

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

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Roland C. Hagan
Roland C. Hagan
Preparer

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Date

David A. Mann
David A. Mann
Technical Reviewer

4-20-89
Date

Henry Paul Nynes
Henry Paul Nynes
Quality Assurance Project Leader

4/24/89
Date

R. J. Herbst
R. J. Herbst
Technical Project Officer

4/26/89
Date

8912190249 891211
PDR WASTE
WM-11
PDC

**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 dried 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 desiccation 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.