



U.S. Geological Survey
Mineral Resources Program – Quality Management System
Technical SOP
MRP-DML-SOP-03.01
SCANNING ELECTRON MICROSCOPY AND MICROANALYSIS

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Effective Date: 10/02/2020

MRP-DML-SOP-03.01– Scanning Electron Microscopy and Microanalysis

1. SCOPE, APPLICATION, AND SUMMARY

1.1. Summary

The scope of this laboratory SOP is to give basic guidance on the use of scanning electron microscopes (SEM) and attached detectors for imaging and chemical analysis in the Denver Microbeam Laboratory (DML). Analysis performed on SEMs differs from project to project due to the varied research goals of the users (i.e. energy, mineral, paleoclimate, water, hazards), different types of samples (i.e. particulates, bulk rock, thin sections, biological), and sample constraints (i.e. beam sensitive, high resolution, elemental analysis). Project-specific analysis methodologies must be recorded by the Laboratory User in their scientific notebook.

1.2. Trademark Disclaimer

The use of trade, product, or firm names in this SOP is for descriptive purposes only and does not imply endorsement by the U.S. Government

1.3. Table of Analytes, CAS Numbers, Reporting units, and Operational Range

Elements beryllium through uranium can be qualitatively measured in the range of 0.1 to 100 elemental weight percent. However, detection limits are element and matrix dependent.

1.4. Interferences

The possibility of peak overlaps and spectral artifacts will depend on the sample matrix and element(s) of interest. Laboratory Users will consult with Laboratory Staff prior to collecting data to communicate possible interferences.

1.5. Laboratory Personnel Required to Use the SOP.

Laboratory Staff, Analysts, and other laboratory users, who use instrumentation in the DML must follow this SOP.

1.6. Demonstration of Capability (DOC) Requirements for the SOP

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In order to produce data, Laboratory Analysts are required to receive training from Laboratory Staff, demonstrate competence and attain documented Demonstration of Capability (DOC) by the Laboratory Manager (see section 12.2). Subsequent DOCs must be performed every five years. If Laboratory Staff witness issues with a Laboratory Analyst which impact safety, the instrument, or data quality, subsequent retraining and DOCs must be performed.

For basic use of the SEMs, Laboratory Analysts must demonstrate that they can properly load and unload a sample, select appropriate beam parameters, manually adjust focus and stigmation, capture and save an image, collect an energy dispersive spectrum, and save a report.

2. HEALTH, SAFETY, AND WASTE DISPOSAL INFORMATION

Refer to the individual laboratory Chemical Hygiene Plan (CHP). The development of a CHP by the lab supervisor for each individual laboratory under his/her jurisdiction is required (29 CFR 1910.1450) by the Occupational Safety and Health Administration (OSHA). A hardcopy is available for immediate use by lab personnel. It is required that all lab personnel read, discuss, practice safety procedures listed within the CHP, and sign the training log within the CHP, signifying that they have read and understand procedures and rules. All lab personnel must understand what to do in the event of an emergency, and where within the CHP to find hazard and/or chemical information, including SDS information.

3. DEFINITIONS, ABBREVIATIONS, AND ACRONYMS

- 3.1. BSE – Backscattered electrons
- 3.2. BSED – Backscattered Electron Detector
- 3.3. CHP – Chemical Hygiene Plan
- 3.4. CL – Cathodoluminescence
- 3.5. DML – Denver Microbeam Laboratory
- 3.6. EBSD – Electron Backscattered Diffraction

3.7. EDS – Energy Dispersive Spectroscopy

3.8. ETD – Everhardt Thornley Detector

3.9. GAD – Gaseous Analytical Detector

3.10. FEG – Field Emission Gun

3.11. Laboratory Staff – Includes the Laboratory Manager and other Laboratory Scientists responsible for the operation and maintenance of the instrument. Laboratory Staff are responsible for: 1) maintaining the SEMs in operational condition; 2) training Laboratory Users for proper use of the SEMs; and 3) maintaining raw data files collected on the instrument and backup these files once a month. The assessment and interpretation of the raw and derivative data, including obtaining secondary data review, are the responsibility of the Laboratory Analyst and not Laboratory Staff.

3.12. Laboratory Analyst – The researcher or analyst conducting the analysis on the instrument. Laboratory Analysts are responsible for 1) reading the appropriate Quality Assurance Requirements (QARs), as assigned, and Quality Assurance Manual (QAM) prior to using the SEM; 2) attaining proper training and developing methodologies with Laboratory Staff prior to commencing work; 3) documenting the procedures and conditions used during analysis in their scientific notebook; 4) filling out the electronic instrument log with sufficient information to maintain traceability of their samples; 5) obtaining secondary data review following data collection; 6) maintaining digital copies of raw and derivative data and archival data; and 7) reporting any issues that arise during the analysis process.

3.13. Other Laboratory Users – Other Laboratory Users are USGS and non-USGS personnel with little or no formal training in microscopy and microanalysis who work under the direction of the Laboratory Manager or designee. Users are not required to perform a DOC. Users must read the SOP and will not stray from parameters decided upon with input from the Laboratory Manager to achieve predefined research goals. Laboratory Staff perform secondary data reviews.

3.14. LFD – Large Field Detector

3.15. SEI – secondary electron image

3.16. SEM – Scanning Electron Microscope

3.17. STEM – Scanning transmission electron microscope detector

3.18. vCD – voltage contrast detector

3.19. WD – Working Distance given in millimeters

4. SAMPLE PRESERVATION, HANDLING, CONTAINERS, ANALYTICAL PROCESSING/HOLDING TIMES, AND DISPOSAL

4.1. Preservation Requirements

SEM/EDS analysis is usually performed on solid material, so preservation is not usually required. If preservation requirements are critical to the Laboratory User's process and affect data quality, they must be documented in their scientific notebook.

4.2. Time Limits for Critical Stages of Analysis

Time limits for any critical stage of analysis are the responsibility of the Laboratory User and are recorded in the Laboratory User's scientific notebook if time limits are critical to the process or affect data quality. There is no holding time as the materials are solid.

4.3. Sample Locations, Sample Retention, and Sample Storage Conditions

Duration and storage conditions are the responsibility of the Laboratory User and must be documented in a scientific notebook if they are critical to the Laboratory User's process and affect data quality. The DML does not receive or store Laboratory User samples. Samples are stored in containers to prevent damage to the sample surface. Handle samples with gloved hands.

4.4. Sample Disposal

Sample disposal is the responsibility of the Laboratory User.

5. PREPARATION OF REAGENTS, STANDARDS, AND SOLVENTS

5.1. Reagents, Standards and Solvents Preparation, Storage and Shelf Life

5.1.1 All solvents, such as methanol and isopropanol, in the laboratory are used to clean the instrument and surface of the samples and do not affect data quality.

5.1.2 Polished solid reference materials for microanalysis are stored in desiccators in the DML. The reference materials are acquired from vendors. The reference materials are carbon coated according to the procedures outlined in MRP-DML-SOP-02.00 Sample Preparation for Electron Microscopy.

5.1.3 There is no shelf life or expiration of these reference materials.

5.2. Reagents, Standards and Solvents Documentation

Solid reference materials information such as chemical compositions, supplier, and source location (if known), are provided in labeled binders in the DML. Ask the Laboratory Manager if you have difficulties finding the information.

5.3. Verification of Reagents, Standards and Solvents

Solid samples of known composition (i.e. reference materials) are from a variety of sources including NIST, the Smithsonian, SPI, and colleagues. The compositions of these reference materials are provided by the supplier, found in external publications, or agreed upon through a round robin evaluation.

6. APPARATUS

6.1. Lab Ware

Sample stubs, carbon tape, copper tape, carbon paint, spatulas, forceps, razor blades, glass plates, glassware.

6.2. Instrumentation

6.2.1 JEOL 5800 (SN:MP16110085)

- Tungsten filament SEM with BSE and SEI imaging detectors
- Thermo Fischer 10 mm² silicon drift detector for EDS running Noran System Seven v. 3.x.
- K.E. Developments panchromatic photomultiplier tube for CL

6.2.2 FEI Quant 450 FEG (SN: D9232)

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- Field emission gun source SEM with BSE, SEI, and STEM imaging detectors running version 4.x for SEM control
- Oxford Instruments 50 mm² MAX silicon drift detector for EDS and XYZ camera for EBSD running Oxford Aztec Software version 3.x.
- Horiba Instruments HCLUE hyperspectral CL collection system.

7. SAMPLE PREPARATION, ANALYSIS, AND INSTRUMENT OPERATION AND SHUTDOWN

7.1. Sample Preparation

Sample preparation requirements are stated in MRP-DML-SOP-02 Sample Preparation for Electron Microscopy.

7.2. Sub-sampling

N/A

7.3. Sample Analysis

Step-by-step machine operation guidelines for specific instrumentation are published or are available online through context sensitive help and in manuals located on the DML bookshelf (MRP-DML-MAN-08, MRP-DML-MAN-09, MRP-DML-MAN-10, MRP-DML-MAN-11; see section 11 References for manual titles). It is not the purpose of this SOP to repeat these procedures, and the Laboratory Analyst is referred to the above publications. The Table of Contents and indices within these publications can provide page numbers for specific queries, and when used in non-routine circumstances, will be documented in the scientific notebook. Scanning electron microscopy and microanalysis is an interpretative procedure and exact specifications will vary depending on research goals and sample response. Below are considerations when conducting typical imaging and qualitative data analysis. Note: EBSD and hyperspectral CL are not routine procedures. If the Laboratory Analyst or User wishes to acquire EBSD or hyperspectral CL, Laboratory Staff will work with the Laboratory Analyst or User to collect the data and document all methods in the Laboratory Users scientific notebook.

7.3.1 Pre-analysis guidelines

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- Fill in the instrument logbook with sufficient detail (i.e. name, date, unique sample identification, operating conditions, and data folder location) such that the data are traceable to the samples.
 - Sample handling: To reduce oil and dirt contamination, always wear powder free gloves while handling sample holders, sample stubs, pellets, and samples as this helps to reduce the buildup of contaminants in the instrument chamber.
 - Particulate sample material – To reduce damage to the SEM and to the attached detectors, particulate samples must be secured to sample stubs or slides using an adhesive (i.e. carbon tape, epoxy). Image quality may also be impacted if samples are not secured adequately.
 - Pre-planning – If the analysis that is to be conducted requires navigation to specific areas on a sample surface, it is important to document where the area(s) of interests are before loading samples into the instrument. This not only improves efficiency but is important when analyzing beam sensitive samples as the longer the electron beam interacts with the sample it increases the chances of charging/surface damage. Laboratory Staff can provide users example sample mapping methods and techniques.
 - Sample holder selection – The DML has a wide selection of sample holder types that can be used for loading samples into the instrument. Most holders are for standard thin sections (27 x 46 mm) or 1" (25 mm) round mounts. Oddly shaped samples can be accommodated. Consult with Laboratory Staff for best mounting practice to ensure that the sample is stable in the chamber. The Laboratory Analyst records in their scientific notebook the relative position and orientation of their samples in the holder.
 - Reducing surface charge build up – For non-conductive samples, it is important to reduce charge build-up on the sample surface as this may damage the sample or impair image quality. It may be necessary to apply carbon tape or colloidal graphite from the edge of the sample surface down to the metal sample holder to allow a path to ground for the electrons. Laboratory Analysts shall consult with Laboratory Staff to advise if this is necessary for their samples/analysis.
- 7.3.2 Sample loading and unloading must be done with gloved hands. Load the sample into the microscope by venting the chamber, lowering the stage to avoid a detector collision, open the chamber, and load the sample into the chamber using appropriate sample holder. Pump down the chamber to desired pressure. This step is only to be performed by laboratory staff.
- 7.3.3 Once the sample chamber has reached the desired working vacuum, select the accelerating voltage and beam current appropriate to achieve the research goals. Imaging conditions and detectors will vary based on the research goals of the analysis. Below are some general guidelines and initial imaging conditions. Electron voltage, beam current, and working distance will vary depending on how electrons are interacting with the sample. The Laboratory Analyst will record conditions in

their scientific notebook. All working distances provided are approximate and vary by the instrument stage positioning system and sample height. Deviations from the provided working distance will not affect the qualitative data quality.

- Thin Section – Use high-vacuum mode with a voltage of 10 – 20 kV and approximately 1 nanoamp beam current, and a working distance of 11 mm. These parameters allow for imaging and sufficient energy for EDS analysis.
- Broken Rock Surface – If the sample is coated, high vacuum mode can be used. If the sample is not coated, it can be analyzed under low vacuum conditions. Generally a pressure of 20 pa is a good starting point for low vacuum mode. It is suggested to start with 15 kv electron voltage and ~1 nanoampere beam current. The electron voltage and beam current should be kept as low as possible to prevent electron build up on the sample.
- ETD (High Vacuum): WD > 20 mm to 3 mm, Optimum WD: 3-5mm; Beam Conditions 15kV and ~0.5 nA beam current. These conditions image the topography of the sample surface.
- BSED and vCD (High Vacuum): WD > 20 mm to 8 mm, Optimum WD: 5mm; Beam Conditions 15kV and ~3 nA beam current. These conditions image the relative change in average atomic number of the sample.
- Simultaneous ETD, BSED, and EDS use (High Vacuum): WD 11.0 mm; Conditions 15kV and ~3 nA beam current.
- BSED & vCD (Low Vacuum): WD > 20 mm to 8 mm; Optimum WD: 8-10mm for BSED, 6-10mm for vCD Pressure range : 10 to 200Pa, Starting pressure= 40Pa Beam Conditions: 10kV, Spot 3.
- LFD + GAD (Low Vacuum): WD >20 mm to 10 mm; Optimum WD: 10mm, Pressure range: 10 to 2600Pa, Starting pressure= 70Pa Beam Conditions: 10kV, Spot 3.

7.3.4 Turn on the beam.

7.3.5 Navigate to your sample or the copper tape (or other calibration material) if it has been loaded for use.

7.3.6 Focus the image using the focus knobs. Fine tune the focus using the stigmation knobs.

7.3.7 Adjust the working distance as needed based on the focused image. Watch the infrared chamber scope while adjusting the working distance to avoid a sample and detector collision.

7.3.8 Navigate to the to the copper tape (or equivalent) if it has been loaded to calibrate the EDS if necessary. Focus on the copper tape then move the working distance to 11 mm. Collect an EDS spectrum of the copper (or equivalent) through the EDS software optimization option. If the optimization fails, contact Laboratory Staff. Although the optimization may have failed, the unit

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may still be utilized for qualitative purposes if the unit produces peaks at the calibration element expected peak energy.

- 7.3.9 Move to an area of interest and refocus if necessary. Adjust the brightness and contrast then slow the raster to achieve optimal image quality. Once the raster is complete, save the image to the Laboratory Analyst/User image folder.
- 7.3.10 If EDS is desired, reacquire the image through the EDS acquisition software. Once the image is loaded, point on the image to deflect the beam to the area of interest and collect the EDS spectrum.
- 7.3.11 Initial element identifications of the EDS spectra are automated through the software. It may be necessary to increase the accelerating voltage to confirm the presence of higher energy lines for heavy elements. A theoretical spectrum based on element identification is displayed for comparison to unknown spectra. Laboratory Staff are available to consult for element identification.
- 7.3.12 Once the acquired field of view is complete and EDS spectra have been reviewed, save a report to the Laboratory Analyst/User EDS data folder. The Laboratory User is responsible for recording the instrument conditions such as accelerating voltage(s), magnification range, and beam current (spot size) in their scientific notebook.
- 7.3.13 After the analysis is finished, turn off the beam, lower the stage, and vent the sample chamber.
- 7.3.14 Open the sample chamber and remove samples. This step is completed only by Laboratory Staff.
- 7.3.15 Close the sample chamber and place under high vacuum. This is the default instrument configuration when not in use.

7.4. Dilutions, Problematic Samples, Carryover

Problematic samples include beam sensitive, vacuum sensitive, and high relief materials. Contact Laboratory Staff if necessary.

7.5. Troubleshooting and Bench Notes

Laboratory Analysts/Users are discouraged from troubleshooting issues and must immediately contact Laboratory Staff for assistance.

7.6. Maintenance

The instruments are maintained to manufacturer's specifications regarding beam alignment, magnification, image resolution, peak width energy resolution, and pattern center through a biannual maintenance visit. Digital copies of the visit summary are printed and stored in an instrument service binder stored on the laboratory bookshelves and in a digital instrument logbook on the instrument computer. A summary of the maintenance visits are recorded in the instrument logbook.

8. DATA ACQUISITION, PROCESSING, AND EVALUATION

8.1. Data Acquisition and Calculations

Acquired images are generally stored in the .tiff file format. It is recommended to use .tiff format as all data regarding date/time, beam conditions, magnification, and system settings are stored in the .tiff header information. If another option is used, such as .jpg image formats which do not save system parameters, the Lab Analysts must record date, accelerating voltage, and spot size, in their scientific notebook. Laboratory Analysts are responsible for retaining original copies of the images.

Raw EDS data are saved in the Vendor's proprietary data formats. Visual displays of the EDS spectrum can be saved by the Laboratory Analyst in any image format or in a MS Word document. Report options exist that will output elemental compositions based on the Vendor's standardless analysis routines. These data are for informational purposes and are not to be used in publication. Quantitative elemental composition by EDS in the DML is not routine and outside the scope of this SOP.

8.2. Handling Sample Data That Is Outside the Calibration Range

-N/A.

8.3. Data Processing

Data processing and evaluation are dependent upon the research being conducted. All Laboratory Analysts are responsible for recording in a scientific notebook all methods and processing used during and after analysis.

9. QUALITY ASSURANCE (QA) AND QUALITY CONTROL (QC)

9.1. Internal QC Samples

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If the Laboratory User will utilize EDS during the course of their investigation, calibration of peak position and count rate is conducted on copper tape or other pure metal standard when appropriate. The peak position and count rate are adjusted by the software using vendor algorithms. If the peak calibration fails, Laboratory Analysts/Users must contact Laboratory Staff.

9.2. External QC Samples

N/A

9.3. QC Charts

-QC is pass or fail so not ameanable to QC charting.

10. DATA AND RECORDS MANAGEMENT

10.1. Data Packages

Prior to secondary data review, the data are provisional and for informational purposes and are not to be used or published without a provisional statement. It is the Laboratory Users responsibility to complete a secondary data review.

Data are stored in electronic folders using the Analyst's last name on the FEI, Thermo, and/or Oxford data drives. Data packages must include a statement of the sample preparation, instrument used, and operating conditions. In addition, the package will include an image of the EDS calibration (if performed), backscattered and secondary images (if collected) with scale bars, EDS spectra with properly labeled peaks (if collected), and element maps (if collected).

The secondary review checklist includes (but not limited to) (see Section 12.1):

- Serial number of instrument used
- Date(s) of analysis
- Operating conditions
- EDS calibration spectrum collected (if necessary)

- Images with scale bars
- EDS spectra have properly labeled peaks

10.2. Archival and Storage Requirements

Data are saved directly to the Laboratory Analyst's project folder created at the beginning of the analysis run. Laboratory Analysts are responsible for collecting their files at the end of their analysis time.

Laboratory Staff are responsible for backing up the data files once a month to an external hard drive and the USGS servers.

11. REFERENCES

- 11.1. FEI, The Quanta FEG 250/450/650 User Operation Manual, 5th Edition, 20 August 2010, Copyright 2010 FEI Company 228p. (MRP-DML-MAN-08)
- 11.2. JEOL, A Guide to Scanning Microscope Observation. (MRP-DML-MAN-09)
- 11.3. Oxford Instruments, AZtec 3.4 User Manual, Copyright 2010-2017, Oxford Instruments Nanotechnology Tools Limited trading as Oxford Instruments NanoAnalysis, 993p. (MRP-DML-MAN-10)
- 11.4. Thermo Scientific, Thermo Scientific NSS Help, Copyright 2002-2010 Thermo Fisher Scientific Inc.(MRP-DML-MAN-11)
- 11.5. Additional information is also found in the Help menus for the specific software packages used for each instrument (Section 7.2).

12. ATTACHMENTS

12.1 Secondary data review checklist

Secondary Data Review Checklist

Laboratory Staff Full Name			Signature		
Laboratory User Full Name			Signature		
Data file location and name	Example: DML, E:\UserData\Lowerys\2020-05-19 amphibole.mdb				
Sample preparation	Examples: 20 nm carbon coat on thin sections, or 10 nm Ir coating on polished pucks, or 20 nm carbon coat on polished billet. Sample is not planar and therefore data may fall outside accepted limits.				
	Analyst Initials	Date	Secondary Reviewer Initials	Date	Comments
Serial number of instrument used is provided					
Dates of analysis documented					
Operating conditions Listed					
EDS calibration image included (when performed)					
EDS Calibration Pass/Fail					
Images and element maps have scale bars					
Additional requirements met (please state in the comments section)					

12.2 Documentation of Capability

Demonstration of Capability (DOC) requirements

for MRP-DML-SOP-3.00 Scanning Electron Microscopy and Microanalysis

In order to produce images using the procedures outlined in ERP-REBSEM-SOP-3.00, Laboratory Analysts are required to receive training from Laboratory Staff, demonstrate competence and attain documented attestation of competence (DOC) by the Laboratory Manager. If Laboratory Staff witness issues with a Laboratory User which impact safety, the instrument or data quality subsequent retraining and DOCs must be performed.

Laboratory Analysts must demonstrate that they can properly perform the following tasks in order to operate the instrument without Laboratory Staff present.

SEM/EDS DOC:

☐

Read MRP-DML-SOP-3.00 Scanning Electron Microscopy and Microanalysis

☐

Completed Basic Operation DOC requirements.

☐

Load and unload a sample

☐

Select appropriate beam and stage parameters.

☐

Manually adjust focus and stigmatism.

☐

Capture and save an image.

☐

Collect an EDS spectrum and save the report.

Laboratory Analyst's Name _____

Date _____

Laboratory Manager Signature _____

13. HISTORY OF CHANGES

Revision No.	Effective Date	Description of Changes
00	06/20/2019	Initial issue
01	10/02/2020	Updates were made based on an internal audit conducted 2/10/2020 through 2/14/2020. These included clarifying the role of laboratory staff and users, adding the location of the maintenance records, and updating the secondary data review sheet.