

STANDARD OPERATING PROCEDURE NO. 27**X-RAY DIFFRACTION ANALYSIS**

REVISION LOG		
Revision Number	Description	Date
27.0	Original SOP	Nov. 12, 2003
27.1	Revisions by V. Lueth after comments by G Robinson on Nov. 17, 2003, incorporate changes to DQOs by McLemore	November 18, 2003
27.2	Revisions by PJP	5/19/2004
27.3	Corrections to procedure made by KMD	2/12/2007
27v3	Final edit and finalizing by LMK and made ready to send to Jack Hamilton to post on website and G. Robinson for audit	3/23/07
27v4	Editorial by SKA	10/23/08

1. PURPOSE AND SCOPE

This document outlines the procedures for sample analysis using x-ray diffraction (XRD). The x-ray diffractograms will be used to identify minerals from rock pile materials, alteration scars, and rock samples. The types of products generated will include x-ray spectra and pattern matching tables including identification of minerals present. All pattern matching will be based on standard powder diffraction files.

2. RESPONSIBILITIES AND QUALIFICATIONS

The Team Leader and Characterization Team 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 accurately.

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 to receive specific training regarding these procedures if necessary. Personnel trained on the Rigaku Gieger-flex (or whichever XRD machine is used) will undertake the procedures outlined in this protocol. Only personnel trained in the use of MDI-JADE 6 (Reference XRD data reduction software) will be allowed to interpret the x-ray patterns. Personnel qualified to provide training in XRD equipment and software use include either the XRD lab manager or mineralogist.

All environmental staff and assay laboratory staff are responsible for reporting deviations from this SOP to the Team Leader.

3. DATA QUALITY OBJECTIVES

The x-ray diffractometer will be used to aid in achieving objectives 2, 3, 4 and 5 in the data quality objectives outlined by Virginia McLemore for the "Geological and Hydrological Characterization at the Molycorp Questa Mine, Taos County, New Mexico".

- Determine how mineralogy, stratigraphy, and internal structure of the rock piles contribute to weathering and stability. XRD analysis will be used to identify mineral phases within samples, both preexisting and authogenic.
- Determine if the sequence of host rock hypogene and supergene alteration and weathering provides a basis to predict the effects weathering can have on mine rock material. Sample analysis by XRD will allow for the identification of secondary minerals formed by both hypogene and supergene processes.
- Determine if cementation forms in the rock piles and the effect of such cementation on the stability of the rock piles. XRD analysis will confirm the presence of minerals identified optically or determine the identity of unknown minerals in cements.
- Determine the relative concentrations of pyrite and carbonate minerals so that a representative sample goes into the weathering cells. Quantitative XRD analysis will assist in determining the relative amounts of pyrite and carbonate minerals in any particular powdered sample.

4. EQUIPMENT LIST

- Rigaku Geiger-flex X-Ray Diffractometer (or similar XRD unit)
- Ceramic or agate mortar and pestle or mechanical sample powdering mechanism
- Glass slides or aluminum sample holder
- Indelible marker and tape or labels to mark samples
- Double stick cellophane tape for small samples
- Si standard for calibrating XRD machine

5. RELATED STANDARD OPERATING PROCEDURES

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

- SOP 1 Data management (including verification and validation)
- SOP 2 Sample management (including chain of custody)
- SOP 24 Petrographic analyses
- SOP 25 Stable Isotope analysis
- SOP 26 Electron microprobe analysis
- SOP 28 X-ray Fluorescence analysis
- SOP 30 ICP-OES analysis
- SOP 31 ICP-MS analysis
- SOP 44 Ar/Ar analysis

DATA COLLECTION:

Data collection is accomplished by recording an x-ray diffraction scan from 2 to 70 degrees 2θ on a goniometer equipped with a photomultiplier. The scan is stored electronically as a datascan file (.mdi) by the software program DataScan, an automated control and data acquisition system for x-ray diffractometers. Raw scans are archived on both a hard drive and on removable media (disks or cd). Hard copies of scans are also archived by the researcher. Detection limits for phase identification vary on procedures and types of materials. Standard powder diffraction procedures on rock samples can detect minerals down to 1 percent. Mineral separation prior to analysis and with favorable matrix materials can reduce the detection limit to 0.07 percent.

DATA REPORTING:

All datascan files (.mdi) are imported into JADE 6 data reduction software for x-ray pattern processing. X-ray diffraction peaks are indexed by computer or by hand by comparison to ICDD PDF standard powder diffraction patterns. Raw scans are converted into various types of reports, depending on the type of information required. Typical XRD identification reports consist of a raw scan, a Peak ID extended report (indexed peaks and matching phases in table form) after scan matching, and a Phase ID report (graphic) that summarizes the raw scan, background correction, and matching phases superimposed on the corrected scan. Electronic copies and hard copies of these reports will be archived until the project is completed. Quantitative XRD reports will consist of phases identified with any relative percentages generated in tabular form archived both electronically and as hard copy.

6. PREPARATION

1. Decide which type of sample preparation mount will be best suited to each sample. Large volumes will utilize standard milled aluminum sample holders. Small volumes will use either a sample slurry on glass slides or double stick cellophane tape for the lowest volume samples.
2. **Grinding Procedure:** Grind sample and/or standards in mortar and pestle or mechanical grinding device to a fine powder ($\sim 75\mu/0029$ mesh).
3. **Mounting Procedure:**

Large volume – pour powder into milled well on aluminum holder. Press powder with glass slide to cause powder to “stick” in well. Label holder with

sample number using tape and indelible marker. Enter sample number into XRD machine to identify electronic output. Analyze.

Small volume –

Tape method - pour sample onto double stick tape mounted on glass slide or smear sample slurry to a uniform thickness on a glass slide at appropriate height on slide (use aluminum holder to check tape height). If using a slurry, allow sample to dry before analyzing. Label slide with sample number using tape and indelible marker. Enter sample number into XRD machine to identify electronic output. Analyze.

7. ANALYTICAL PROCEDURES

1) At the start of the day, check the calibration of the XRD machine with a silicon (Si) standard. Mount the silicon standard in the XRD. Go to Controller in DataScan. Under Controller, go to Scan Line. Make sure the silicon standard peak will be scanned (make sure the scan range is set so the Si peak will be covered). After the machine has stopped, it will display a single peak with a vertical line. The vertical line is the position of the Si (111) peak where it appears according to the computer. This should match the peak the XRD found. If the two don't match, you will have to move the vertical line to calibrate the XRD to the Si standard. The computer will ask if you want to change the set angle to the new value. If the new value is okay, press the YES button with the cursor, if not, press the NO button and move the vertical line until it matches the peak.

2) Fill in the File ID and Scan ID. File ID is the name of the computer file the data will be stored in and Scan ID is the sample number or name.

3) Scan from 2.0 degrees to 35.0 degrees 2θ , with an s stepsize of 0.03 degrees and a dwell time of 0.5 seconds. Always scan with the axes coupled

C. INSTRUCTION FOR USING JADE

1) Open powder pattern(s) saved from the Datascan analysis

2) Select background correction or background edit to remove or modify background pattern

3) Select scan match, set to Minerals, to match pattern to known powder patterns.

4) Select Peak ID extended to print table of peak match data.

5) Select Phase ID report to print graphic file of scan and superimposed match spectra

6) Save manipulation files in appropriate directory.

8. SAMPLE HANDLING

Once samples have been run, the remaining powder should be stored in numbered and sealed vials. Samples will be archived at least until the end of the project.

9. REFERENCES

Rigaku, Datascan, and Jade manuals