1.0 Purpose and Scope

The purpose of this procedure is to establish a method for taking scanning electron microscope (SEM) images and various energy dispersive spectroscopy (EDS) measurements.

This document is a supplement to manufacturer’s manuals for the operation of the JEOL JSM-5900LV SEM and the NORAN System 7 Spectral Analysis system (an EDS system). This supplement is not intended to be formal instrument documentation, nor does it cover all features or use of the instrument, but rather a hands-on user’s guide to the typical operation of both the microscope and analyzer. Details of microscope operation, the EDS acquisition system, and the computer software can be found in the JEOL Institute Basic Scanning Electron Microscope Operations Course book and the manufacturer’s manuals kept in the lab.

Acronyms and definitions for terms used in this procedure may be found in the Glossary located at the Sandia National Laboratories (SNL) WIPP Online Documents web site.

2.0 Implementation Actions

2.1 Responsibility

The Principal Investigator (PI) or designee is responsible for implementing the requirements of this procedure.

The Technical Staff is responsible for performing the measurements following the requirements of this procedure, documenting all required information described in this SP, and assuring the latest revision of this document is followed.

If the procedure cannot be worked as written, the user has the responsibility to stop work and resolve all concerns with the PI/designee or safety, as appropriate, prior to proceeding with the work.
2.2 Qualification and Training

Qualification and training of users will be documented on the instrument qualified users list that is maintained at the instrument. Users should not use this instrument without on the job training from an experienced user.

2.3 Safety

The activities described in this SP shall conform to SNL Environmental Safety and Health programs (ES&H). The SEM/EDS system is classified as an inherently safe radiation generating device (RGD) according to the Radiological Protection Procedures Manual (RPPM), which requires operators to be current in General Employee Radiological Training (RAD102) as a minimum. Additionally, the RPPM requires RGD operators to complete on-the-job (OJT) training on the instrument, which will instruct users in the safe operation of the instrument. OJT will be recorded on the instrument qualified users form.

2.4 Data Collection and Storage

Data collected on this instrument will be collected on printouts, in scientific notebooks (SN), or on electronic media. Records of instrument use will be made in the instrument log book. Notes on what measurements were performed will be documented in the scientific notebook or in the instrument log book.

2.5 Instrument Procedures

Follow the instructions in the Typical Operation section of The JEOL Institute Basic Scanning Electron Microscope Operations Course manual (JEOL Course), the JEOL user’s manual (User’s Manual), the NORAN System 7 user’s manuals, and the online help files. Below is additional information and procedures that should be performed. Details on how to perform these operations are contained in the above references. In the below text, there are references to knobs and buttons on the instrument control panel and/or methods for computer control. Most features have alternative control methods (such as control/panel or computer control) which are available and described in the user’s manuals.

2.5.1 Preparation of Instrument

A. Open Valve at back of chiller to cool compressor.
B. Turn on chiller.
C. Turn on instrument, following the instructions in Sections 3.1 – 3.2 of the user’s manual.
D. Turn on infrared camera.

2.5.2 Sample Preparation

A. Choose the appropriate sample carrier and type of sample mount (stub, cylinder, etc.).
B. Mount sample on sample mount using desired adhesive (carbon sticker, silver paint, graphite paint, etc.).
C. If desired, coat the sample using the Denton Vacuum Desk V Sputter/Etch Unit. Follow the directions in the Sputter manual. Basic start-up information is given below for Manual Sputtering:
1. Ensure that the Isolation Valve on the Vacuum Pump is in the open position (straight up).

2. Ensure that the Gas Flow valve on the coater is closed (turn valve clock-wise).

3. From the Manual Sputter screen, turn the mechanical pump ON. Wait for the Vacuum pressure to drop below 4.0e-2 Torr.

4. Continue pumping and from the Manual Sputter screen, turn the Gas Valve ON.

5. Adjust the gas pressure using the manual Gas Flow valve (GFV) on the top of the sputter/etch unit to a pressure between 5.0e-2 and 7.5e-2 Torr. Allow the pressure to stabilize in the pressure range. (NOTE: GFV clockwise = decrease flow reduce pressure; GFV counter-clockwise = increase flow, increase pressure.)

6. Type the desired mA into the box at the bottom of the screen (10-20 mA for approximately 10-45 seconds is usually enough to get a decent thin coating).

7. From the Manual Sputter Screen turn the Sputter Power ON.

8. Sputter for the desired time.

9. From the Screen, turn OFF the Sputter Power and reduce the Sputter SetPoint to zero.

10. From the Screen, turn the Gas Valve OFF.

11. From the Screen, turn the Mechanical pump OFF. The Instrument will automatically vent when this button is pushed.

12. NOTE: If you would like to use the sputter/etch instrument for timed coating, do not adjust the GFV on the top of the coater.

13. For all other operations of the Denton Vacuum Desk V instrument (e.g. timed sputtering, etching etc.) consult the Denton Vacuum Desk V Manual.

D. After sample has been mounted, place mounted sample into appropriate sample carrier and gently tighten down set screw (if it has one).

E. Place sample carrier into specimen exchange tool with the flat part of the carrier facing away from the specimen exchange tool.

### 2.5.3 Sample Exchange

Follow the instructions in Section 3.6 of the user’s manual. Additional information is given below.

*Take note! Important warnings are set in boxed text.*
операция в вакуумном режиме

2.5.3.1 Предварительные процедуры

A. Проверьте состояние прибора. На панели состояния, проверьте, чтобы предыдущий пользователь оставил прибор на соответствующих настройках:

1. Детектор SEI должен быть включен.
2. Напряжение ускорения составляет 20 кВ.
3. Высота стадия составляет Z=30 мм или больше.
   
If the stage height is less than 25mm, see section B below. Note that the higher the Z value is the further the stage is from the detector.

4. Позиции X, Y, T и R должны быть приблизительно равны 0.

B. Если высота стадия не соответствует желаемому значению, выполните следующие действия:

1. Нажмите кнопку T/Z, чтобы перейти к функции "Z" настроечной панели.
2. Повышайте стадию, нажимая на позиционный джойстик, и уменьшайте, двигая джойстик к вам (отрицательное направление Y).
3. На мониторе камеры, наблюдает за положением стадия относительно нижней щели объектива.
   
When raising the stage, set the desired working height at the highest part of the sample. DO NOT allow the sample to contact the objective lens or the backscattered electron (BSE) detector.

4. Нажмите кнопку X/Y, чтобы перейти к функции "X-Y" настроечной панели.

If at any time, the sample appears that it might, for whatever reason, contact the BSE detector, immediately move the joystick in any direction to abort the move.

C. Если вы планируете проводить EDS работу, бак твердого азота должен быть заполнен не менее 6 часов перед использованием.

If you are planning on turning on the EDS front end (small white computer looking box to the right of SEM), the liquid N\textsubscript{2} dewar should be filled at least 6 hours prior to use. DO NOT turn on the EDS front end without liquid N\textsubscript{2} in the dewar.

2.5.3.2 Загрузка образцов

A. Пустите камеру.

1. Нажмите и удерживайте кнопку VENT, чтобы активировать.
2. Unlatch the door latch.

3. Wait until VENT light stops flashing. Instrument will beep once (door may open by itself before that!).

B. Insert specimen holder.

You must be sure that you don’t hit the backscattered electron detector (BEC) or any other sensitive components when you insert the specimen. The only thing you should be making contact with is the stage.

1. Use the Specimen Inserting Tool to load sample holder into the chamber.

2. Align holder with dovetail on stage.

3. Push holder all the way in until it stops.

4. Press both rubber buttons on tool and carefully withdraw tool.

At this point there should be at least 1 cm between the top of your sample and the BEC. Now with the door open and your eyes focused on your sample, raise the sample to the desired height, leaving plenty of room between the top of the sample and the BEC. Follow the directions above in B.

C. CLOSE the door and latch.

D. PRESS and Hold EVAC button until it activates

E. When the EVAC light stops flashing and the instrument beeps once the system is ready to proceed. At this point the H/T button will indicate ready.

2.5.4 Initial Setup

2.5.4.1 Preliminary procedures

A. Set accelerating voltage. Appropriate accelerating voltage should be set according to the type of work that is planned. Note that you can easily change the accelerating voltage during operation, but doing so may require readjustment of some instrument parameters, so it is best to get everything setup properly at the beginning.

B. Set computer to appropriate sample holder.

1. From the Menu Screen, SELECT STAGE.

2. POINT to and SELECT correct HOLDER.

C. Switch on the chamber scope if you haven’t already done so.

D. Move the sample position to an appropriate place on the sample or holder. For the most part, beam saturation and alignment can be done anywhere on the sample. However, to avoid damaging the sample, it is recommended that setup be done on the rim of the sample holder.

1. Make sure the X/Y button is lit on the keyboard.
2. The Menu Screen should be on STAGE menu.

3. Use the joystick to move to the outer rim of the sample holder while observing the position of the crosshair on the graphical display of the Stage menu. You can also click on the screen a point on the sample holder and then click ok.

4. TURN the MAGNIFICATION knob counterclockwise to the lowest magnification.

2.5.5 Instrument Startup

You may proceed with this section once you have turned on the accelerating voltage (HT or High Tension), by following the JEOL Course instructions, or the instructions in the JEOL user’s manual Section 3.7.

A. Filament saturation. If the instrument has not been used for some time, check filament saturation. Follow the instructions in the JEOL Course. Auto filament heating and Auto gun alignment is not recommended.

B. Gun tilt. Check gun tilt to achieve maximum brightness. Follow JEOL Course instructions.

C. Current axis (gun) alignment.

**Note 1:** You will need a sample area suitable for magnification up to 20,000X. You can probably find a small milling mark or other discernible feature on the sample holder (bright spherical objects work best).

**Note 2:** Bracket the most commonly used current range (spot size 20 to 40). If other spot size will be used, bracket gun alignment in that range.

1. ADJUST the FOCUS knob (coarse focus) to initially focus at low magnification on the surface of the sample holder.

   a. Find a small feature. Gradually increase magnification while focusing to 20,000X.

   b. Approximately center the object in the monitor.

2. PRESS SPOT SIZE button. Use CRS knob to set spot size to 20 on the Status Panel.

3. CHOOSE RESET LENS from Tools pulldown in software button on keyboard.

   a. Refocus (probably will turn focus clockwise). If object leaves field of view after refocus, back down magnification to find it again.

   b. RESET LENS again and refocus if necessary.

   c. Repeat reset until there is no movement or change in focus.

4. Closely center object on monitor using the joystick.

   a. Place left index finger on object at center of monitor and keep it there.
b. **ADJUST SPOT SIZE** to 40.

c. **ADJUST X** and **Y** gun alignment to recenter object under finger. This is in Gun Alignment under the Menu button in the software.

d. Observe object while turning **SPOT SIZE** back to 20.

e. While turning spot size down, object may loop out from under finger and return back. This is a function of the microscope itself and is ok.

5. Check gun tilt again. You shouldn’t have to change this much, if any, at this point.

D. Check Objective Aperture alignment, follow directions in JEOL Course manual.

E. Stigmation adjustment

   **Note:** You will need an area of the sample holder that has fine details (a cluster of bright, spherical objects is best).

1. Increase magnification to an appropriate level (5,000-20,000X).

2. **ADJUST** the **FOCUS** knob (coarse focus) to best focus.

3. **PRESS** **COARSE** button off, and carefully focus as well as possible.

4. **PRESS** **STIG** button.
    
a. Adjust **X** and **Y** stigmation knobs one at a time until image becomes as clear and sharp as possible.
    
b. Readjust fine focus.
    
c. Repeat until image is sharp and clear.

### 2.5.6 Instrument Shutdown

A. Return detector to SEI.

B. Turn filament off by clicking the H/T button.

C. Lower stage to **Z=30 mm**.

D. Wait for 3 to 5 minutes for filament to cool before continuing.

E. Remove sample holder.

1. **PRESS** and **HOLD** the **VENT** button until it activates.
    
a. **UNLATCH** the door latch.
    
   b. Wait until **VENT** light stops flashing. Instrument will beep once (door may open by itself before that!).

2. Use the Specimen Inserting Tool to remove sample holder from the chamber.
a. ALIGN holder with slots on bottom of sample holder.

b. PUSH holder all the way in until it stops.

c. Carefully withdraw tool and sample holder (don't press rubber buttons on tool).

3. CLOSE the door and latch.

4. PRESS and HOLD EVAC button until it activates.

5. Remove sample holder from Tool by pressing rubber buttons on Tool handle.
   a. Remove sample adapter from holder; return holder to shelf.
   b. Remove sample stubs from adapter; return adapter to shelf.
   c. Return Inserting Tool to drawer.

F. Secure the instrument.

   1. Turn off chamber scope.
   2. Once chamber is evacuated exit JEOL software and turn off instrument.
   3. Fill out logbook.
   4. Record in the SN images taken, location of image files and image parameter files, i.e. a SNS or electronic media.
   5. Clean up around instrument and sample prep counter.
   6. Allow chiller to run for 30 minutes after turning off instrument. Then shut down chiller and close water valve at back of chiller.

2.5.7 Primary Imaging

2.5.7.1 Backscattered Electron Imaging

A. Make sure the infrared camera is off, if it's on you'll get white bands going down the image and extremely poor contrast. If you need to put the contrast bar all the way to the right, chances are the camera is on.

B. Your sample should be fairly close to the detector (see Subsection B). If you have a couple of cm from the top of your sample to the detector, you won’t get decent pictures.

C. You may have to adjust the spot size, try going up a few and down a few until you can see some detail in you image.

D. Increase contrast incrementally, while lowering (or raising) brightness to keep the screen moderately dark with snow, until you find the image.
2.5.7.2 Additional information on Backscattered and Secondary Electron Imaging

A. The secondary electron (SE) detector can be used in any mode of operation EXCEPT low vacuum.
   1. The SE detector will be automatically turned off in low vacuum operation.
      a. The solid-state backscattered electron (BSE) detector will automatically be switched on.

2. To switch between detectors in high vacuum mode:
   a. go to STATUS DISPLAY: Signal:
   b. SELECT the SEI (#1) detector for secondary electron imaging.
   c. SELECT the BSE Compo (#2) detector for compositional or "shadowed" topographical backscattered electron imaging (BSE Topo [#3]) is used to emphasize the DIFFERENCES in topography rather than produce a topographic image).
   d. SELECT the AUX signal for EDS imaging.

B. The BSE detector can be used in any mode of operation.
   1. During low vacuum operation, it is the ONLY image detector available.

2. For collection of topographical images (especially in low vacuum mode), the "shadow" detector must be switched on.
   a. SELECT BEIW from STATUS DISPLAY: Signal:
   b. In the BEIW window, choose SHADOW.
   c. On the SHADOW ENHANCE slider, set the degree of topographic enhancement (settings between 5 and 7 seem to work best).

3. Higher values produce more topographic contrast, but darken the image.

4. Lower values result in less topographic contrast and brighter images.

5. For pure BSE images, e.g. working on polished thin sections, switch the shadow detector off.

2.5.8 Microscope Maintenance

Refer to Section 4 of the JEOL user’s manual, and the Filament Maintenance section of the JEOL Course book, and obtain training before attempting any maintenance. Below is additional information.

2.5.8.1 Filament Exchange

To be performed when the filament has burnt out. Read and understand the instructions in Section 4.5 of the JEOL user’s manual. See below for additional information.
A. After venting the gun chamber allow the microscope to cool for at least an hour.

B. Don’t use the Wehnelt extraction tool, instead wait until the microscope is cool and use your hands (wearing powder free gloves) to pull the Wehnelt off the electrical terminals.

C. If the Wehnelt seems particularly dirty (when viewed using the available microscope), you may need to use acetone or methanol for cleaning.

D. Wear powder free gloves.

E. Use the Pink POL polish, and the q-tips in the same drawer to polish the Wehnelt. Change q-tips often to avoid scratching the Wehnelt.

   1. Polish all the crust off the tip of the Wehnelt.
   2. Polish all the Pink POL polish off the Wehnelt using a clean q-tip.
   3. Use the stereo microscope to view the Wehnelt and look for remaining crud or polish.

F. Look at the broken filament in the stereo microscope. Compare to picture on page 4-9 of the JEOL user’s manual. If the break is abnormal, follow instructions in Section 4.5 of JEOL user’s manual to correct the problem.

G. Clean the spacer as you cleaned the Wehnelt.

H. Don’t forget to align the filament, as described in Subsection 2.5.5.

### 2.5.9 NORAN System 7 EDS system

Follow the directions in the NORAN System 7 user’s manuals located in the laboratory. Below is additional information on the use of this system.

#### 2.5.9.1 Startup of EDS System

A. Be sure that there is liquid $N_2$ in the EDS liquid $N_2$ dewar. If not, fill dewar and wait 6 hours.

   \textbf{The dewar is not necessarily full when you see liquid $N_2$ spilling out the top during filling. You can observe this with as little as 1 in of liquid $N_2$ in the dewar. You should use a flashlight to see if there is liquid in the dewar before you turn on the EDS front end and energize the detector.}

B. Turn on the microscope first (if it is not already on), but not the filament.

C. Make sure the infrared camera is off before turning on the EDS system. It may get confused, if the camera is on, and you may have to restart the EDS system as described below in step F.

D. Turn on the EDS front end and the computer. Turn on filament (if filament is ready) and begin microscope procedures as described in Subsection 2.5.5.
E. If at any point in time the EDS alarm goes off, turn off the EDS front end immediately. This means there is not liquid N₂ in the EDS dewar.

F. If the EDS system fails to take a spectrum or the computer freezes up (for more than several minutes) shutdown the computer then shutdown the EDS front end and restart EDS system.

2.5.9.2 Collection of Spectra

A. Begin by taking a spectrum as described in the NORAN System 7 user's manuals.

B. If using Spectrum or Point & Shoot, on the lower right portion of the screen choose the Detector Status tab. The detects should be around 1000 /sec. The deadtime should be around 25%. If not, performing the following may help to obtain better spectra:

1. If detects/sec is not around 1000/sec, adjust instrument parameters to bring detects/sec up to (or down to) 1000/sec.

2. If deadtime is not around 25% go to the Edit menu click on acquisition properties and go to the EDS tab. Under Pulse Processor, make sure that time constant is set to auto.

C. Now continue taking spectra, mappings or other EDS measurements.

2.5.9.3 Shutdown of NORAN System 7

A. Shutdown NORAN System 7 computer first.

B. Shutdown NORAN System 7 front-end using power switch on the front of the unit.

3.0 Records

The following records, generated through implementation of this procedure, shall be prepared and submitted to the WIPP Records Center in accordance with NP 17-1 (Records):

QA Record

- SN (Scientific Notebook)
- SNS (Scientific Notebook Supplement)

Non Quality (NQ) Record
- Electronic media

4.0 Appendices

There are no appendices in this SP.
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