OPERATING PROCEDURE FOR GOLD COATING SAMPLES USING THE TECHNICS HUMMER II SPUTTERING SYSTEM

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Roland C. Hagan
Preparer

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Date

Marjorie Snow
Reviewer

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Date

Henry Paul Nunes
Quality Assurance Project Leader

4/10/89
Date

R. J. Herbst
Technical Project Officer

4/12/89
Date

OPERATING PROCEDURE FOR GOLD COATING SAMPLES USING THE TECHNICS HUMMER II SPUTTERING SYSTEM

1.0 PURPOSE

This procedure provides instructions for gold coating samples using the Technics Hummer II Sputter Coater.

2.0 SCOPE

This procedure applies to any Los Alamos Yucca Mountain Project (LA YMP) sample to be gold coated for study using the Scanning Electron Microscope.

3.0 PRINCIPLE

All samples to be analyzed or imaged using the Scanning Electron Microscope must be coated with a conductive material prior to analysis.

4.0 PROCEDURE

4.1 Remove Bell Jar by holding glass and twisting slightly.
4.2 Place specimen on Sample Stage.
4.3 Replace Bell Jar.
4.4 Gently close both Pressure Control Knobs by turning them clockwise.
4.5 Turn on Power Toggle Switch.
4.6 Wait until Vacuum Gage Meter indicates 70 millitorr or less. This may require 2 to 15 minutes depending on the rate of sample outgassing.
4.7 Open main Argon Gas Valve (silver handle) and small valve (#3), located on Argon Gas bottle.
4.8 Turn Increase-Decrease Valve counter-clockwise until Vacuum Gage Meter indicates 500 millitorr or greater. Wait about 1 minute. This will flush the chamber.
4.9 Turn Increase-Decrease Valve clockwise until Vacuum Gage Meter indicates 70-80 millitorr.
4.10 Turn Mode Switch to Plate.
4.11 Switch Process Control to Manual.
4.12 Verify that Volts Knob is fully counter-clockwise at 0.
4.13 Turn High Voltage Switch ON.
4.14 Slowly turn the Volts Knob to indicate 11. Watch the DC Milliampere Meter and maintain 10 milliamps by adjusting the Increase-Decrease Valve. Maintain 10 milliamps for 1 minute or refer to Table 1 in the Technics Hummer Operation and Maintenance Manual for Time vs. Thickness per Milliampere Setting.
4.15 Turn Volts Knob to 0.
4.16 Turn High-Voltage Switch to OFF.
4.17 Gently close (clockwise) the Increase-Decrease Knob.
4.18 Open the ATM Knob by turning it counter-clockwise about one quarter turn and quickly turn Main Power OFF.
4.19 Enter into the QA Notebook the sample names or numbers, date and time of coating, length of time sample was coated, and name of person performing the coating. Sign the entry.

4.20 Remove the Bell Jar by holding the glass and slightly twisting it.
4.21 Remove sample.
4.22 Replace Bell Jar and leave it at atmosphere.
4.23 Close Argon Gas Bottle Valve and small valve (#3).
4.24 Suitable environmental conditions require that this procedure be performed in a clean work area

5.0 QUALITY ASSURANCE

5.1 Personnel
Only YMP certified persons will perform gold coating of YMP samples. Training for this procedure consists of reading the detailed procedure and performing the procedure under the supervision of a trained person. The preparer of this detailed procedure is considered trained to perform this procedure and to train others. Evidence of training and certification shall be documented in accordance with the YMP Personnel Certification Procedure.

5.2 Calibration
No calibration is required for this procedure.

5.3 Documentation
All samples receiving a gold coating shall be entered into a YMP controlled logbook. Logbook entries shall include complete sample ID, date and time of receipt, name of the responsible investigator, name of the person performing this procedure, and the date and time the procedure was performed. Any procedural deviations will be recorded in this logbook and/or in the investigator's notebook. All entries shall be signed.

5.4 Accept/Reject Criteria
Adherence to this procedure should result in a satisfactory coating, however, if charging effects are noticed during examination with the SEM, then this procedure may be repeated. Refer to Table 1 in the Hummer Operators Manual for suggested time per milliampere information.

5.5 Responsibilities
The laboratory custodian is responsible to see that this procedure is followed correctly. This person is also responsible for the proper care and use of the equipment. The custodian may delegate these responsibilities to a YMP person certified to perform this procedure.

5.6 Storage Requirements
5.6.1 All YMP samples received for gold coating will be stored in a locked YMP designated cabinet in the sample preparation lab from the time of receipt until they are retrieved by the responsible investigator.
5.6.2 Samples will be tracked, handled, and stored in accordance with the YMP procedure for Sample Identification and Control for Mineralogy-Petrology Studies (TWS-ESS-DP-101).
5.6.3 There are no specific storage requirements for the equipment used in this procedure.

5.7 Potential Sources of Error
A gold coating that is too thick will result in poor imaging using the Scanning Electron Microscope.

6.0 REFERENCES

6.1 Technics Hummer II Operation and Maintenance Manual
6.2 TWS-ESS-DP-101: Sample Identification and Control for Mineralogy-Petrology Studies.

METTLER AE100 OPERATING PROCEDURE
(X-Ray Fluorescence Analysis Sample Weighing Procedure)

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Roland C. Hagan — 20 April 1989
Roland C. Hagan, Preparer — Date

David A. Mann — 4-20-89
David A. Mann, Technical Reviewer — Date

Henry Paul Nunes — 4/24/89
Henry Paul Nunes, Quality Assurance Project Leader — Date

Karen A. Westfall — 4/26/89
R. J. Herbst, Technical Project Officer — Date

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METTLER AE100 OPERATING PROCEDURE
(X-ray Fluorescence Analysis Sample Weighing Procedure)

1. PURPOSE

This procedure describes the process for weighing geologic samples in preparation for fusing XRF samples.

2. SCOPE

This procedure applies to all samples prepared for X-Ray Fluorescence (XRF) analysis for the Yucca Mountain Project (YMP).

3. PROCEDURE

Sample Drying Procedure

3.1 After the sample is ground to a powder in the shatter box (TWS-ESS-DP-53), it must be dryed in an oven before being weighed in preparation for fusing.

1) Remove the lid from the sample, making sure that both the lid and glass bottle have the sample number written on them.
2) Place the open glass bottles in the drying oven for a minimum of 24 hours at 110°C.
3) Log the samples in the sample drying logbook (YMP controlled).
4) After 24 hours the samples may be removed and the caps replaced (CAUTION: be sure the lids are not mixed). Place the capped bottle in a dessication jar and cool to room temperature before weighing.
5) Before pouring sample into a graphite crucible, scratch the fusion number on the inside bottom face of the crucible. Blow the graphite dust from the crucible.

3.2 Care of Balance

3.2.1 This is a delicate instrument designed to weigh within a tolerance of 0.1 milligrams. Clean the interior of the weighing box by opening both sliding doors and gently, using a duster, blow all loose material away. If the pan is dirty, remove and clean with alcohol.

3.2.2 Do not attempt to weigh items greater than 100 grams. Place items on the pan with tweezers or tongs when possible. Do not drop items to be weighed on the pan, but place them gently on the pan.

3.2.3 Cleanliness is important. Clean all spatulas and spoons with Kimwipes and alcohol, and place utensils on a clean tissue next to the balance.

3.3 Care of Samples

3.3.1 Great care shall be exercised not to mix or contaminate samples. All subsequent analyses rely on this procedure. Ensure that all crucibles and spatulas are clean and dry before each use.

3.3.2 Carefully arrange all crucibles next to labeled sample bottles so that samples cannot be mixed up.

3.3.3 Only graphite crucibles procured for YMP use are acceptable for this procedure.

3.4 Use of program "SCALE."

3.4.1 Turn on IBM-PC.

3.4.2 Type SCALE.

3.4.2.1 Editing functions are available through the arrow keys. The left and right arrow keys allow nondestructive editing of a current line. The up and down arrow keys allow movement to the previous or next menu option; data is not changed.

3.4.3 Follow the instructions on your screen for performing daily calibration of the AE100 scale.

3.4.4 Press enter when you are finished calibrating.

3.4.5 <Operator> is prompting for the persons name doing the weighing.

3.4.6 <File Name> is prompting for the File ID listed in the upper right hand corner of the XRF submittal sheet.

3.4.7 <Fusion#> is prompting for the fusion number from the XRF weighing logbook, also found on the XRF submittal sheet.

3.4.8 <Sample id> is prompting for the sample YMP identifier from the XRF submittal form.

3.4.9 <Instruction> is prompting the operator to place the graphite crucible (CR-40) on the weighing pan.

3.4.10 Press enter after placing crucible on pan, the program will now tare the scale.

3.4.11 <Instruction> will now display "Add 9 grams of dried flux ± 0.005". Add flux to the crucible until you make the tolerance and close the balance door to check the final weight. When the scale is stable, press RETURN. The program will beep to signify 9 grams ± 0.005 and then tare itself.

3.4.12 <Instruction> will now display "Add 1 gram of dried sample ± 0.005." Add sample to the crucible until you make the tolerance and close the balance door to check the final weight. When the scale is stable, press RETURN. The program will beep to signify 1 gram ± 0.005.

3.4.13 The cursor will now return to 3.4.7 prompt and the cycle is repeated until all samples are weighed.

3.4.14 When all samples are weighed for this session at 3.4.7 press the <ESC> key. All files will be closed and saved to disk.

3.4.15 All weighing results are appended to the operating system file so as not to erase any previous results.

4. QUALITY ASSURANCE

4.1 Personnel

Only YMP certified personnel may weigh samples using this procedure. Training for this procedure consists of reading the written detailed procedure and performing the procedure under the supervision of a trained person. The preparer and technical reviewer of this detailed procedure are considered trained to perform the procedure and to train others. Evidence of training and certification shall be documented in accordance with the YMP Personnel Certification Procedure.

4.2 Documentation

All calibration checks are to be recorded in the YMP laboratory logbook. File name, name of operator, sample ID, fusion #, weight of flux, sample weight and date and time of mixture are automatically recorded by program "SCALE".

4.3 Storage
When samples are not in use, they are to be covered and stored and locked in an appropriately labeled place. They are to be kept separate from all other samples. There are no storage requirements for any instruments used in this procedure.

4.4 Sample Traceability
Samples will be tracked in accordance with the procedure for Sample Handling and Control for Mineralogy-Petrology Studies (TWS-ESS-DP-101).

4.5 Accept/Reject Criteria
Adherence to this procedure produces acceptable results. The computer software has the proper weighing tolerances built in and will not proceed to next step until tolerance is achieved. Flux or sample is added or removed until the proper weights are achieved.

4.6 Potential Sources of Uncertainty and Error
Use of the computer software effectively eliminates any source of uncertainty in the weighing process. The balance is situated away from excessive drafts and free from vibrations as these may lead to inaccuracies in weighing.

4.7 Procedural Deviations
Any procedural deviations are to be recorded in the controlled YMP logbook.

4.8 Responsibilities
Samples are submitted for x-ray fluorescence analysis in accordance with ESS-DP-111 (Procedure for XRF Analysis).

The thin section lab supervisor is responsible to see that this procedure is followed correctly. This person is also responsible for the proper care and use of the equipment and to see that all calibrations are up to date. This person may delegate these responsibilities to a YMP certified person.

4.9 Calibration
- 4.9.1 Calibration of the balance is performed yearly.
- 4.9.2 Weights are recalibrated and certified every two years.
- 4.9.3 Certificate of calibration and calibration results for the balance and the weights will be documented in accordance with the procedure for NNWSI Instrument Calibrations (QP-12.1).
- 4.9.4 Calibration of the balance is also checked at each use (see step 3.4.3).

5. REFERENCES

5.1 The Mettler A100 Operating Manual.
5.2 TWS-ESS-DP-101: Sample Identification and Control for Mineralogy-Petrology Studies.

CLAY MINERAL SEPARATION AND PREPARATION FOR X-RAY DIFFRACTION ANALYSIS

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David L. Bish
Preparer

1/4/89
Date

Steve J. Chipera
Technical Reviewer

1/4/89
Date

Henry Nunes
Quality Assurance Project Leader

1/12/89
Date

D. T. Oakley
Technical Project Officer

1/12/89
Date

CLAY MINERAL SEPARATION FOR X-RAY DIFFRACTION ANALYSIS

1. PURPOSE

The purpose of this procedure is to describe separation of clay minerals from bulk rock samples and preparation of oriented sample mounts of clay minerals for analysis by x-ray diffraction.

2. SCOPE

The procedure applies to separation and x-ray diffraction investigation of clay minerals for the Yucca Mountain Project.

3. PRINCIPLES

This procedure is based on Stokes Law settling of particles in a solution.

4. DEFINITIONS

None Applicable.

5. RESPONSIBILITIES

The principal investigator (PI) has the responsibility to assure implementation of this procedure for clay mineral separations being conducted for the Yucca Mountain Project. The PI may delegate performance of the procedure to any duly certified individual.

6. PROCEDURE

6.1 Overview

6.1.1 Equipment and Software Used

- o Shatterbox (or large ball mill) -- most all acceptable.
- o Ultrasonic probe capable of ~200 W.
- o Centrifuge capable of 8000 RPM with a Sorvall GSA head.
- o Sorvall GSA centrifuge head.
- o Sorvall SS-34 centrifuge head (optional).
- o Spray Drier (optional).
- o 1000 ml beakers.
- o No softvare is used.

6.1.2 Critical Laboratory Setup Parameters

None Applicable.

6.1.3 Special Environmental Conditions

The use of de-ionized or distilled water to wash and disaggregate the samples is necessary to ensure that no chemical alteration of the clays occurs due to cation exchange.

6.2 Cleanliness

Thoroughly clean all sample preparation equipment and work areas before use.

6.3 Traceability

6.3.1 Complete records shall be kept in a controlled YMP notebook, including full sample identification, date of preparation, and signature of person preparing each sample.

6.3.2 Great care shall be exercised not to mix or contaminate samples. All subsequent analyses rely on this procedure.

6.3.3 Carefully label containers into which the pulverized sample will be placed and keep rocks with their labeled bags and containers. Label side and top of each container so that tops cannot be switched.

6.4 Set Up for Separation

6.4.1 Obtain approximately a one-to-two-inch slice of drill-core or an equivalent amount of drill cuttings or bulk-rock.

6.4.2 Rid the sample of surface contamination using one of the following:

- o Brisk strokes of a clean, dry bristled brush.
- o Rinse with de-ionized or distilled water and dry thoroughly -- preferably at room temperature.

6.4.3 Break the bulk sample down into smaller pieces (less than 0.25 inch in diameter using:

- o A Platiner mortar and pestle or,
- o A mortar and pestle/hydraulic press apparatus.

6.4.4 Powder sample using either a shatterbox or large ball-mill.

6.4.5 Combine the entire powdered sample and mix thoroughly to ensure homogeneity. Place in a bottle properly labeled with the sample identification.

6.5 Separation

6.5.1 Take approximately 20-30 grams of the powdered sample and suspend it in approximately 500 to 700 ml of deionized or distilled water contained in a large plastic (~1000 ml) beaker. Disaggregate the sample for approximately 10-20 minutes at ~200 W using an ultrasonic probe.

6.5.2 Remove the beaker containing the suspended sample from the ultrasonic probe enclosure and cover the beaker. Place the beaker on a vibrationally stable surface and allow the sample to settle gravitationally without interruption ideally for a time equal to 47 minutes per cm depth of water. This will allow sedimentation of all particles greater than 2μm diameter. For a typical 14 cm water depth, total settling time should be about 10.9 hours. Overnight settling using a water depth of 14 cm will result in sedimentation of slightly smaller particles, e.g., 15 hours settling will sediment particles >1.75 μm and 20 hours settling will sediment particles >1.5 μm.

6.5.3 Decant or syphon the supernatant from the beaker into large centrifuge tubes, taking care not to disturb the sediment at the bottom of the beaker. The sediment in the beaker may be discarded.

6.5.4 Place the centrifuge tubes in the centrifuge and separate the ~2.0-0.35 μm size fraction from suspension at a centrifugation speed of ~5000 revolutions per minute for a spin time of ~5 minutes. (Refer to Sorvall Centrifuge Instruction Manual for Superspeed Angle Rotors: GSA and SS-34.)

6.5.5 Remove the centrifuge tubes from the centrifuge and decant the supernatant into large receiving centrifuge tubes, taking care not to disturb the sediment (>0.35 μm in diameter) at the base of the tube. Save this sediment fraction if needed.

6.5.6 Place the centrifuge tubes containing the cloudy supernatant in the centrifuge and separate the fine clay fraction (~0.35-0.10 μm in diameter) at a centrifugation speed of ~8000 revolutions per minute for a spin time of 40 to 60 minutes.

6.5.7 Remove the centrifuge tubes from the centrifuge and decant the supernatant into labeled beakers.

6.5.8 Remove the fine clay sediment fraction (~0.35-0.10 μm in diameter) from the base of the centrifuge tubes using a spatula and, if necessary, a small amount of deionized or distilled water. Save in a properly labeled sample bottle.

6.5.9 There are several options for processing the remaining supernatant material from step 6.5.7, (this supernatant contains the <0.10 µm fraction). The options are as follows:

1: If not needed, it may be discarded.
2: Place the beakers on a low-temperature hotplate or warm surface to evaporate/concentrate until it will fit into a labeled sample bottle.
3: It can be further centrifuged using the Sorvall SS-34 head at a speed of ~15000 RPM for ~1 hour to remove some of the remaining clays.
4: It can be run through a spray drier which evaporates the liquid and allows the clay to be collected.

6.6 Preparation of Oriented Mounts

6.6.1 Suspend a clay mineral fraction in a small amount (approximately 10 to 20 ml) of deionized water contained in a small beaker.

6.6.2 Thoroughly clean and dry a glass or quartz slide appropriately sized for the diffractometer sample holder being used, and place it on a level, stable, warm surface. The sample number is written with indelible ink on the back of the slide.

6.6.3 Drop the suspended clay sample, using an eye dropper or pipette, onto the slide using only enough sample to cover the slide. Do not overfill the slide and do not add additional suspension after the suspension on the slide has begun to dry to ensure the best orientation of clay particles possible.

6.6.4 Allow the sample to dry undisturbed on the slide.

6.6.5 The sample is now ready for x-ray diffraction analysis (refer to Siemens X-ray Diffraction Procedure, TVS-ESS-DP-16).

6.7 Data Analysis

Not Applicable.

7. QUALITY ASSURANCE

7.1 Personnel

Only those persons certified in accordance with the Los Alamos YMP Personnel Certification Procedure shall perform YMP related clay mineral separations and preparations.

7.2 Calibration

No calibration of equipment is necessary for this procedure.

7.3 Records

Full records of a clay separation, including full sample name or number, date of separation, and signature of the person separating each sample are to be recorded in a controlled YMP notebook.

7.4 Accept/Reject Criteria

7.4.1 Adherence to this procedure results in an acceptable sample for x-ray diffraction analysis. The purity of the individual samples and their size fractions are determined by x-ray diffraction. It is up to the person conducting the separation to determine if the clay fractions are of adequate purity for their work.

7.4.2 There are no real potential sources of error since this is a qualitative procedure.

7.4.3 The notebook entry for a sample shall constitute evidence that the procedure has been implemented and satisfactorily accomplished for that sample.

7.5 Procedural Deviations

Deviations from this procedure shall be fully documented in the controlled YMP notebook of the person conducting the work and shall explain the deviation and the effects it may have on the resulting work.

7.6 Storage, Shipping, and Handling

7.6.1 Samples will be tracked, handled, shipped, and stored in accordance with the procedure for Sample Identification and Control for Mineralogy-Petrology Studies (Ref 8.3).

7.6.2 The equipment require no special handling, shipping, or storage considerations.

8. REFERENCES

8.1 SPEX 8500 SHATTERBOX/CONTAINER, instruction sheet
SPEX Industries, Inc., 3880 Park Avenue, Metuchen, NJ 08840

8.2 Sorvall Superspeed Angle Rotors, GSA and SS-34 Instruction Manual
DuPont Company, Biomedical Products Division, Newtown, CT 06470

8.3 TWS-ESS-DP-101: Sample Identification and Control for Mineralogy-Petrology Studies.

9. ATTACHMENTS

None.