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USGS TECHNICAL PROCEDURE GCP-07, R1
USGS - Yucca Mountain Project

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USGS TECHNICAL PROCEDURE GCP-07, R1

Mineral Separation for Geochemistry and Isotopic Analysis

1.0 PURPOSE.

- 1.1 To assure the accuracy, validity, and applicability of the methods used for the preparation of a mineral separation, this procedure provides a guide for USGS personnel and contractors to perform the described activity. From this procedure, the Department of Energy (DOE) and the Nuclear Regulatory Commission (NRC) can evaluate these activities for meeting requirements of the NNWSI Project, and competent, trained personnel can reproduce the work.
- 1.2 This procedure describes the components of the work, the principles of the methods used, and their limits. It also describes the detailed methods to be used for calibration, operation and performance verification of any equipment. In addition, it defines the requirements for data acceptance, documentation, and control; and it provides a means of data traceability.

2.0 SCOPE OF COMPLIANCE.

- 2.1 This procedure applies to all USGS personnel and their contractors who may perform work referred to in Para. 1.1, or use data obtained from this procedure if it is deemed to potentially affect public health and safety as related to a nuclear waste repository.
- 2.2 All data derived from this procedure that are presented to support licensing of the NNWSI Project repository, and any equipment calibrations or recalibrations that may be required shall be in accordance with this technical procedure. Variations are allowed only if and when this procedure is formally revised, or otherwise modified, as described in Section 8.

- 3.0 PERSONNEL RESPONSIBILITIES. The Principal investigator (PI) is responsible for assuring full compliance with this procedure. Per QMP-2.02 and QMP-2.03, the PI shall require that all personnel assigned to work under this procedure shall have the necessary technical training, experience, and personal skills, to adequately perform this procedure; and they shall have a working knowledge of the USGS QA Manual. Responsibilities of others including the reviewer(s), contributing investigators, Branch/NHP Chief, QA Office and the Chief, Branch of NNWSI are as described in Para. 4.3, QMP-5.01.

- 4.0 DETAILED PROCEDURE. Determination of the ages and conditions of formation of various minerals in rocks or of secondary precipitates is needed in order to evaluate possible future tectonic events, erosion rates, climatic changes, hydrologic pathways, and rock-water interaction in the area. This procedure outlines the techniques by which minerals, glass, and

amorphous precipitates can be separated for subsequent isotopic, geochemical, or fission-track analyses.

4.1 Objective: The separation of mineral grains of one kind from another is done by taking advantage of differences in physical properties, such as specific gravity or magnetic susceptibility.

4.2 Methods Used:

1. Geologist should prepare a thin-section of a rock sample and examine it for the specific mineral to be separated prior to sending samples to mineral separation. This section preparation can be by any method which yields a section thin enough that mineral grains can be identified with a petrographic microscope.
2. Crush rock in crusher to 6 inch fractions. Pretreatments such as disaggregation, removal of adhering clay, or acid (~10% HCl) rinse of CaCO_3 or MgCO_3 may be necessary before crushing.
3. Adjust pulverizer to particle size desired and pulverize the crushed rock. Pulverizer size is determined by mineral which is looked for and by experience.
4. Perform first sizing using a sieve size that is compatible with the pulverizer size. Save the oversized material for the geologist's use.
5. Previously used heavy liquids need to have the acetone in them removed before they can be used again. Mix water and heavy liquid 1:1 by volume in a heavy liquid flask and shake vigorously by hand. Pour off water and acetone down to the heavy liquid. Repeat 3 times to remove the acetone and return the heavy liquid to the correct specific gravity.
6. Add an appropriate amount of crushed rock sample to the filter assembly consisting of a glass separatory funnel fitted with appropriate filter paper, and wash sample with the appropriate heavy liquid. A clean separation of biotite and hornblende can be made using methylene iodide (G=3.15-3.20). Bromoform (G=2.85), tetrabromoethane (G=2.96), or their mixtures with varying amounts of acetone are used to separate muscovite, feldspar, apatite, and other rock-forming minerals and glass according to specific gravity. Calcite (G=2.71) can be separated from opal (G=2.0-2.2) by use of appropriate dilutions of heavy liquids.
7. Rinse sample with acetone and allow to air dry.
8. If sample has heavy cross-contamination it is returned to the geologist for preliminary cleaning. Upon return repeat Steps 5, 6 and 7.

9. A ball mill can be used to further separate stubborn grinds such as mica-rich composite grains. Set up is determined by the researcher's experience with mineral separation.
10. Refine the sample according to mineral sought. Magnetic separation and dielectric separation are used to separate magnetic from non-magnetic minerals. The sample is fed down a track through a magnetic or dielectric field, and magnetic minerals are separated from non-magnetic minerals.
11. Repeat Steps 5, 6, and 7 to increase mineral purity.
12. Place mineral sample in glass vial. Ensure that all containers used are properly labeled with the sample number for traceability. Labeling will be done with indelible ink and recorded accurately to maintain traceability from sample preparation through data recording as per GCP-02, R1 (see Section 6.0).
13. Traceability shall be achieved by recording or labeling each sample processed from the place of field collection through separation and disposition.

4.3 Alternative Method(s) Considered: No alternative methods were considered; however, any procedure which provides pure separates of the minerals of interest will be considered acceptable provided the alternate procedure is adequately described and documented.

4.4 Materials/Equipment Required:

- o Petrographic microscope
- o Acetone
- o Methylene Iodide
- o Bromoform
- o Tetrabromoethane
- o Glass vials
- o Distilled water
- o Rock crusher
- o Rock pulverizer
- o Tyler sieves 28-150 mesh
- o Magnetic separator
- o Dielectric separator
- o Ball mill
- o Hydrochloric acid (HCl), 10%
- o Filter paper: 24 cm (Whatman #2)
18.5 cm (Whatman #541)
12.5 cm (Whatman #1)
- o Glass separatory funnels

4.5 Assumptions Affecting the Procedure: The method assumes that minerals can be separated by heavy liquids of a given density because minerals themselves have different densities.

4.6 Data Information: Data from this procedure consists of a list of final prepared samples, labeled, and identified according to NWM-USGS technical procedure GCP-02.

4.6.1 Quantitative/Qualitative Criteria - Purity of minerals separated can be checked by use of a petrographic microscope or by X-ray diffraction analysis.

4.7 Limitations: Limitations of this procedure are: (a) the performance of the mechanical devices, and (b) the skill, experience, and care of the operator in performing mineral separations.

4.8 Other: This procedure interfaces with NWM-USGS technical procedures GCP-02, GCP-03, GCP-06, GCP-08, GCP-12, and GCP-13.

5.0 CALIBRATION REQUIREMENTS. Calibration is not required as a part of this technical procedure. When calibrations are required, all instruments and methods when applicable, will be calibrated in compliance with the Instrument Calibration Procedure (NNWSI-USGS-QMP-12.01) prior to obtaining data that will be cited to support licensing the NNWSI Project.

6.0 IDENTIFICATION AND CONTROL OF SAMPLES. Samples will not be collected as part of this procedure. (However, samples that were collected by other technical procedures given in Para. 4.8 will be used in this procedure.)

6.1 Sample Identification: As part of the data records and documentation, and in compliance with QMP-8.01, all samples will be identified per NWM-USGS technical procedure GCP-02.

6.2 Control and Storage: In compliance with QMP-8.01, the collected and identified samples shall reside in the custody of a project scientist or technician who shall store them until used in analysis.

6.3 Special Treatment: None.

7.0 QUALITY ASSURANCE RECORDS. All information collected and recorded under this procedure that is to be used in support of the NNWSI Project licensing process is required to be a part of the official USGS record. Input needed to process the information as a record includes: title or description, subject, originator, date of the document, and whether it is an original, a revision or an addendum.

Specific items from this procedure that will constitute a record are laboratory data sheets.

7.1 Notebooks or other organized documentation will be prepared as appropriate by the PI or a contributing investigator to record data from this procedure and shall include any information considered by the originator to be pertinent. When data are kept in loose-leaf form, each page will be numbered consecutively and chronologically. All documents will be signed or initialed and dated by the investigator on a daily basis when entries are made. Any revisions will be lined out, initialed, and dated.

7.2 All data collected and the applicability of methods used in this procedure will be reviewed and cosigned by a peer or supervisor of the investigator knowledgeable with the objectives of this procedure in accordance with NNWSI-USGS-QMP-6.01, Para. 4.2.2; and as such are acknowledged by both the investigator and the reviewer to be acceptable and meaningful data that meet appropriate quantitative and qualitative acceptance criteria. Unacceptable data shall be identified appropriate to the form of the data.

8.0 MODIFICATIONS. When field modifications become necessary, per Para. 4.8, QMP-5.01, the PI shall fully document the changes, submit the documentation for the same review signature and distribution process as for the original procedure, and indicate whether the change should result in a subsequent revision to the technical procedure. The documentation will be reviewed within 30 days.

9.0 REFERENCES CITED.

NWM-USGS-GCP-02, R1 Labeling, Identification, and Control of Samples for Geochemistry and Isotope Geology.

10.0 ATTACHMENTS. There are no attachments included with this technical procedure.

11.0 APPROVAL. This technical procedure shall become effective upon its approval as noted by completion of all the following signatures and dates.

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5/23/88
Date

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