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Identifier:	Revision:	Effective Date:	Review Date:
SOP-09.05	1	04/11/01	12/16/2003

ER Document Catalog Number: **ER2001-0148**

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environmental restoration project

A Department of Energy Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Clay Mineral and Zeolite Separation

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NATIONAL LABORATORY
Los Alamos, New Mexico 87545

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Revision Log

Revision No.	Effective Date	Prepared By	Description of Changes	Affected Pages
R0	3/16/92	David Bish	New procedure	all
R1	04/11/01	David Vaniman	Updated to incorporate processes that had been in SOP 9.06 (Zeolite Purification and Separation)	all
Review	12/16/2003	Mark Thacker	Deemed process adequate.	All

Clay Mineral and Zeolite Separation

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Clay Mineral and Zeolite Separation

1.0 PURPOSE

This SOP describes the process for clay mineral and zeolite separation from geological samples at the Los Alamos National Laboratory (Laboratory) ER Project. The separation process is based on sedimentation.

2.0 SCOPE

This SOP is a manadatory document and shall be implemented by all ER Project participants when performing clay mineral or zeolite separations for the ER Project

3.0 TRAINING

3.1 All users of this SOP are trained by self-study, and the training is documented in accordance with QP-2.2.

3.2 The Geology Task Leader (TL) will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with QP-2.2.

4.0 DEFINITIONS

Note: A glossary of definitions can be located on the ER Project internal homepage *http://erinternal.lanl.gov.*

4.1 *Clay Mineral*—One of a complex and loosely defined group of finely-crystalline hydrous silicates. Common clay minerals at the LANL site include illite-smectites with a basic structure of two tetrahedral sheets on either side of an octahedral sheet, or kaolinite with one tetrahedral sheet to each octahedral sheet.

4.2 *Zeolite*—One of a group of silicate minerals analogous in composition to feldspars but with an open structure that permits cation exchange. The zeolite mineral clinoptilolite has been found at the LANL site.

5.0 BACKGROUND AND PRECAUTIONS

This procedure is based on Stokes Law settling of particles in solution. Clay- or zeolite-rich samples can be purified or extracted from a sample by means of sedimentation in water. This method relies on differences in particle sizes and densities of mineral components in a sample.

Precautions in the use of this procedure include the use of only de-ionized or distilled water to wash and disaggregate the samples, in order to avoid cation-exchange effects in the concentrated clays or zeolites. For safety purposes, the ultrasonic probe used in disaggregation is operated in a sound-dampening box to protect the hearing of the operator.

6.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

6.1 Focus Area Leader

6.2 Geology Task Leader

6.3 Quality Program Project Leader

6.4 Author

6.5 ER Project personnel

7.0 EQUIPMENT

The equipment used in this procedure is listed below.

7.1 *Shatterbox* — A shatterbox or comparable equipment (e.g., ballmill) can be used to powder solid samples before ultrasonic disaggregation.

7.2 *Ultrasonic Probe* — An ultrasonic probe capable of operation at 200 W is used to separate mineral grains.

7.3 *Centrifuge* — A centrifuge capable of operation at 8000 RPM is used to sediment fine particles.

8.0 PROCEDURE

Note: Subcontractors performing work under the ER Project's quality program may follow this standard operating procedure (SOP) for clay-minera and zeolite separation or may use their own procedure(s) as long as the substitute meets the requirements prescribed by the ER Project Quality Management Plan, and have been approved by the ER Project's Quality Program Project Leader (QPPL) before the commencement of the activitie(s).

Note: ER Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

Note: Deviations from SOPs are made in accordance with QP-4.2, Standard Operating Procedure Development and documented in accordance with QP-

5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

8.1 Samples of loose soil or fine, uncemented sediment may be processed without grinding in a shatterbox. Most samples, however, must be crushed before individual minerals can be separated by sedimentation.

8.2 Samples that require processing in a shatterbox or other crusing device should be ground for at about 1-2 minutes to break the sample down to a particle size comparable to that of its constituent phases. Sample size will depend on the abundance of clays or zeolites in the original material. For enriched materials a few grams may suffice; clay- or zeolite-poor materials may require several tens of grams of the parent material.

8.3 The powdered sample or the original fine-grained material is placed in ~700-800 ml of distilled or deionized water in a 1000 ml plastic beaker. This sample is disaggregated for ~10-20 minutes using an ultrasonic probe operated at ~200 W. Longer times may be used if the sample is difficult to disaggregate. Smaller samples can be processed using appropriately scaled beakers.

8.4 Remove and cover the beaker. Place the covered beaker on a vibrationally stable surface without cooling it (the ultrasonic probe generates heat in the suspension) and allow the suspension to settle for 30 to 60 seconds. The sediment is composed of the coarse fraction (> ~20-30 μm) which includes most of the quart, feldspar, and other unwanted minerals, as well as any larger aggregates that were not crushed to component mineral size. The sediment may be discarded or saved for other analyses.

8.5 Decant or syphon the supernatant into a second beaker and allow it to rest on the vibrationally stable surface for ~1 hour, to settle out the ~20-3 μm size fraction. In zeolite-rich samples, this will generally be a fairly pure zeolite fraction.

8.6 Decant or syphon the remaining supernatant into a third beaker and allow it to rest on the vibrationally stable surface overnight (15-20 hours) to settle out the ~3-1 μm size fraction. This fraction may also be a rather pure zeolite sediment if the host rock is zeolite-rich.

8.7 The remaining supernatant will contain most of the clay fraction. Place this supernatant in centrifuge tubes and centrifuge at ~5000 RPM for ~5 minutes to separate the ~2-0.35 μm size fraction. This sediment may be analyzed by XRD to determine the purity of clay separation. If further processing is indicated for clay separation, proceed to step 8.8.

8.8 Decant or syphon the supernatant from the first centrifugation, place in clean centrifuge tubes, and centrifuge at ~8000 RPM for 40 to 60 minutes. The sediment collected after this process will consist of the ~0.35-0.1 μm size

fraction. This sediment may be analyzed by XRD to determine the purity of clay separation. If further processing is indicated for clay separation, proceed to step 8.9.

8.9 Further processing may consist of placing the residual supernatant on a low-temperature hotplate to evaporate the liquid, or further centrifuging the sample at ~15000 RPM for ~1 hour.

8.10 If a clay sample is collected, an oriented mount for XRD analysis can be prepared by suspending the sample in ~10-20 ml of deionized water, placing some of this suspension on a dry glass or quartz slide, and setting the slide on a warm surface to evaporate the suspension.

8.11 Lessons Learned

During the performance of work, ER Project personnel shall identify, document and submit lessons learned in accordance with QP-3.2, Lessons Learned. This QP can be located at: http://erinternal.lanl.gov/home_links/Library_proc.htm.

9.0 REFERENCES

ER Project personnel may locate the ER Project Quality Management Plan/ER Project QP requirements crosswalk at http://erinternal.lanl.gov/home_links/Library_proc.htm.

The following documents have been cited within this procedure:

QP-2.2, Personnel Orientation and Training

QP-3.2, Lessons Learned

QP-4.2, Standard Operating Procedure Development

QP-4.3, Records Management

QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities

10.0 RECORDS

The Geology Task Leader (TL) is responsible for submitting the following records (processed in accordance with QP-4.3) to the Records Processing Facility.

10.1 Data submittals for the ER electronic database.

10.2 Notebook records of the sample handling and results of analysis relevant to production of X-ray diffraction data.

Using a token card, click here to record "self-study" training to this procedure.
If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.