



Chapter A1

Rock Sample Preparation

By Cliff D. Taylor and Peter M. Theodorakos

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Principle

Most samples of naturally occurring material require some kind of physical preparation prior to chemical analysis. Samples require preparation to effect one or more of the following: (1) reduce the sample to a size that is more conveniently transported; (2) increase the sample surface area to enhance the efficiency of subsequent chemical attack; (3) homogenize the sample to ensure that a subsample is representative of the entire sample; and (4) separate the sample into components based on mineralogy, grain size, or physical and morphological criteria. Sample preparation is an important step in the analytical process. Without careful preparation, and attention to inter-sample contamination, the worth of the subsequent analyses is significantly diminished.

Rock samples are reduced to ½-cm fragments in a jaw crusher. The crushed sample is split, if necessary, and fed into an operating and properly adjusted Braun vertical pulverizer equipped with ceramic plates. The sample is ground to approximately minus 100-mesh (<150 µm) and mixed to insure homogeneity for subsequent analysis. Mineral samples with distinctive cleavage planes (i.e. mica flakes) can present a problem in grinding due to the crystal structure of the sample. In some methods where the quality of pulverization is critical in obtaining accurate results, shatterboxing the sample is required. The sample is placed in either a ceramic or agate shatterbox and pulverized until 100 percent passes a 100-mesh screen.

Scope

Approximately 50 samples can be processed per day.

Apparatus

- Sample cartons, 3-oz (1⁷/₈" x 2¹/₂")
- Large sample funnel, plastic
- Compressed-air source, dry air, 40 psi
- Core splitter, if available
- Rock hammer
- Flexible hand pad, 6" x 4" x 1" (15 cm x 10 cm x 2.5 cm)
- Steel plate, approximately 8" x 8" x 1" (20 cm x 20 cm x 2.5 cm)
- "Chipmunk" jaw crusher
- Knife
- Brush, automotive parts cleaning, stiff bristle
- Brush, wire
- Vertical pulverizer with ceramic plates, catch pan, and cover plate
- Silicon carbide, approximately 60- to 80-mesh (250 µm to 180 µm)
- Jones splitter, with ½" riffles (1.25 cm)
- Tube-type revolving mixer, with tube diameter to accommodate sample cartons

- Rectangular aluminum “loaf” pans to fit under Jones splitter, to serve as catch pans for the crusher, and to hold the samples during various stages of processing
- Grease gun for lubricating equipment
- Kimwipes or paper towels
- Assortment of tools for equipment maintenance

Safety precautions

Eye and ear protection and a dust mask must be worn and it is recommended that a lab coat be worn. Caution must be exercised in operating the equipment, particularly the jaw crusher and the Braun pulverizer, which have the potential of inflicting serious injury if not properly and carefully used. Keep your hands, hair, and clothing away from any moving machinery parts. Remove all jewelry before you begin work. Belts on equipment must be guarded to prevent catching clothing, hands, hair, etc. Power should be turned off prior to dislodging any jammed material from the equipment with a push stick. Power should likewise be turned off prior to making adjustments to the equipment, except when adjusting the grinding plates of the pulverizer. Rock particles and fragments ejected from the crusher and grinder can cause injury, operate grinder behind safety shield. Compressed air, used to clean the equipment and work area, presents a safety hazard, especially to the eyes. Particles of debris propelled by the high velocity air stream present an additional hazard. The compressed-air stream should never be directed toward the face. A fan or hood exhaust should be used to vent dust. See the *CHP* for further information.

Preliminary procedure

Check the Request for Analysis form (RFA) for notes on mineralogy of samples, requests for preparation that vary from standard procedure, and disposition of excess sample.

Verify that the number of samples received and the field numbers on the sample collection/transport bags correspond to the number of samples and field numbers listed on the RFA. If they do not correspond, contact Sample Control.

Properly label the correct number of sample cartons with the laboratory number assigned to each sample. Label both the container tops and sides using permanent ink markers, or premade labels. Affix premade labels to the tops and side of the cartons with clean transparent tape.

Place the labeled sample containers in a cardboard tray labeled with the required information: (1) assigned job number, (2) submitter's last name, and (3) number of samples in the job.

Procedure

Check to see sample-size of chips produced by jaw crusher is adequate. If not, adjustments to the jaw crusher are made by varying the number of metal shims inserted behind the stationary jaw plate. Increasing the number of shims reduces the crushed rock fragment size. The spacing between the sides of the movable jaw and the cheek plates can also be varied with metal shims inserted between the cheek plates and the body of the crusher.

Open the sample bag and place the sample into a loaf pan in preparation for crushing and splitting. Using a core splitter or a rock hammer and steel plate, break all large pieces down to approximately 2½" x 2", a size that readily fits into the crusher. Clean the core splitter, hammer, and plate with a wire brush and compressed air prior to use and between samples.

Place a second loaf pan under the jaws of the crusher to catch the crushed sample and begin feeding the sample into the jaws. Do not overload the jaws. Overloading may cause the crusher to bind. Hold the 6" x 4" x ½" hand pad over the mouth of the crusher to prevent rock chips from flying out of the jaws. Crush the entire sample, using more than one loaf pan if necessary.

Turn the power to the crusher off and thoroughly clean the crusher mouth, jaws, and cheek plates by alternately scrubbing the components with the parts brush and blowing away dust and fragments with compressed air. Lodged rock fragments and buildup of powdered rock material can be removed with a long, thin steel knife blade.

When extreme cleanliness is required to avoid low-level contamination or when ore-grade samples are being prepared, a small quantity of quartzite gravel should be crushed before crushing each sample to clean the apparatus. If such a cleaning gravel is not available, a small amount of the next sample to be prepared should be crushed and discarded with the Jaw crusher scrubbed out prior to preparing the whole sample.

When necessary, split the entire crushed sample by distributing it evenly into a Jones splitter to obtain a representative split of sufficient size to fill the sample carton. Save or discard the remainder of the sample, whichever is noted on the RFA. It is suggested that the bulk material be saved until after all the samples have been pulverized. In the event of sample loss during pulverizing, additional sample is then available. Clean the splitter and splitter pans with compressed air prior to splitting the next sample. Use the knife to dislodge fragments caught in the riffles of the splitter.

Turn on the Braun pulverizer and check the adjustment of the ceramic grinding plates. Plate adjustment is checked visually with the aluminum catch pan removed and by sound. The rotating lower plate should be evenly contacting the stationary upper plate and there should be a slight "skipping" sound. Adjusting the plates closer than this without any sample material present can cause the plates to chatter and bind. If the chattering becomes severe enough, the plates can crack or shatter, rendering them useless.

Adjust the plates by holding the threaded adjustment shaft, at the top of the pulverizer, stationary with a 12" crescent wrench and loosening the two lower adjustment nuts. The upper adjustment nut is then *slowly* turned to raise the shaft. This brings the lower revolving plate closer to the stationary upper plate; the closer the spacing between plates, the finer the size of the ground sample. When the desired spacing is achieved, the lower nuts are screwed tight against the lower side of the topmost, fork-shaped, pulverizer frame piece, locking the adjustment shaft in place. Proper adjustment of the plates is verified by pulverizing several ounces of quartz sand. Sieve the ground material through a series of mesh sizes, bracketing the desired particle size. With experience, the operator can quickly determine the suitability of a grind by visually examining, and by feel of, the ground sample.

Proper adjustment of the ceramic plates extends the useful lifetime of the plates. Even a small improper adjustment of the plates results in uneven plate wear and/or grooves and ridges forming on the plate surfaces. This has obvious implications on grinding efficiency and quality. Minor plate imperfections can be removed by running several ounces of silicon carbide through the operating pulverizer, and with great care, slowly closing the gap between the plates while the carbide is being ground. The process is repeated with the now-used portion of carbide until the desired even spacing and skipping sound of the plates is achieved. Test the adjustment by pulverizing a small amount of quartz sand and check the result as described above.

Caution: The plates can be over-adjusted; plates that are run too close together can easily bind, chip and crack. The used carbide is probably still suitable for at least one more use and should thus be saved in a cardboard container marked "Used SiC". The adjustment procedure is the most critical step in consistently producing acceptably prepared samples. It is also the most difficult step to perform. With increasing experience, the degree of difficulty diminishes.

Carefully place the catch pan under the plates of the running pulverizer, lift and rotate the pan until the two wedges on the upper lip of the pan are firmly engaged in the slots of the hopper. Clean the pulverizer by passing several ounces of approximately 20-mesh quartz sand through the pulverizer. Examine the ground sand for adequate fineness and adjust plates, if necessary, as described above. Discard the sand and thoroughly blow out any excess sand and dust from the pulverizer and pan with compressed air. Replace the catch pan.

Pour the crushed rock from the sample carton into the hopper and place the removable cover plate over the hopper to prevent the sample from flying out of the pulverizer. Keep the catch pan in place until all of the sample is ground, which is readily determined by sound.

Place a mixing card into the sample carton which held the crushed sample and place the powder funnel over the top of the carton. Carefully remove the catch pan containing the completely pulverized sample from the pulverizer by turning the pan in the opposite direction used in placing the pan, and lowering the pan below and away from the revolving plate (and the plate lock nut located below the plate). Examine the fineness and thoroughness of grind. The sample is considered acceptable if 100 percent passes an 80-mesh screen ($<180 \mu\text{m}$) and at least 80 percent passes a 100-mesh screen ($<150 \mu\text{m}$). Pour the prepared sample from the pan through the funnel into the carton. Fill the carton $\frac{2}{3}$ to $\frac{3}{4}$ full. Discard or save excess sample according to the submitter's request. If the quality of the grind is not acceptable, the sample should be reground. If this fails to improve the quality, the plates likely need adjusting and/or smoothing, as described above.

Thoroughly clean the pulverizer and catch pan using compressed air. Remount the catch pan on the pulverizer and grind about 1 oz of quartz sand to further clean the plates of the residue from the previously prepared sample. Remove the pan, discard the sand and again thoroughly blow remaining dust and particles from the pan and pulverizer with compressed air. When some "sticky", fibrous, micaceous, or ore-grade samples are prepared, the cleaning process should be repeated two or more times until no visible traces of the sample remain in the pulverized sand.

Pulverize the remaining samples in the same manner.

Secure the lids of the sample-bearing containers with tape if the lids are not snug and place the containers into the tube-type mixer. Turn on the mixer and allow the samples to mix for 15 min. Mix all of the samples in the same way.

This completes the preparation process. Clean the work area and return the completed job to sample control.

Equipment maintenance

All mechanical equipment should be lubricated at least once each week, or more often as may be required by heavy use. Use a grease gun containing metal-free grease (i.e. free of elements of interest in analysis) and make certain the lubricant is injected into all of the grease fittings. Do not over-lubricate and wipe excess grease from the fittings with a Kimwipe or paper towel.

Check and make sure all nuts and bolts are securely tightened, prior to turning on any equipment. Check moving parts, including crusher and pulverizer belts, crusher jaws and arms, grinding plates, and pulverizer bushing for wear. Replace worn parts.