

23 AUGUST 2024

SCHOOL OF MATH AND SCIENCE STANDARD OPERATING PROCEDURE 5104.7

From: Dean, Division of Mathematics and Science

Subj: JEOL MODEL JSM-6010LA SCANNING ELECTRON MICROSCOPE OPERATING AND EMERGENCY PROCEDURES

Ref: (a) NAVSEA S0420-AA-RAD-010 (RAD-010) Revision 2A with ACN-1 "Radiological Affairs Support Program Manual"
(b) NAVMED P-5055 "Radiation Health Protection Manual"
(c) JEOL MODEL JSM-6010LA SEM Instructions Manual (ISM6010LV/6010LA-6E, July 2012)

Encl: (1) Facility Description
(2) Facility Radiation Safety Evaluation
(3) General Controls and Practices
(4) General Operating Procedures
(5) Emergency Procedures
(6) Radiation Monitoring
(7) Authorized User Qualification Requirements
(8) Records – Sheets and Logs

1. Purpose. This standard operating procedure (SOP) contains procedures for the safe operation of the JEOL Model JSM-6010LA Scanning Electron Microscope (SEM).

2. Cancellation. This document updates MATHSCIDIVINST 5104.7 and converts it to a Standard Operating Procedure for the School of Math and Science as part of the required 5-year review by the USNA Radiation Safety Committee and re-authorization for the Physics Scanning Electron Microscope.

3. Applicability. This SOP shall be used to govern operation of the JEOL Model JSM-6010LA SEM located in Chauvenet 046.

4. Discussion. The JEOL Model JSM-6010LA SEM uses electron beams and the resulting secondary X-ray radiation to produce detailed digital images of materials placed inside a shielded housing. This SOP establishes the responsibilities of individuals involved with the safe operation and maintenance of the SEM located in Chauvenet Hall. The SEM is part of the SEM Laboratory, and the individuals responsible for the SEM are the SEM Laboratory Manager and the Radiation Safety Officer (RSO).

5. Responsibilities.

a. The Laboratory Manager shall:

(1) Ensure the safe usage and security of SEM in accordance with this SOP and investigate and report alleged or suspected incidents of overexposure to the Radiation Health Officer via the

Radiation Safety Committee.

(2) Ensure that only personnel, trained, and qualified in accordance with this SOP and Enclosure (7) are involved in the operation of the SEM.

(3) Conduct periodic surveillance checks to ensure that hazard areas are posted.

(4) Be responsible for keeping qualification, radiation survey, and operation records up to date including utilization logs and radiation survey logs.

(5) Report any malfunctions, alignment operations or repair operations to the RSO.

(6) Investigate and report to the RSO any suspected instances of overexposure.

(7) Conduct inspections, at least once every six months, to determine that the necessary interlocks are functioning on the instrument.

(8) Complete a quarterly Audit of the facility, records and postings, and maintain the record in the facility binder.

(9) Keep RSO informed of any issues related to management of the PANalytical laboratory, to include recommending (to the RSO) the removal of Authorized User qualification as needed.

(10) Endorse qualified users to the RSC to be approved Authorized Users.

b. Authorized Users.

(1) Machine sources at USNA are only to be operated by or under the supervision of Authorized Users. In order to qualify as an Authorized User, the individual must meet the following conditions:

(a) The individual shall be a DoD employee or student or shall have an official appointment to the USNA faculty/staff.

(b) The individual shall have been designated as an authorized user by the Radiation Safety Officer.

(c) The individual shall have attended the training program provided by the Radiation Safety Office. The training will be commensurate with the type of risk they are exposed at the workplace.

(d) The individual shall have received on job training from the Laboratory Manager covering the specific procedures and attendant hazards associated with the work to be performed in accordance with this SOP.

(e) Students taking the SP425 course and SP495/SP496 (or other) research courses will not normally be classified as Authorized Users.

(2) Authorized Users shall:

(a) Be responsible for all aspects of SEM operation, including the radiation safety of everyone in and around the SEM room.

(b) Ensure that he/she is a Qualified Authorized User, as defined in Enclosure (7) of this SOP before attempting to operate the SEM.

(c) Ensure that he/she is familiar with the contents of this SOP, its enclosures and the relevant parts of the references.

(d) Operate the SEM only in strict accordance with this SOP.

(e) Complete and record all checks required on the Usage Log sheet before starting the SEM.

(f) In the event of an abnormal shutdown or equipment malfunction, stop all operations, make a note on the Usage Log and contact the Lab Manager.

(g) Ensure that the SEM lab facility and the computer screen is securely locked when an Authorized User is not present or in the immediate vicinity.

(h) Ensure that radiation surveys are current prior to commencing routine operations.

c. Other Personnel.

(1) Other personnel, midshipmen and guests, are allowed access to the Diffraction Lab Facility. All other personnel entering the diffractometer laboratory shall:

(a) Be accompanied by an Authorized User.

(b) Only operate the instrument under the guidance of an Authorized User.

(c) Control their radiation exposure in accordance with the ALARA principle – As Low As Reasonably Achievable.

(d) Follow all directions of the Authorized User.

(e) Follow all provisions of this SOP.

d. The RSO and/or Assistant Radiation Safety Officer (ARSO) shall:

(1) Have authority to terminate immediately any operation that he/she finds to be unsafe or not in accordance with this or other applicable SOPs.

(2) Ensure that area monitoring surveys are conducted and recorded every 6 months and prior to commencing routine operations after equipment maintenance or modification.

(3) Conduct initial radiation safety training of Authorized Users as required by reference (a), provide appropriate annual refresher training and certify the satisfactory completion of such training.

(4) Provide support as necessary to ensure compliance with higher authorities.

(5) Provide immediate assistance should any radiological safety incident occur.

6. Review. This SOP will be in effect for the entire time that the JEOL JSM-6010LA SEM is installed and operational at the U.S. Naval Academy. The SOP will be reviewed on an annual basis by the Lab Manager and the RSO to ensure that the procedures are current and in full compliance with reference (a). Authorized users must review this SOP on an annual basis. This SOP must undergo complete review every five years by the US Naval Academy Radiation Safety Committee.

7. Approvals. This SOP and all references contained therein have been reviewed and approved by the US Naval Academy Radiation Safety Committee. Operation or maintenance of the SEM in any manner or in reference to any procedure not described herein must first be approved by the RSO and RSC. Approval of such requested operation, procedure, or both will be temporary and will not constitute general approval by the RSO of the requested operation or procedure.

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Physics Department

Radiation Safety Officer

FACILITY DESCRIPTION

The JEOL JSM-6010LA SEM is a 20-kV scanning electron microscope. The instrument uses a filament-produced electron beam to perform visual and energy dispersive spectrometry (EDS) studies of samples on scales from 500 μm down to 5 nm. The primary purpose of the “Atoms laboratory” at USNA is to give midshipmen practical experience using research grade analytical equipment and methods to observe and study quantum phenomena such as optical pumping, the Zeeman effect, interactions of high energy particles with matter, and the properties of materials on various length scales. The laboratory is also used to support midshipman and faculty research.

The SEM, its operating controls, experimental areas and data acquisition/analysis computers are all located in Chauvenet Hall, CH-046, as illustrated in figure 1. This room is surrounded by one exterior wall on the east side, research labs on the north and south sides, and a hallway on the west side.

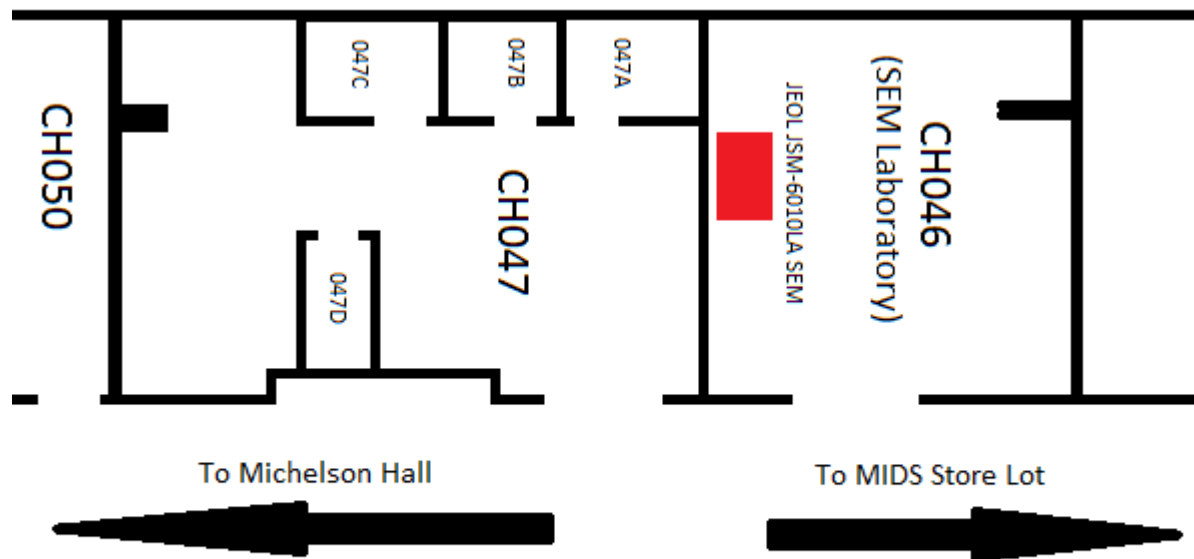


Figure 1: The JEOL JSM-6010LA SEM is located in Chauvenet 046, a room on the eastern wall of the Chauvenet Hall zero deck (lab deck).

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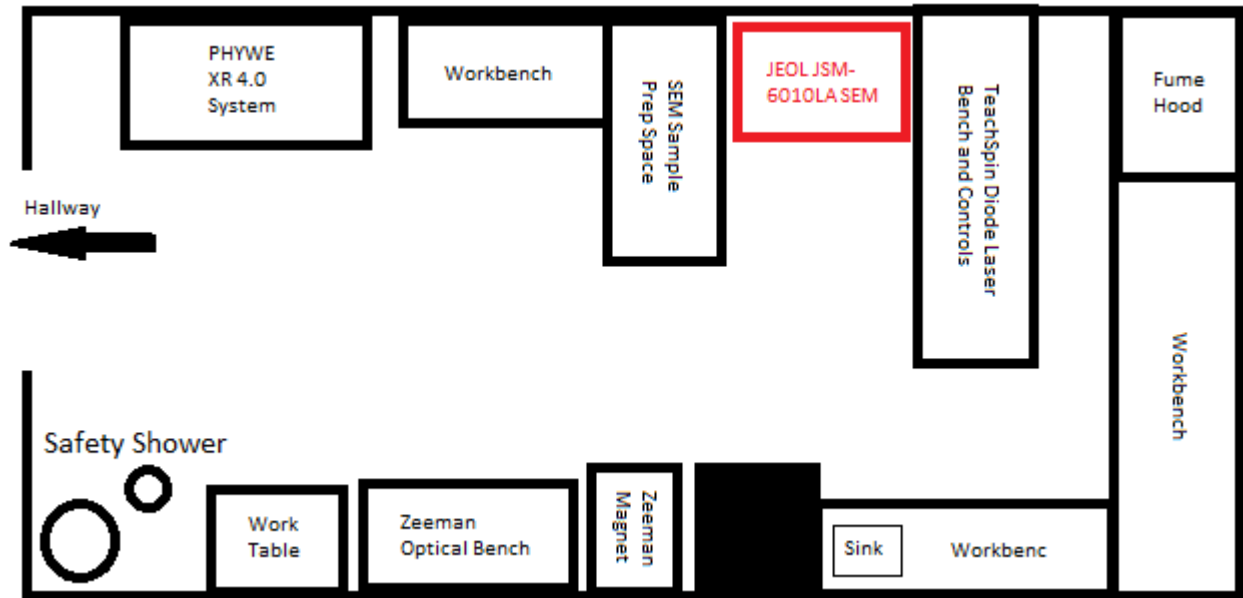


Figure 2: Location of SEM equipment in Chauvenet 046.
The relative orientation of the diagram is specified.

Entrance to the SEM room CH-046 is available through the hallway door. The hallway is kept locked when the room is unattended. The room has one standard floor drains into the sanitary sewer system. There is 1 window in the room on the east wall. The hallway door is posted with emergency contact information. There is no radiological hazard when the SEM is off. A manually operated status light above the hallway door outside Chauvenet 046 indicates operational status of any machine source in the room.

Volatile radioactive materials are not used in Chauvenet 046. The SEM does not generate any radioactive material or measurable field during operation.

JEOL JSM-6010LA Scanning Electron Microscope

The JEOL JSM-6010LA SEM is a 20-kV scanning electron microscope designed for the visual inspection of samples on the micron and sub-micron scale as well as energy dispersive spectrometry via collection of characteristic X-rays produced by the interaction of the electron beam with material in the sample.



Figure 3: View of the SEM and operation console.

Experimental Area

The SEM is equipped with a 20 kV Wehnelt filament electron gun, secondary and backscatter electron detectors, and a silicon drift EDS detector with 133 eV resolution. Operating parameters for the probe current and chamber pressure are 1 pA to 0.3 μ A and 10 Pa to 100 Pa respectively. The beam is produced by a pre-centered tungsten hairpin filament and focused by a system consisting of a 2-stage zoom condenser lens, a super conical minilens, a stigmator, and scanning coil. The beam is completely contained in the column and sample chamber of the device. To prevent exposure of personnel to the particle beam, interlocks keep the beam from entering the sample chamber unless the enclosure is secure (referred to as “beam blanking”). Further inherent protection is provided by the evacuation of the sample chamber. The chamber cannot be physically opened when under vacuum.

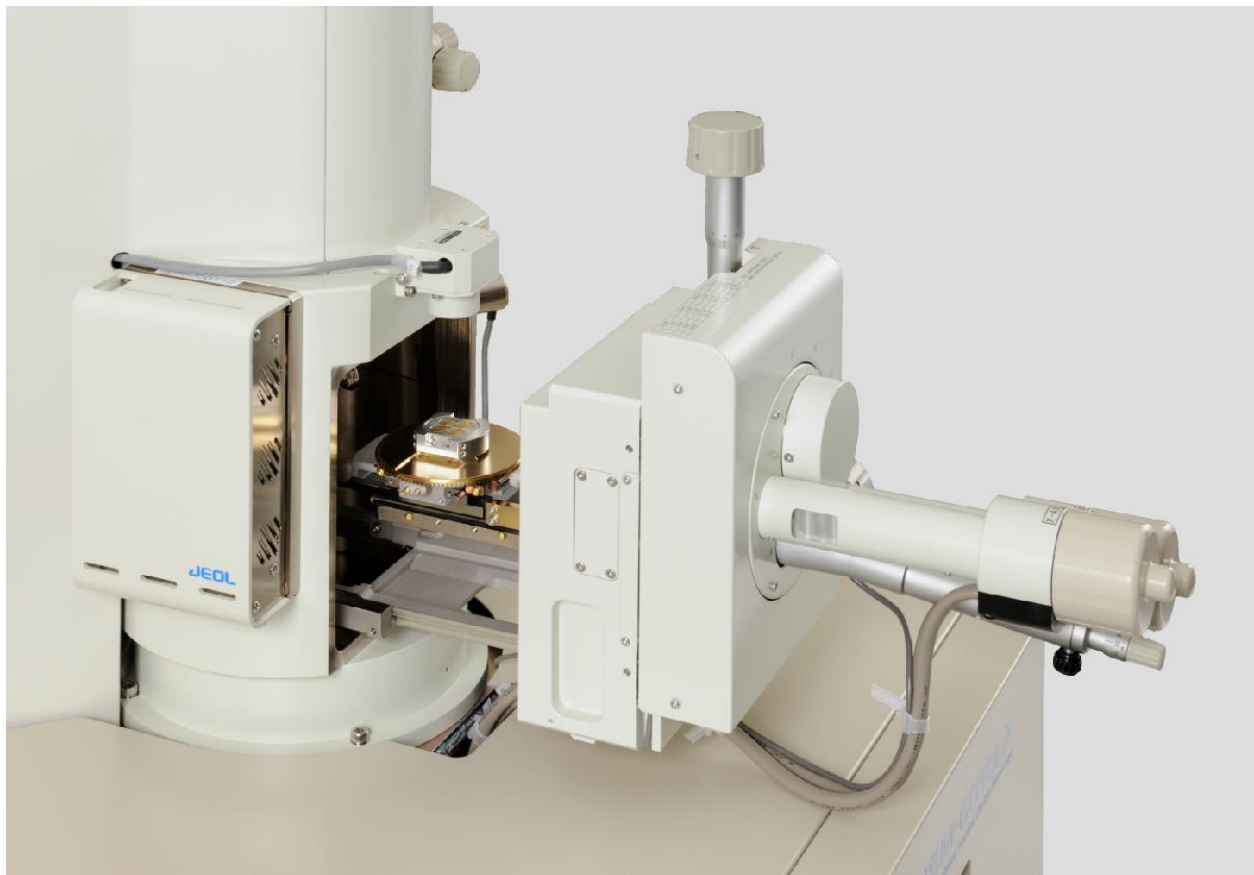


Figure 4: View of the open sample chamber and stage of the JEOL JSM-6010LA SEM.

FACILITY RADIATION SAFETY EVALUATION

Hazard Discussion

The JEOL JSM-6010LA SEM is a scanning electron microscope, the regulations of which are found in Section 13.4 of reference (a). Issues concerning radiation safety that pertain to the SEM are:

1. Radiation levels outside of the sample chamber - No radiation field measurable with RASO-approved RADIACS has been observed.
2. Repairs of the SEM are only to be performed by personnel who have been trained and/or designated by JEOL or its authorized representatives. A Radiation Survey must be performed in any of the following circumstances:
 - a. Prior to placing a new unit in service and annually thereafter not to exceed 12 months
 - b. Immediately after any maintenance or repair. The RSO may review and accept the post maintenance survey conducted by the vendor.
 - c. After shock, earthquake, etc., or suspected deficiency
 - d. Whenever equipment is modified or relocated.
3. The SEM does not activate material and, as a result, does not produce any airborne radioactivity.

GENERAL CONTROLS AND PRACTICES

Physical Controls for Chauvenet 046

Security:

1. The SEM room is a Controlled Area and shall be locked when no authorized user is present. Individuals who are not authorized users may be in the room at any time, but the SEM console must be off or password locked if no authorized user is present.
2. When the SEM is in operation, the room shall be locked to prevent unanticipated access.
3. The SEM vacuum and high voltage system is powered on by a keyed switch.
4. The PC is password controlled. The control software will not start unless the SEM is powered on.
5. The key shall be removed from the switch and secured when the SEM has been powered down. When not in use, keys must remain in the possession of qualified Authorized Users.

Warning Devices:

1. Lighted warning signs.
 - a. The vacuum status buttons light up on the front panel of the SEM cabinet indicating the vacuum status of the sample chamber. The SEM cannot produce an electron beam unless the sample chamber is closed and evacuated. No X-rays are produced if the electron beam is not in operation.
 - b. A button in the upper right of the software graphical user interface (GUI) indicates the status of the beam as “ON” or “OFF” and is colored green.



Figure 5: Front of SEM showing beam column and front control panel on front of cabinet.



Figure 6: Detail of front control panel showing keyed start switch and control buttons for vacuum system. Each button lights up to show current status of system (solid for stable, flashing for transitions).

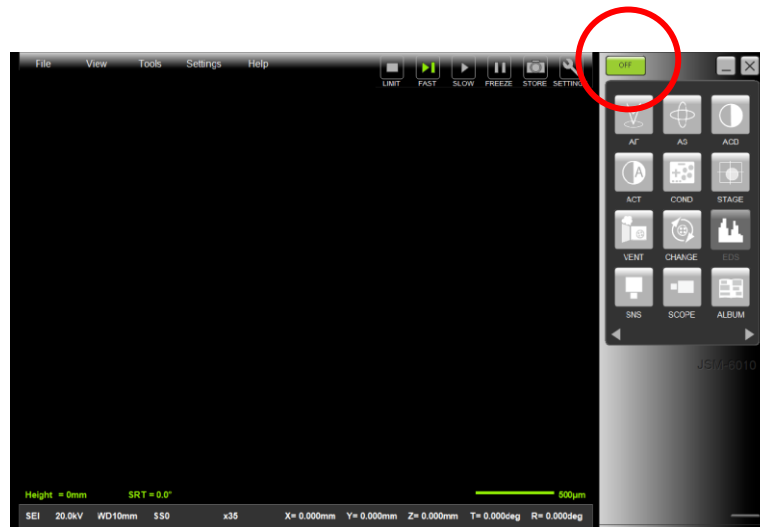


Figure 7: Main screen of the software graphical user interface (GUI). The beam status and blanking button can be seen circled in red in the upper right (colored green with “OFF” written across it).

2. Other signs and barriers.

a. A sign is posted on the SEM cabinet bearing the conventional yellow and magenta radiation symbol and the words: "**CAUTION: RADIATION. THIS EQUIPMENT PRODUCES RADIATION WHEN ENERGIZED. TO BE OPERATED ONLY BY QUALIFIED PERSONNEL**".

b. The entrance to CH-046 is posted with yellow and magenta signs bearing the words "**CAUTION: RADIATION. THIS EQUIPMENT PRODUCES RADIATION WHEN ENERGIZED**".

GENERAL OPERATING PROCEDURES

These procedures represent a basic outline for safely operating the SEM. More detailed data collection procedures can be found in Reference (c). Only qualified authorized users are allowed to operate the SEM. An Authorized user is completely responsible for ensuring this instrument is used in a safe manner. If the instrument is left alone while on, the controlling computer must be locked (password required to resume) and the room secured (door locked).

1. Record the starting time and date on the sheet in the Usage Log. Enclosure (8) contains a copy of the usage log sheet.
2. If the SEM is not powered up, proceed with step 3. The SEM is generally left in off condition. If the SEM is already powered on, proceed to step 10.
3. Obtain permission to operate the SEM and the key to the SEM from the SEM Laboratory Manager.
4. Check that the oil level in the rotary pump (located on the floor behind the SEM cabinet) is above the lower limit. See Figure 8.

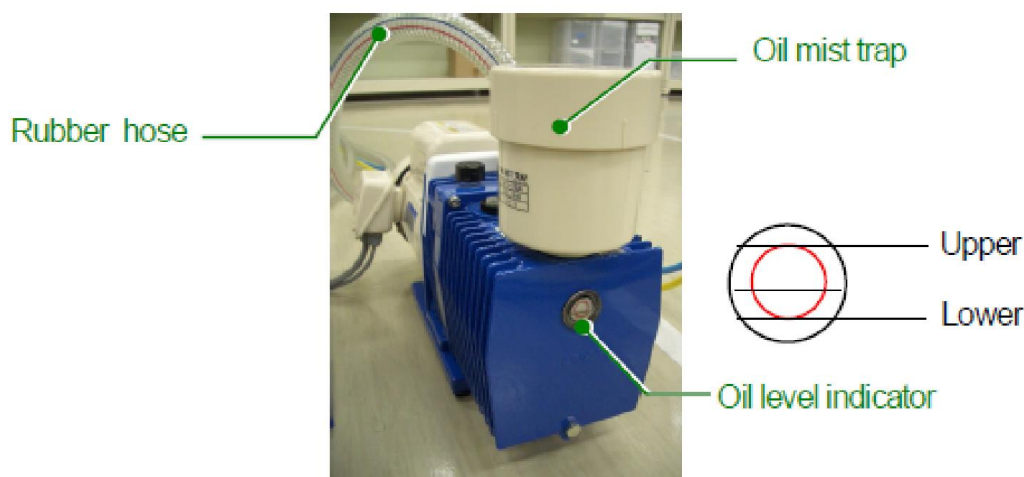


Figure 8: Rotary pump oil level indicator.

5. Check that the main power cable is connected to the wall outlet.
6. Check that the MAIN BRAKER on the rear of the SEM cabinet is set to "ON".
7. Check that the power cable of the controlling PC is connected to the instrument.

8. Insert the key and turn it to the “START” position (see Figure 9), then release (the key will stop at the “I” position). The pump will turn on and the system will begin pumping down the sample chamber (indicated by flashing “EVAC” light on the front panel of the SEM cabinet).

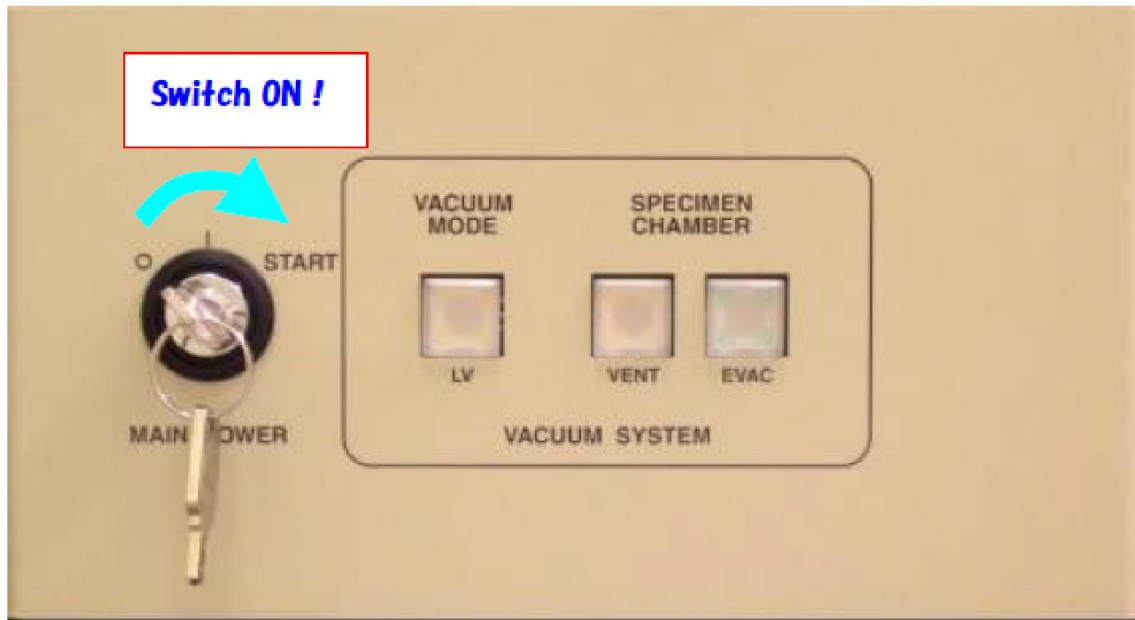


Figure 9: Detail of the front control panel on the SEM cabinet.

9. After about 10 seconds turn on the controlling PC if it is not already on.

10. Start the InTouch Scope Software in either “Normal” mode or “Navigation” mode by double clicking on the respective desktop icons. The icon with the green and yellow oval is for “Navigation Mode”. See Figure 10 below.



Figure 10: Desktop icons for starting the user interface. The icon on the left is for “Normal” mode while that on the right is for “Navigation” mode.

11. Once the control GUI has started, follow the on-screen prompts and the instructions in Reference (c) if the sample is already in place in the sample chamber (possibly from previous measurements). If the sample must be changed, proceed to step 12.



Figure 11: Change sample button on GUI.

12. Sample Exchange: Begin by blanking the beam with the green button at the top of the GUI (circled in red in Figure 11 above). Ensure the button reads “OFF”.
13. Next click the “Change” button (boxed in red and white dashed lines in Figure 11 above).
14. Select either “Set Specimen” or “Observation Conditions” in the popup window that appears (Figure 12).

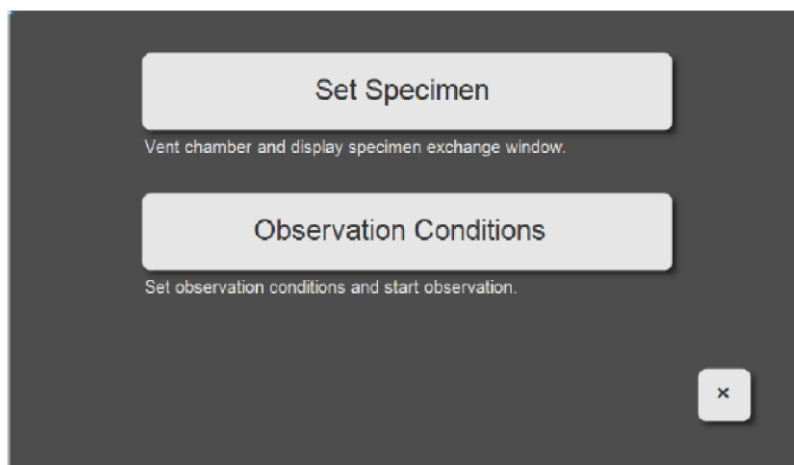


Figure 12: Sample exchange popup window.

15. Once the sample chamber is fully vented (VENT button stops blinking on front panel), the chamber may be opened and the sample may be removed. A different popup will appear depending on which option was chosen in step 14 above. Follow the on-screen prompts to complete the sample exchange. A basic flow diagram is provided below for the two processes.

Be certain to close the sample chamber before clicking on the green “START” button!

The specimen exchange steps are as follows.

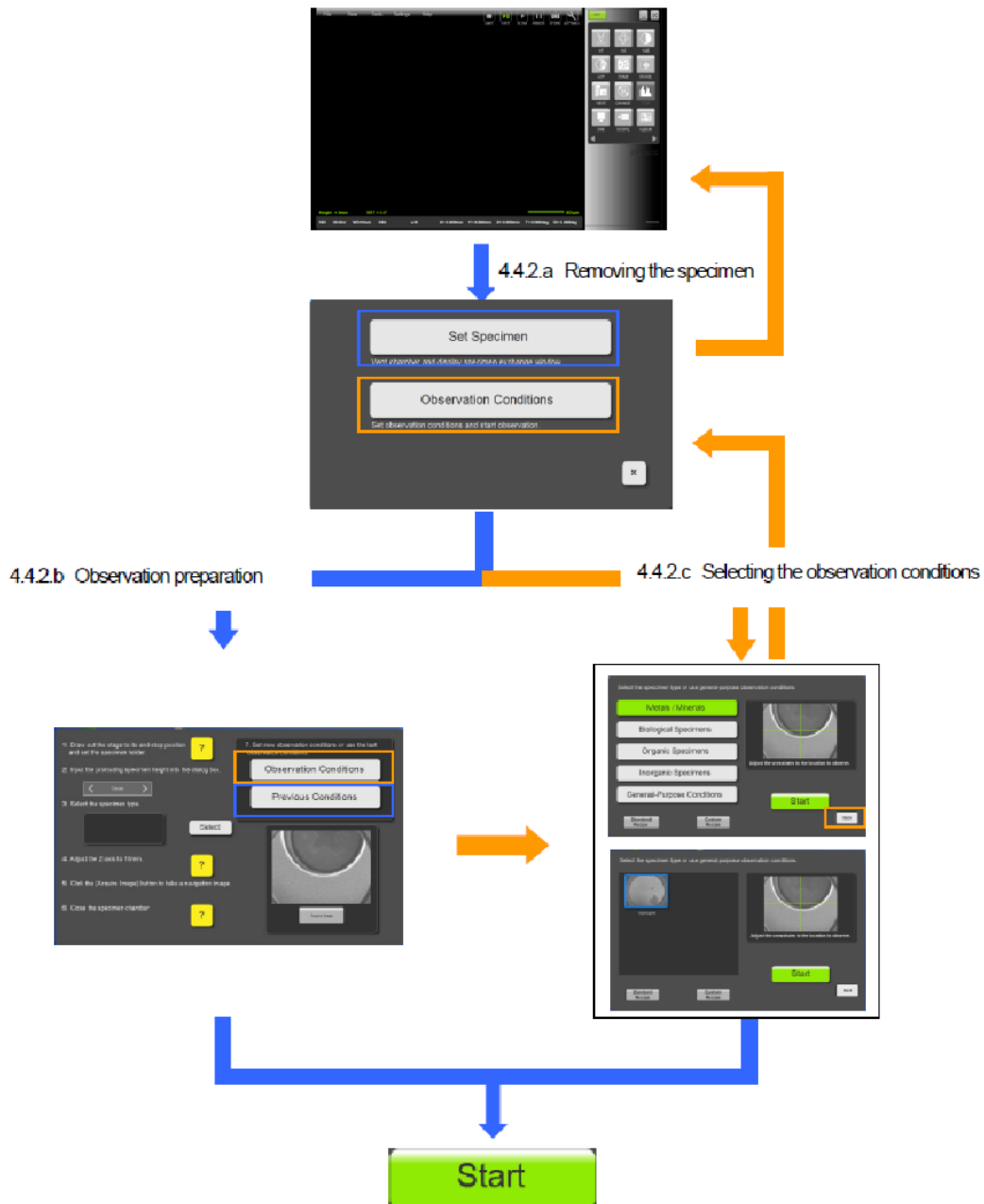


Figure 13: Sample exchange flow chart.

16. The instrument should only be shut down by the Lab Manager using the procedures that are found in Section 4.2.2 of the Basics/Operation Manual (page 4-6 of reference (c)) and summarized as follows:

- a. Evacuate the chamber (optional).

- b. Close the software GUI.
- c. Turn off the controller PC (optional).
- d. Turn key to “O” on front control panel.
- e. Remove key.
- f. Ensure room is locked upon departure.

Maintenance Procedures

Filament replacement, cleaning, and maintenance shall be performed according to reference (c) section 7.5 and ONLY by a qualified authorized user upon permission from the SEM Laboratory Manager. Hardware repairs procedures will be performed by personnel authorized and/or certified by JEOL or its designated service providers.

EMERGENCY PROCEDURES JEOL MODEL JSM- 6010LA SEM

In the event of an emergency (fire, flood, serious malfunction of SEM, etc.), the following procedures must be followed.

1. Shut down the SEM by turning the MAIN POWER key switch to the OFF position and removing the key (see Figure 14 below).

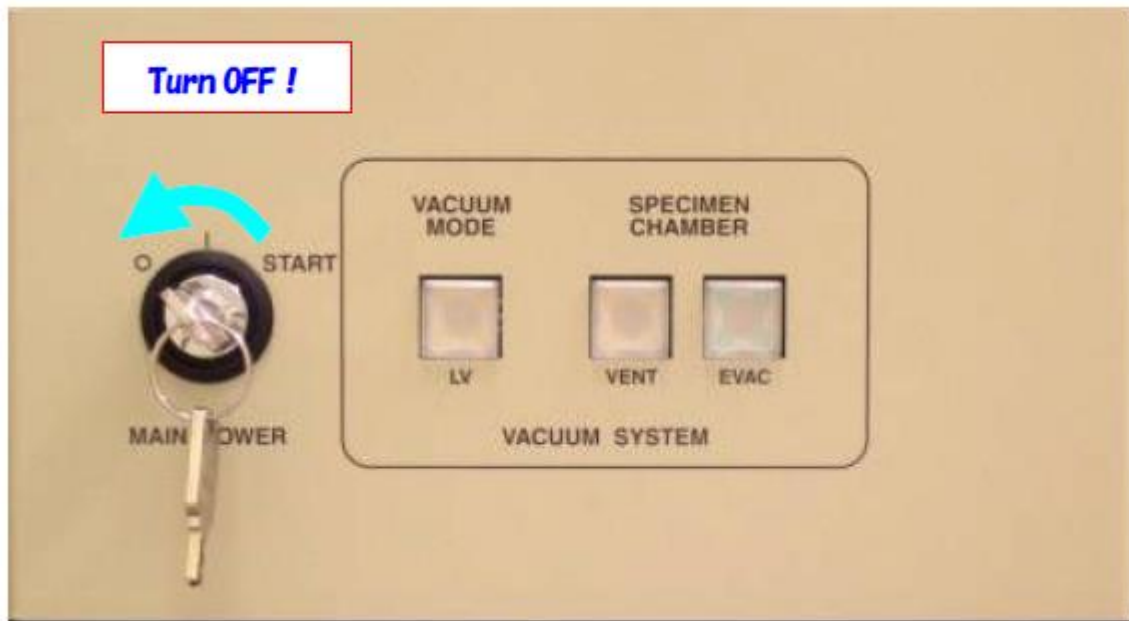


Figure 14: Detail of front panel with indication of how-to shutdown the system in the event of an emergency.

2. Secure cooling water flow to the SEM. Note that cooling water should automatically stop flowing when MAIN POWER is secured. If cooling water flow does not stop, pull the cooling unit electrical plug from it associated outlet to secure power.
3. Notify the RSO and the SEM Laboratory Director.
4. Notify other emergency contacts, such as fire or police departments, if appropriate.

RADIATION MONITORING

Personnel Radiation Monitoring Equipment:

Authorized Users of the JEOL JSM-6010LA SEM are non-radiation workers and do not require personnel dosimetry.

Equipment Radiation Monitoring Equipment:

1. According to Section 13 of reference (a), a radiation survey of the SEM, is required to be performed every 6 months, after service, and whenever an issue with the beam alignment is suspected.
2. Surveys will be performed with an Ion Chamber Survey Meter (RADIAC), suitable for X-ray radiation. Only RASO approved meters shall be used to measure radiation levels needed to satisfy legal and health related documentation requirements.
3. RADIACS must be in calibration and source-checked prior to use.
4. The survey will be performed and documented by the RSO or ARSO.
5. Full requirements and the sheet used for surveys are found in Enclosure (8).

Permanent Area Radiation Monitors:

The SEM does not produce a measurable radiation field outside the shielding of the device. Therefore, no area radiation monitor is installed.

AUTHORIZED USER QUALIFICATION REQUIREMENTS

As the SEM presents little to no risk in regard to radiation exposure, all authorized users will be deemed non-radiation workers.

To be qualified as an authorized user, the individual shall:

1. Complete Non-Radiation Worker Training through the Radiation Safety Office OR the laboratory manager
2. Complete training with the Laboratory Manager to identify radiation safety hazards, warning systems, and precautions as well as understand the guidelines to safely operate the SEM.
3. Be acknowledged and approved by the department chair.
4. Be acknowledged and approved by School Leadership.
5. Be voted on and approved by the RSC.

The list of qualified Authorized Users shall be maintained by the SEM Laboratory Manager using the sheet in this SOP. Any qualified Authorized User, who does not operate the instrument within the span of one year, must be requalified.

Individuals who will independently use the instrument with immediate oversight of an Authorized User will be required to complete Non-Radiation Worker training, but will not need to become an Authorized User. This would extend to midshipmen, who would use the instrument while in the presence of an Authorized User.

Qualification sheets are found in Enclosure (8).

RECORDS – SHEETS and LOGS

Utilization Log:

1. Each use of the SEM shall be documented by the Authorized User in the Usage Log maintained for that purpose at the SEM control console. Data recorded shall include the following items.

- a. The instrument manufacturer, make, model, and serial number(s).
- b. The name and location of the lab where the instrument is used.
- c. Laboratory Manager's name, office, and office phone
- d. User name and signature.
- e. Date/time on and Date/time off (use date and time) and Total Exposure Time.
- f. Maximum operating voltage
- g. Maximum operating current
- h. Target type and material
- i. Remarks on samples measured
- j. Verification that radiation surveys are current
- k. Date and signature of RSO log review

2. The Usage Log sheet is shown on page 2 of Enclosure (8).

Radiation Survey Log:

1. A complete radiation survey for each source, with the diffractometer running under typical conditions, shall be conducted every six months or whenever maintenance has been performed which might result in an increase in radiation levels. Data recorded shall include the following.

- a. The date and time of the survey
- b. The name of the surveyor
- c. Date of last survey and interlock check
- d. Verification of the interlock check

- e. Survey meter used to include: Model #, Serial #, Calibration and Due date
 - f. System parameters including: beam energy, current, and sample (if any)
 - g. Diagram showing survey points
 - h. Readings (mR/hr) at the survey points
 - i. RSO or ARSO signature
2. This radiation survey satisfies the requirement for 6-month radiation protection surveys and 6-month interlock checks.
3. The sheet for the radiation survey is shown on page 3 of Enclosure (8).

Authorized User Authorization Sheet: This sheet is found on page 4 of Enclosure (8).

Lists of Qualified Authorized Users: This sheet is found on page 5 of Enclosure (8).

EXAMPLE USAGE LOG
(JEOL MODEL JSM- 6010LA SEM, serial number MP1100002290229)

[illegible]

EXAMPLE RADIATION SURVEY
JEOL MODEL JSM- 6010LA SEM, Chauvenet Hall (CH-046), Serial #
MP1100002290229

Date _____ Time _____ Performed by _____

Select one: _____ General radiation survey (semiannual)

Date of last: General radiation survey (semiannual) _____
 Interlock check (semiannual) _____

Interlocks verified (initials) _____

Survey Meter

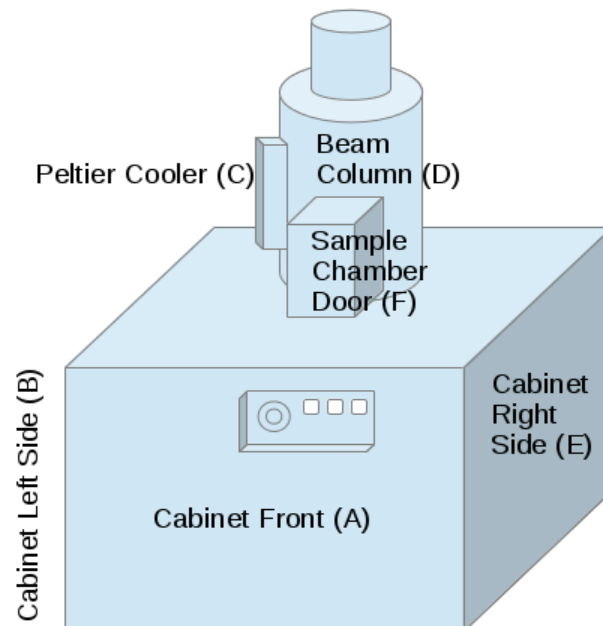
Model # _____
 Serial # _____
 Calibration Date _____
 Due Date _____

System Parameters

Max kV _____
 Max current _____
 Sample _____

Survey points are 5 cm from the enclosure.

Readings (mR/hr) A _____ B (at seal) _____ C _____ D _____ E (at seal) _____ F _____



Radiation Field at Control Console _____

RSO review: _____

**EXAMPLE AUTHORIZED USER QUALIFICATION REQUIREMENTS
JEOL MODEL JSM-6010LA SEM**

Name: _____

1. Familiar with radiation health requirements of NAVMED P-5055 and RAD-010.

Laboratory Manager/Date

2. Demonstrated understanding of operating procedures, and fully able to safely operate the JEOL Model JSM-6010LA Scanning Electron Microscope. This includes demonstrating the ability to:

Topic	LM Initials
Identify major components of the SEM both externally and internally.	
Safely load and unload samples from the SEM maintaining cleanliness including maintaining sample clearances required to prevent damage to the ring backscatter and EDS detector in the chamber.	
Turn the beam on and off	
Safely maneuver the stage with a specimen loaded	
Return the SEM to STANDBY condition	
Read and understands the SEM SOP (annual requirement).	
Understands radiation safety hazards, warning systems and safety precautions associated with the instrument.	

3. Completed Non-Radiation Worker Training.

Laboratory Manager/Date

4. Qualified.

Laboratory Manager/Date

Annual Review Date	Authorized User Signature

**EXAMPLE AUTHORIZED USER QUALIFICATION REQUIREMENTS
JEOL MODEL JSM-6010LA SEM**

Name: _____

1. Qualified by Laboratory Manager.

Laboratory Manager/Date

2. Department Chair Authorization (Physics).

Department Chair/Date

3. Dean School of Math and Science Authorization.

Dean Math and Sci/Date

4. RSO Review.

RSO/Date

5. Designated authorized user by RSC majority vote.

RSC Chair/Date