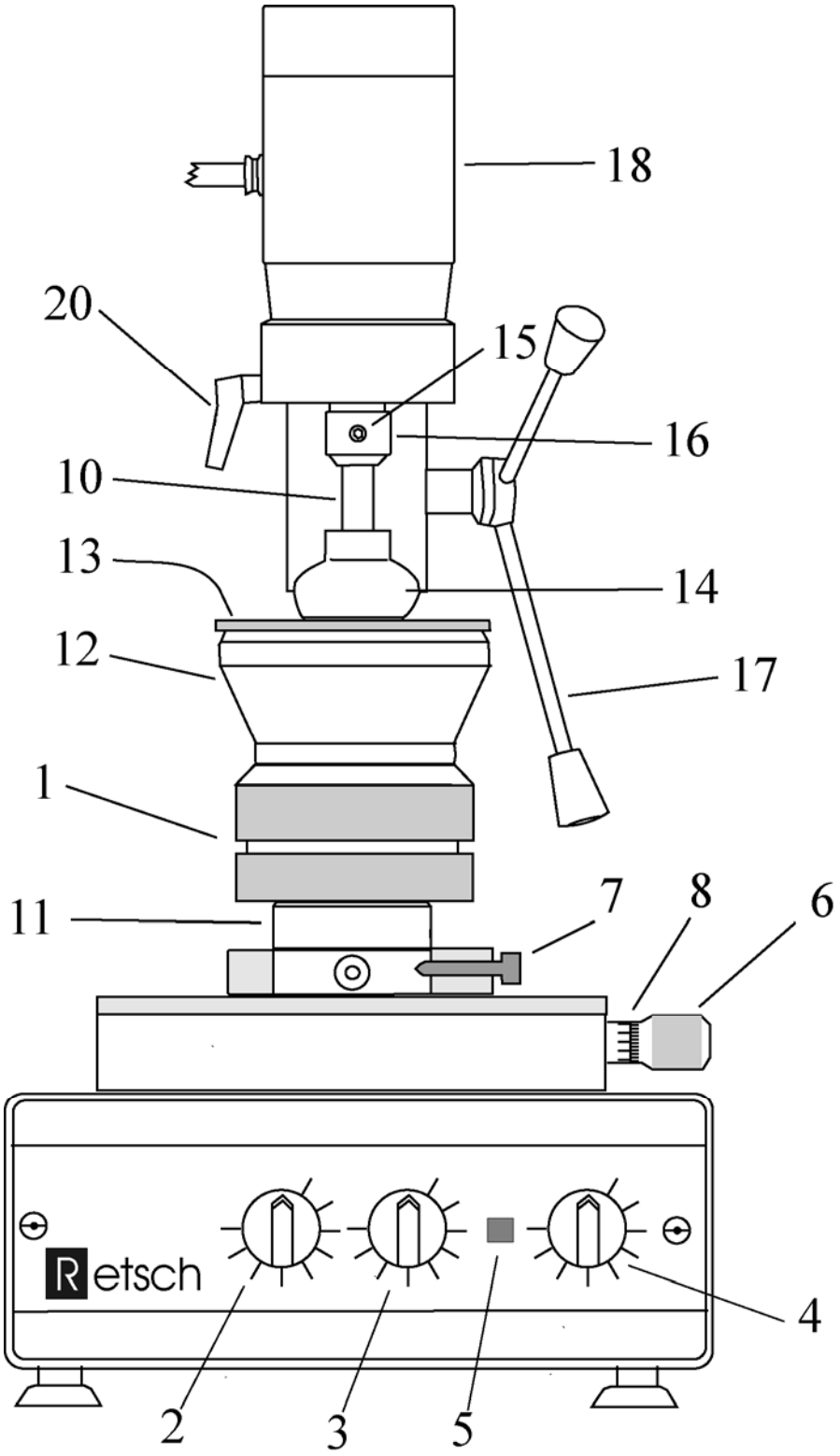

Figure1. Front-view of Retch/Brinkmann MRM (with plexiglas mortar cover installed) showing parts referred to in text.

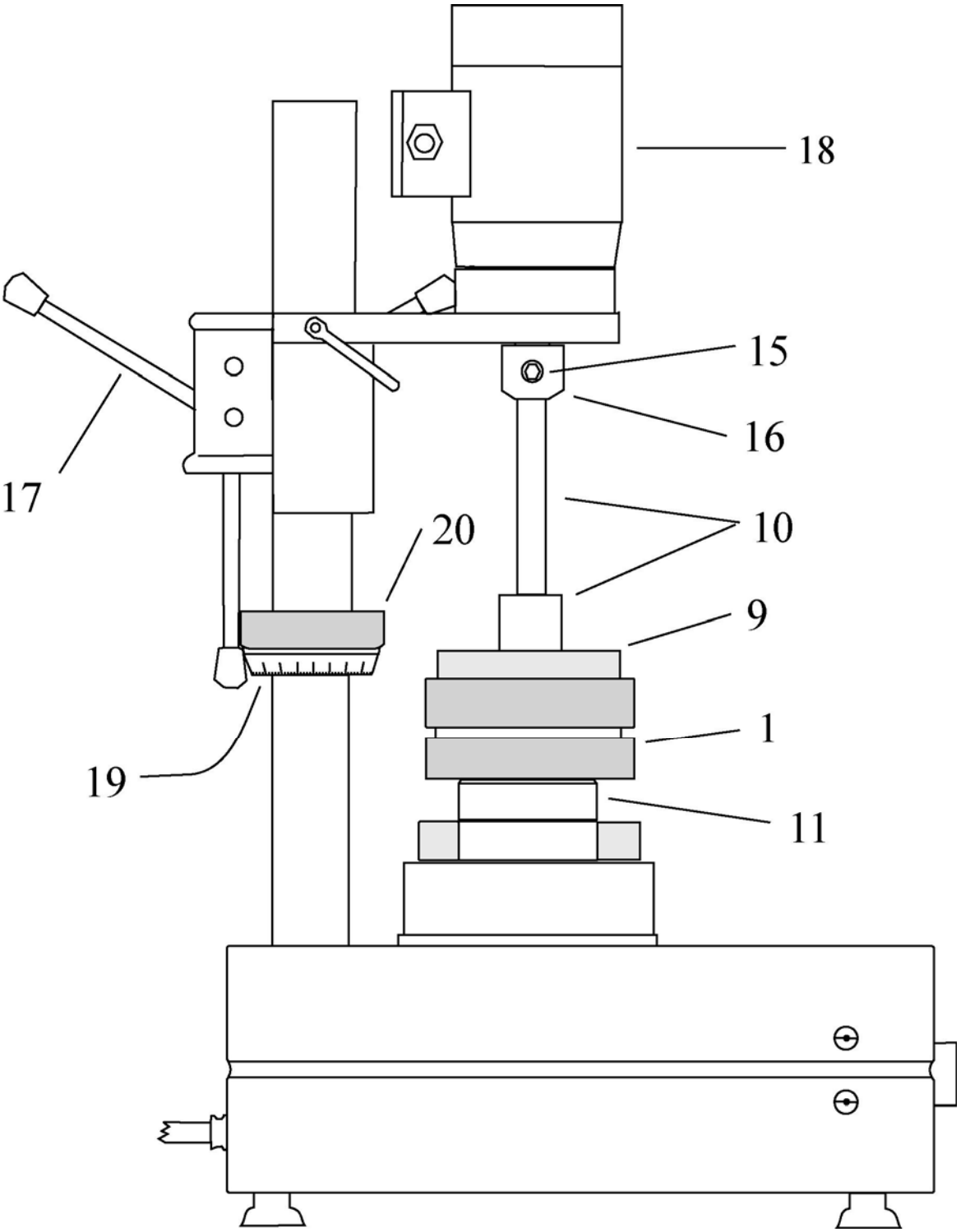

Figure 2. Side-view of Retch/Brinkmann MRM (without plexiglas mortar cover) showing parts referred to in text.