BRINKMANN AUTOMATED GRINDER PROCEDURE

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BRINKMANN AUTOMATED GRINDER PROCEDURE

1.0 PURPOSE

The purpose of this procedure is to describe the methods, procedures, and documentation used when operating the Brinkmann Automated Grinder to grind rock povder samples.

2.0 SCOPE

This procedure applies to Yucca Mountain Project samples being ground, for example, for x-ray diffraction analysis.

3.0 PRINCIPLES

N/A

4.0 DEFINITIONS

N/A

5.0 RESPONSIBILITIES

The principal investigator (PI) has the responsibility to assure implementation of this procedure for samples being ground for the Yucca Mountain Project. The PI may delegate performance of the procedure to any duly certified individual.

6.0 PROCEDURE

6.1 Overview

6.1.1 Equipment and Software Used

This DP is for the Brinkmann Automated Grinder (Spectro Mill Model MS), no other equipment or software is used.

6.1.2 Critical Laboratory Setup Parameters

To ensure proper grinding, the Brinkmann Automated Grinder must be set up in accordance with the instruction manual, reference 8.1.

6.1.3 Special Environmental Conditions

Although not required, the grinder is generally placed inside of a portable fume hood vented to remove acetone vapors and fine particulate material.

6.2 Cleanliness

6.2.1 Thoroughly clean the mortar and pestle after every use using an appropriate liquid (e.g., deionized water or alcohol).

6.2.2 Ensure that the underside of the pestle is clean and that both mortar and pestle are dry before using.

6.2.3 Transfer sample between sample bottle and mortar using clean sheets of weighing paper.

6.3 Traceability

6.3.1 Great care shall be exercised not to mix or contaminate samples. All subsequent analyses rely on this procedure. Work on only one sample at a time, and try to process samples in some logical order.

6.3.2 Carefully label containers into which the ground sample will be placed on both the side and top of each container so that tops cannot be switched. Label the container with some identifying feature that the sample within has been ground (i.e., "GROUND" on the side of the container, an * placed on the top, etc.)

6.3.3 Complete records shall be entered into a controlled YMP logbook for each sample, and shall include the complete sample name or number, date of grinding, name of the person grinding the sample, and length of grinding time. All entries shall be signed.

6.4 Sample Preparation

6.4.1 In order to prevent damage to the mortar and pestle of the automated grinder, samples must initially be crushed to a coarse powder. It is preferable that the samples be able to pass through a number 40 (425 μm) sieve, although if sieving is performed, <u>all</u> of a bulk-rock sample must be passed through the sieve.

6.4.2 Methods for reducing large samples to a size that is grindable are numerous. Smaller samples can be broken up into half inch pieces with a hammer and then placed into a percussion mortar to reduce them to a coarse powder. Samples may also be crushed using a hydraulic press or may be powdered using a shatter box or large ball mill.

6.5 Sample Grinding

6.5.1 Set up the mortar and pestle as outlined in the instruction manual, reference 8.1.

6.5.2 Pour the desired amount of sample onto a clean piece of weighing paper, then with the pestle fully down, pour the sample into the mortar. (Do not handle or touch the sample to avoid contamination).

6.5.3 Avoid grinding too much sample at one time as it will inhibit adequate grinding and may cause the sample to creep up the sides and out of the mortar while grinding. Using the agate mortar and pestle, a maximum of ~3 grams may be ground at one time. Using the tungsten carbide mortar and pestle, a maximum of ~1.5 grams of sample may be ground at one time.

6.5.4 Squirt several full eye droppers of acetone onto the sample. The sample should be suspended in a slurry. (Samples will grind finer if ground wet, and wet grinding minimizes structural damage to the crystalline phases being ground).

6.5.5 Make sure that the speed regulating dials for both the mortar and pestle have been reset to zero.

6.5.6 Turn the grinding time dial to the desired time and press. (This lights the green "power on"light and sets on internal timer). For suggested grinding times, see section 6.7.

6.5.7 Slowly turn both the mortar and pestle dials clockwise until the mortar dial is set at approximately 20 and the pestle dial at approximately 40. (To achieve proper grinding, it is necessary that the pestle turn faster than the mortar. The instruction manual suggests twice as fast).

6.5.8 As the sample dries, periodically squirt acetone down the side of the pestle to ensure that the sample stays wet.

6.5.9 The grinder will automatically stop as soon as the selected time runs out.

6.6 Removal of Sample and Clean up of Grinder

6.6.1 Turn both the mortar and pestle speed dials to zero.

6.6.2 Rotate the mortar micrometer counter-clockwise several turns to pull the mortar back away from the pestle. Raise the pestle until the pestle is just above the mortar and lock it in place.

6.6.3 Wait until the acetone has evaporated and the sample is dry.

6.6.4 Scrape the sample from the sides of the pestle into the mortar (a clean razor blade works well). Scrape any sample off the bottom of the pestle and into the mortar.

6.6.5 Removing the mortar, scrape the sides and bottom of the mortar and pour the powdered sample onto a clean piece of weighing paper. The sample may then be allowed to dry further before pouring it back into its respective sample bottle.

6.6.6 Wipe off the pestle and inside of the mortar with a clean cloth or tissue (e.g. Kimwipe) to remove the majority of the sample residue. Placing alcohol on a second clean cloth or tissue, wipe off the mortar and pestle once again, any tools used, (spatulas, razor blades, etc.), and the inside of the mortar holder.

6.6.7 Clean up any dust or waste sample that has accumulated in the immediate area.

6.6.8 Enter into a controlled YMP logbook the complete sample name or number, date of grinding, signature of the person doing the grinding, and the length of time used for the grinding.

6.7 Grinding Times

6.7.1 Softer minerals will grind faster than harder minerals, and in an inhomogeneous mixture such as a rock, a particle size distribution will develop. For quantitative x-ray diffraction analysis, it is desirable to get the average sample particle size as small as practically obtainable, less then 5μm and, preferably, less than 3μm.

6.7.2 Grinding a volcanic tuff for 10 minutes is usually adequate. A grinding time of 5 minutes is usually adequate for soft materials such as the mineral clinoptilolite. However, hard materials such as pure feldspars must be ground for at least 20 minutes to attain an average particle size of less than 3μm.

6.7.3 The law of diminishing returns also applies to grinding times. For example, the first 10 minutes of grinding oligoclase (a feldspar) produced a significant drop in particle size (~5μm). The next 20 minutes of grinding continued to decrease the particle size (~3μm), but after this point, additional grinding did not produce any significant decrease in particle size.

6.8 Data Analysis

Not Applicable.

7.0 QUALITY ASSURANCE

7.1 Personnel

Only those persons certified in accordance with the Los Alamos YMP Personnel Certification Procedure shall perform YMP-related sample grinding on this equipment.

7.2 Calibration

Not applicable.

7.3 Records

7.3.1 All samples ground shall be entered into a controlled YMP logbook, including complete sample name or number, date of grinding, signature of the person grinding the sample, and grinding time used.

7.3.2 All sample bottles containing ground sample shall be labeled as such.

7.4 Accept/Reject Criteria

7.4.1 The sample should have a consistency similar to baby powder. If the sample does feel "gritty" it should be reground. It is up to the person grinding the sample to determine whether the sample has been ground fine enough for their work.

7.4.2 Several potential reasons for inadequately ground samples are as follows:

- o Sample is not ground long enough.
- o Mortar and pestle are mal-adjusted.
- o Too much sample is being ground at one time.
- o Sample was not ground wet.
- o Sample is harder than the mortar-pestle material.
- o The crystallite shapes are not conducive to grinding (i.e. platy minerals such as micas).

7.4.3 The logbook entry for a sample shall constitute evidence that the procedure has been implemented and satisfactorily accomplished.

7.5 Procedural Deviations

Deviations from this procedure shall be fully documented in a controlled YMP logbook explaining the deviation and the effects it may have on the resulting work.

7.6 Storage, Shipping, and Handling

7.6.1 Samples will be tracked, handled, shipped, and stored in accordance with the procedure for Sample Identification and Control for Mineralogy-Petrology Studies (Ref 8.2).

7.6.2 The equipment require no special storage, shipping, or handling considerations.

8.0 REFERENCES

8.1 Operating Instruction #202, Spectro Mill Model MS, Brinkmann Instruments Co., Cantiaque Road, Westbury, New York.

8.2 TWS-ESS-DP-101: Sample Identification and Control for Mineralogy-Petrology Studies.

9.0 ATTACHMENTS

None.