Procedures for Operation of the SPEX 8150 Shatterbox for preparation of powders for analysis

(modified from instructions provided by the manufacturer, SPEX Industries Inc., 3880 Park Ave., Edison, NJ 08820)

James R. Connolly
Department of Earth and Planetary Sciences
University of New Mexico
Albuquerque, New Mexico 87131

Latest Revision: December 28, 2004

Introduction

The Spex 8510 Shatterbox is used for the rapid grinding of brittle materials in batch sizes up to 100ml. The material to be ground is placed in a grinding container with a puck (and in some containers a ring in addition to the puck) of the same composition as the container walls. The motion of the shatterbox agitates the puck (and ring) inside the container and crushes the sample against the bottom and side walls.

The primary use of the shatterbox is to prepare finely ground (less than about 150 μm) powders of rock samples, standards or other engineered materials for X-ray diffraction, whole rock analysis, or any other task in which finely powdered material is required.

The shatterbox is located in the Analytical Geochemistry Laboratory and access to use the equipment must be cleared with Dr. Abdul Mehdi-Ali who is in charge of the laboratory.

Description of the Spex Shatterbox

In the description that follows, the numbers in parenthesis refer to various parts of the shatterbox labeled on the accompanying drawing (Figure 1 at end of document). Only those parts which are user-servicable or used in routine operation are described here. This information is taken from instructions provided by the manufacturer, SPEX Industries Inc., 3880 Park Ave., Edison, NJ 08820.

The shatterbox is driven by a special open motor, permanent split capacitor type, 50 C temperature rise, class B insulated, rated for 50% duty cycle, with a built in thermal protector.

The shatterbox is enclosed in a soundproof and vibration isolating housing. The top of the shatterbox consists of the container mounting equipment. The mounting equipment is made up of a cowling (1), clamping arm assembly (2), and hand knob assembly (3). Located in the center of the cowling is the capsule container recess (4). The clamping arm guide (5) is located on the clamping arm assembly. The guide and recess work in combination to hold grinding containers in place. The clamping arm assembly is held in place by its mounts (to the cowling) and the hand knob assembly. The hand knob assembly is held in place by the locking pin (6) located on the arm assembly. The mounting equipment is fastened to further assemblies (underneath) that cause it to move in an acicular motion when the motor is in operation. The entire shatterbox is enclosed in a Spex 8511 sound absorbing housing (not shown) which allows access to the mounting equipment from the top. Mounted on the right side of the front panel of the housing, are the shatterbox controls. The control on the left is the power switch and consists of a two-way (on-off) toggle switch. The control on the right is the timer and start assembly and consists of a turnable dial (timer) and a button located in the center of the dial to start the machine.

Grinding Procedure

1. Before grinding, sample particle size and volume of material should within the range specified in Table 1 for the particular grinding container being used. In general, minimum size should less than between 7 to 3 mm. Grinding with less than the minimum required amount can result in expensive damage to the containers ***for which operators are responsible***. (Replacement cost for containers ranges from $450.00 to $4,500.) Grinding with more than the maximum amount will usually result in poor grinding performance.

2. If needed, clean the container and puck to be used. First wash the container and puck in warm, soapy, tap water and lightly scrub them with a Scotchbrite (or equivalent) pad. Rinse the assembly with deionized water and dry thoroughly with compressed air. ***Grinding with a damp container can result in a poorly ground and "caked" sample.*** Wear clean, powder-free latex gloves (or equivalent) at all times while handling the puck or the inside of the container to avoid contamination by skin oils.
3. What follows are general instructions for all Spex brand grinding container assemblies. Maximum and minimum grinding amount, maximum grain size, etc. for the various grinding containers available for use are listed in Table 1. For most samples the zirconia (8506) or alumina (8504) grinding containers should be used to minimize possible contamination. If less than 20 to 25 ml of sample is available, then the 8508 tungsten steel container is recommended.

   All of the containers have different contamination "profiles", and will, in general, contribute small amounts of contaminating material. Staying within the minimum-maximum amount limits will minimize all types of contamination while grinding.

Table 1. Sample requirements for various container types and sizes.

Stock Num.	Container Name	Maximum Size	Minimum Amount	Maximum Amount	Notes
8401	Large Hardened Steel	7mm (1/4")	20ml	50ml	b
8504	Tungsten Carbide	7mm (1/4")	25ml	70ml	
8505	Alumina Ceramic	7mm (1/4")	25ml	60ml	
8506	Zirconia	7mm (1/4")	20ml	60ml	
8507	Small Hardened Steel	3mm (1/8")	5ml	20ml	a
8508	Small Tungsten Steel	3mm (1/8")	5ml	20ml	a
8521	Large Steel	7mm (1/4")	20ml	100ml	b

a Use with 8507R container rack

b If sample is too hard, use 8504 or 8505 containers

After the ring (if one is present) and puck are in place, place the sample to be ground in the container between puck, ring and container wall. ***Load only materials that meet size and volume criteria in Table 1.*** Make sure there is no material on top of the grinding puck, ring, or top surface of the container wall.

After the sample has been loaded, replace to lid of the container, making sure the rubber o-ring seal is in place. Place the container in the Shatterbox, seating it firmly in the capsule

container recess. Bring down the clamping arm and tighten the hand knob to between 4 and 6 foot-pounds of torque (snugly hand-tight but still easily loosenable). To prevent loosening of the hand knob, pull the locking pin under the hand knob up. Be sure that the clamping arm is seated properly on the lid of the grinding container. If the 8507 or 8508 containers are to be used, they must be used in conjunction with the 8507R rack. To do this, simply place the container onto the center of the base of the rack, making sure that it "locks" into place and then center the top of the rack directly on the lid of the container. The rack and container combination are used like any of the other containers.

Note: ***Failure to correctly load the container and tighten the clamping arm can (and usually does) result in catastrophic loosening of the clamping arm, spillage of sample and damage to the container and/or shatterbox. Users are financially responsible for damage that they cause, and the shatterbox and containers are very expensive.*** You have been warned.

Set grinding time by turning the timer dial to the desired interval. Place the toggle switch in ON position, and start the grinding process by pressing the start button in the center of the timer dial. The grinding process may be stopped before the time interval has passed by turning the timer dial to 0, or placing the toggle switch in the OFF position. Certain sample materials may cause the grinding action to stop (due to packing around the puck). When this happens there will be a sound change (the puck will cease tapping the interior wall of the container). To relieve this situation, turn the toggle switch to the OFF position and wait until the puck can be heard hitting the sides of the container, then return the toggle switch back to the ON position to continue grinding. Optimal grinding times will vary from material to material and are also somewhat dependant on the type of container used. Three minutes is recommended as an initial interval but may require adjusting. To avoid overheating the motor and the sample, avoid exceeding four minutes running time. If more total grinding time is needed, multiple runs of equal time intervals are suggested.

If, for some reason the machine overheats and the thermal protector interrupts the power to the machine, open the cover of the shatterbox and allow it to cool for several (suggest 10) minutes.

4. When the grinding process is complete, remove the container from the shatterbox (wear gloves as described in part two of this section). Remove the lid of the container and, using a small piece of weighing paper, carefully scrape any powder that has adhered to the lid, onto a clean sheet of large weighing paper. Repeat the process for the puck and then the container. Once done, transfer the powder to a clean, sealable, properly labeled container. In the unusual situation where sample has adhered to the container walls, a double-edged razor blade or scoopula may be used to remove powder from the container.
5. Prior to next use and when a grinding session is completed, clean the container as described in part two of this section. Since all containers contain a significant quantity of steel which is susceptible to rust, ***it is absolutely essential that all surfaces are clean and thoroughly dry before containers are put away***.
6. When done for the day, make sure everything is cleaner than it was when you started, and that all shatterbox containers are clean and put away.

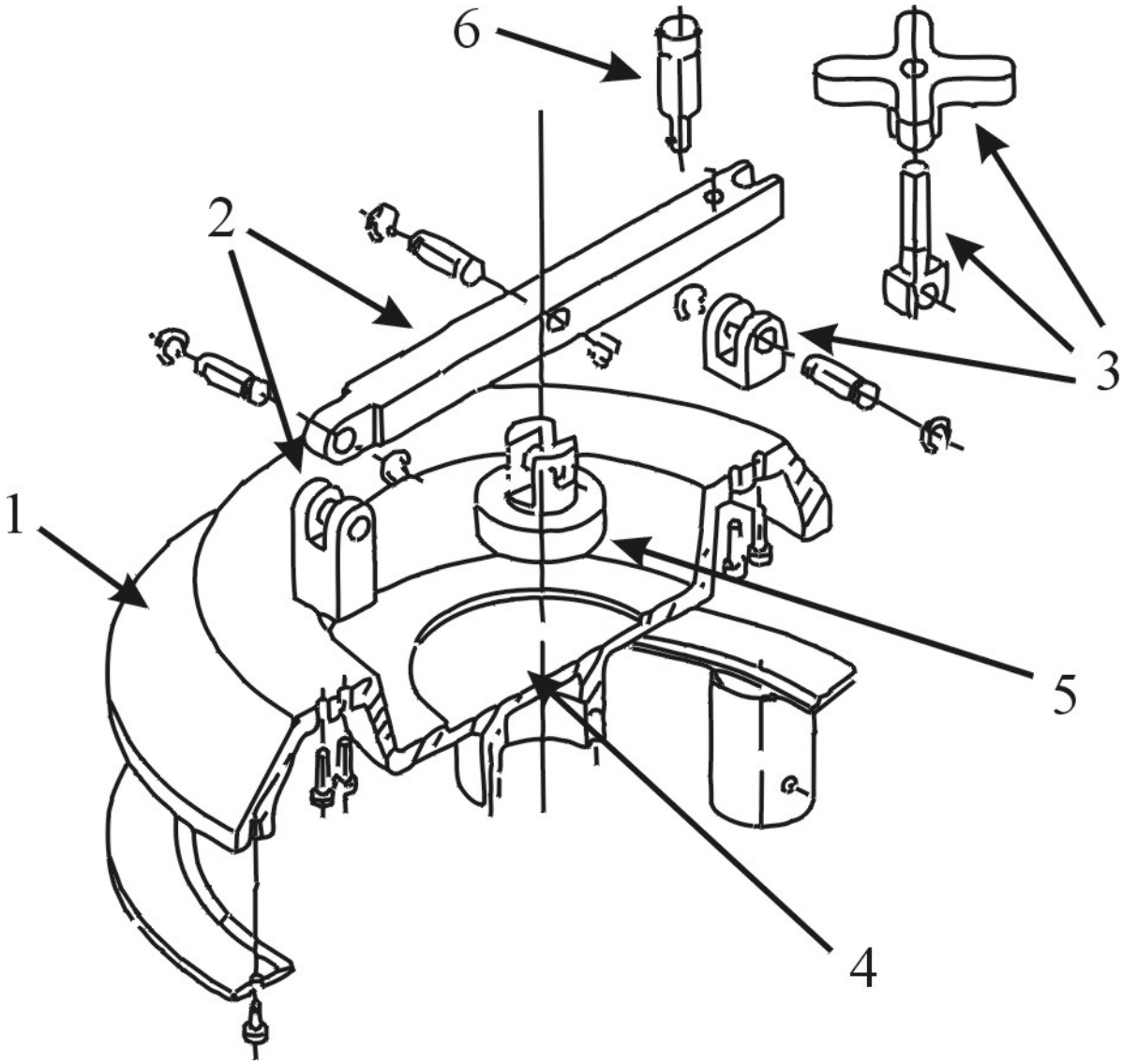

Figure 1: Cut-away cross section of container mount table of Spex shatterbox.
Numbers refer to parts referred to in text.