

Standard Operating Procedure No. 90

Heavy Mineral Separate X-Ray Diffraction Sample Preparation

REVISION LOG		
Revision Number	Description/Revised By	Date
SOP 90.0	Erik Oerter and George Brimhall authors	7/08/04
90v1	Fixed some of the formatting and Sent to Jack Hamilton to post on Utah website LMK	8/19/05
90v1	Finalized by LMK for posting on Molycorp project website and to send to George Robinson for lab audit	4/4/07
90v2	Editorial by SKA	10/28/08

1.0 Purpose and Scope

This Standard Operating Procedure (SOP) provides technical guidance and procedures for the preparation of heavy mineral separates for analysis by X-Ray Diffraction. The procedures outlined here are specific to the labs and instrumentation of UC-Berkeley's Earth Resource Center, and may be applied to other labs with some modification.

2.0 Responsibilities and Qualifications

The supervisor(s) will have the overall responsibility for implementing this SOP. They will be responsible for assigning appropriate staff to implement this SOP and for ensuring that the procedures are followed.

All personnel performing these procedures are required to have the appropriate health and safety training. In addition, all personnel are required to have a complete understanding of the procedures described within this SOP, and receive specific training regarding these procedures, if necessary.

All laboratory staff are responsible for reporting deviations from this SOP to the supervisor.

3.0 Data Quality Objectives

By maintaining highly consistent sample preparation techniques from sample to sample, systematic errors can be minimized. Additionally, by employing the most simple and direct methods, opportunities for human error are reduced.

4.0 Related Standard Operating Procedures

The procedures set forth in this SOP are intended for use with the following SOPs:

- 1 Data management (including verification and validation)
- 2 Sample management (chain of custody)
- 5 Sampling outcrops, rock piles and drill core
- 6 Drill logging, sampling of subsurface materials
- 7 Decontamination of sampling equipment
- 8 Sample preparation
- 9 Test pit excavation logging and sampling
- 34 Remaining Pyrite Model
- 35 Sample preservation storage and shipment
Heavy Mineral Separation procedure

5.0 Equipment List

The following materials are required for this SOP:

- Heavy mineral separate material
- Agate Mortar and Pestle
- Large glass slide (approx. 3" x 2")
- 2 Small glass slides (1.5" x 1")
- Circular glass slide (~1" diam)
- Dow Corning silicone high vacuum grease
- Putty knife – ground and lapped for a very flat knife edge
- Aluminum sample holder
- Utility syringe (~3 cc)
- Stainless steel lab spatula
- Micro-balance with a minimum 0.0001 gram precision
- Weighing dishes
- Tweezers
- Acetone in a squirt bottle
- Marking pens
- 1 ml sample vial with attached cap
- Kimwipes
- Temporary adhesive (gluestick)
- Paper towels
- Rubber gloves
- Lab notebook

6.0 Sample Preparation

1. Weigh out 0.2000 ± 0.0010 grams of heavy mineral separate (HMS) (refer to SOP XX) in a weighing dish on the lab micro-balance. Record this value in lab notebook.
2. Pour HMS into agate mortar and add a small (~5 ml) of acetone to mortar. Use pestle to grind HMS until no more crystallites are felt breaking and a smooth feel to the grinding is obtained. Wash material from edges of mortar into center and continue to grind sample. Consistency between samples is critical during this grinding procedure. This powder diffraction method is dependent upon as fine a sample grain size as possible, along with consistency amongst large numbers of samples.
3. Allow acetone to evaporate from mortar. While sample is drying, label backside of sample holder with appropriate notation for sample tracking. Also label a 1 ml sample vial with sample number.
4. When sample is completely dry, use metal lab spatula to scrape HMS from sides of mortar, taking care to not scrape any sample other than the technician is completely sure that it has been ground completely, i.e. do not include sample from high on the sides of mortar. Take care to break up any clumps of HMS and homogenize the sample as much as possible.
5. Remove a small amount of HMS and place into 1 ml sample vial to save for grainsize analysis.
6. Place a large glass slide onto micro-balance and tare the balance. Load vacuum grease into syringe and dispense .15 grams of grease onto large glass slide. It may be useful to mark the approximate volume of grease with a felt tip marker on the backside of the glass slide to aid in future sample preparation.
7. Pour HMS from mortar onto grease on large glass slide. Use small glass slide to mix the grease and HMS into a homogenous paste. It may be helpful to visualize a great artist mixing oil paints or perhaps a drywall worker mixing up wall spackling. It is important to ensure that the HMS and grease are properly homogenized into the grease.
8. Place a circular glass slide into the aluminum sample holder. Transfer the HMS/grease paste onto the putty knife. Use the putty knife to fill the space between the glass slide and the level of the sample holder rim with a wiping motion. The goal is to produce a sample holder filled with HMS/grease to exactly the same height as the rim of the sample holder. The final surface of the HMS/grease can be made extremely smooth and regular with good technique and a little practice.

9. Use Kimwipes to clean excess grease off the outer rim of the prepared sample holder.
Use Kimwipes to clean all equipment and maintain a clean working environment.

7.0 Sample Archiving

1. Prepare a small glass slide (1.5" x 1") with a small amount of temporary glue on one end and label other end with sample number using an indelible marking pen.
2. After successful XRD analysis, remove glass slide from aluminum sample with tweezers and place circular slide with HMS/grease onto the pre-glued glass slide.
3. Archive samples in sample drawers away from dust and heat. Circular glass slide can be removed from archive mount if necessary for reanalysis.
4. Assign a quantitative color number (Standardized Rock Color Charts) to each sample and record in lab notebook.