# SHAKER MILL: Standard operating procedure

Rev. 06/03/2009

**Description**

The mill operates by moving milling vials in a shaking motion pattern. The only adjustable milling parameter is the milling time. Two mills are available. The double mill must be balanced so that both vials must be loaded to the same overall mass.

**Before starting:**

1) Ensure that the work area is clear of dust, debris, material samples, and any lab components not directly used for the experiment. Use cleaning solution to prepare the countertop surface prior to start.
2) Determine the type of milling experiment desired. Identify milling parameters for your specific experiment. Select among the following milling parameter options:
    a. *Use of Process Control Agent (PCA)*: PCA inhibits formation of agglomerates and/or cold-welding. Some PCAs used are stearic acid and hexane.
    b. *Wet or Dry Milling:* "Wet" milling requires the use of liquid process control agent during the milling.
    c. *Vial Atmosphere:* Inert atmosphere requires loading vials inside the glove box which contains argon. See glove box instructions for details. For standard vials, alternatively, inert gas can be filled using customized vial lids equipped with valves.
    d. *Milling media*: Varied ball sizes are available for use as well as other milling media.
    e. *Charge ratio*: Mass ratio of the milling media to the powder sample
    f. *Milling time*: Needs to be determined before the experiment
3) Locate the necessary tools for opening, closing, loading, and mounting milling vials. Clean the tools before use.
4) Locate materials to be used as starting components

**Loading milling vials:**

1) Place the vials inside a fume hood.
2) Weigh the milling balls and the powders in separate weighing cups, one cup for each type of powder, and transfer them into the vials.
    Note: when loading powders, ensure no cross-contamination between samples and stock material.
3) For wet milling measure the necessary amounts of process control agent in separate jars.
4) For *inert atmosphere* milling, PCA should be preloaded into the sealable jars, one jar for each vial. The sealed jars, the vials with starting material and milling media, vial lids and respective bolts or clamps, and the wrench should be placed inside the evacuation chamber of the glovebox. Make sure that all the vials are partially open during the evacuation of the chamber; otherwise, the vials will be evacuated and will be difficult to open inside the glovebox. If no inert atmosphere milling is required, pour the prepared PCA into the vials placed inside the fume hood and immediately secure lids. Continue to step 7.
5) Inside the glovebox, pour the process control agent into the vials and immediately secure lids.
6) Important: **double check that the vials are tightly sealed**.

7) Important: **double check that the jars that contained PCA are empty** which means that PCA was poured into the vials. It is important to ensure that materials requiring wet milling are not milled without PCA.
8) If loading was done in the glovebox, remove the vials and all the accessories (the wrench, the empty jars etc.) from the glovebox.
9) Install the vials into the holding clamps. Make sure that rubber gaskets at the bottom of the vials are properly positioned.
5) Tighten the vials with the clamps. Tighten the stoppers on each clamp. Verify that the vials are held securely by the clamps. It is important that no vials will become loose during milling.
10) If thermistors are used, secure sensors to each vial with Velcro bands; verify the temperature signal at the output of the data logger.

**During the milling run:**

1) Monitor milling operation at least once every thirty minutes
2) It is possible to set up "after hour" runs for preparing samples using an established protocol
3) If thermistors are used, monitor the milling temperature

**Extraction of the prepared samples:**

1) After completion of the ball-milling run, clean surface of the lab bench and make sure there is a clean open area inside the fume hood. Verify that no flammable chemicals are placed on the bench and inside the fume hood to be used to recover the newly prepared material.
   **NOTE: The material prepared may be reactive in air, it is important that the protocol is followed. Make sure to wear goggles, lab coat, and gloves.**
2) If a new, unknown material is formed, determine if the material is pyrophoric.
   a. Load an UNOPENED vial and necessary tools inside the glove box
   b. Clamp the vial in a vise applying pressure along its axis.
   c. Slowly loosen the sealing nut.
   d. Slowly loosen the vise grip and crack the lid open
   e. Carefully remove a small powder sample with a spatula
   f. Place the spatula with this small sample inside the airlock and expose it to air for at least 3 minutes to determine the material's pyrophoricity.
   g. If the material is pyrophoric, leave it inside the glovebox inside the milling vial with the lid cracked open for 24 hours. Check whether the material is passivated. If it is not passivated, store it inside the glovebox.
3) Remove materials from the vials by washing them out with hexane. A suction assembly (found in the fume hood) can be used to collect the solution. If vials need to be cleaned of the sample leftovers, it is possible to use brush only for wet material. One approach is to simultaneously brush and apply the solvent using a squeeze bottle. **Do not use brush, spatula, or any other tool to remove dry powder from the vial.** Use of spatula with dry powder is only possible when
   a. the vial contains substantial amount of powder
   b. is inside the glovebox
   c. or if the powder is well known to be inert

4) Upon collection completion, transfer the material to the collection container.
5) For most materials, it is recommended to store them under hexane.
6) After completely extracting the material, clean all tools used and wipe down the surfaces with solvent. Properly dispose all the waste.