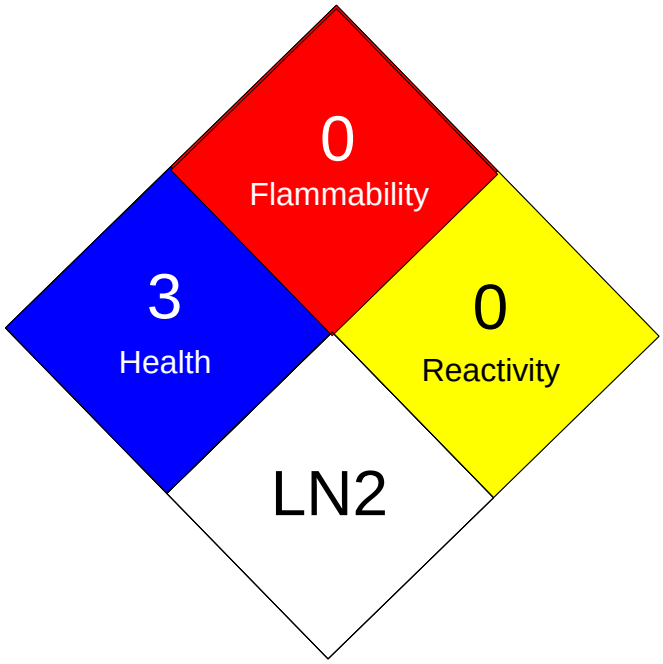
WVU SHARED RESEARCH FACILITIES

TEM Lab
Chemical Hygiene Plan

B63 Engineering Science Building



Emergency Contact:

Marcela Redigolo, Ph.D.
Office: G75D Engineering Sciences Building
Cell Phone: (304) 680-3007

SECTION 1: NFPA CHEMICAL HAZARD LABEL

NFPA Rating - The National Fire Protection Association (NFPA) has developed a standard system (ANSI/NFPA 704) for indicating the health, flammability, and reactivity hazards of chemicals. In addition, a special precaution symbol may be used where necessary.

This system of identifying hazards associated with various materials was developed primarily for fire protection and emergency personnel but can be useful to anyone who needs to handle potentially hazardous material. As stated in NFPA 704, "This standard provides a simple system of readily recognizable and easily understood markings, which will give at a glance a general idea of the inherent hazards of any material and the order of severity of these hazards as they relate to fire prevention, exposure, and control."

General Rating Summary: (Also in Table 1)

Health (Blue)

0- Hazard no greater than ordinary material
1- May cause irritation; minimal residual injury
2- Intense or prolonged exposure may cause incapacitation: residual injury may occur if not treated
3- Exposure could cause serious injury even if treated
4- Exposure may cause death

Flammability (Red)

0- Will not burn
1- Must be preheated for ignition, flashpoint above 93°C (200°F)
2- Must be moderately heated for ignition flashpoint above 83°C (100°F)
3- Ignition may occur under most ambient conditions, flashpoint below 83°C (100°F)
4- Extremely flammable and will readily disperse through air under standard conditions, flashpoint below 83°C (100°F)

Instability (Yellow)

0- Stable
1- May become unstable at elevated temperatures and pressure, may be mildly water reactive
2- Unstable; may undergo violent decomposition, but will not detonate. May form explosive mixtures with water
3- Detonates with strong ignition source
4- Readily detonates

Special Symbols (White)

OX- oxidizer
~~**W**~~- Water reactive, use no water

Non-Standard Symbols (also put in the White area)

Note: These hazard symbols are not part of the NFPA 704 standard, but are occastionally used in an unofficial manner. The use of non-standard symbols or text may be permitted or required by the authority having jurisdiction.

COR - corrosive

ACID – acids

ALK – alkalines

BIO – biological hazard

POI – poisonous

☢ – radioactive

CYL or **CRYO** – cryogenic (e. g. Liquid Nitrogen)

LN2 – Liquid Nitrogen

NFPA Rating Explanation Guide

RATING NUMBER	HEALTH HAZARD	FLAMMABILITY HAZARD	INSTABILITY HAZARD
4	Can be lethal	Will vaporize and readily burn at normal temperatures	May explode at normal temperatures and pressures
3	Can cause serious or permanent injury	Can be ignited under almost all ambient temperatures	May explode at high temperature or shock
2	Can cause temporary incapacitiation or residual injury	Must be heated or high ambient temperature to burn	Violent chemical change at high temperatures or pressures
1	Can cause significant irritation	Must be preheated before ignition can occur	Normally stable. High temperatures make unstable
0	No hazard	Will not burn	Stable

RATING SYMBOL	SPECIAL HAZARD
ALK	Alkaline
ACID	Acidic
COR	Corrosive
OX	Oxidizing
☢	Radioactive
₩	Reacts violently or explosively with water
₩ OX	Reacts violently or explosively with water and oxidizing

This chart for reference only - For complete specifications consult the NFPA 704 Standard

NFPA-Chart_1 www.ComplianceSigns.com

Table 1: NFPA Rating Explanation Guide

SECTION 1.1: EMERGENCY CONTACTS

In case of FIRE, INJURY, or EMERGENCY ASSISTANCE, contact in the following order:

9-911 from any campus phone
or
Campus Security
Phone: **(304)293-3136 (293-COPS)**

Then Call

Marcela Redigolo, Electron Microscopy Manager
Cell Phone: **(304) 680-3007**
Office Phone: **(304) 293-9973**
Room: **G75D ESB**

or

Kolin Brown
Cell Phone: **(304)366-6551**
Office Phone: **(304)293-9683**
Room: **G75D ESB**

If no one responds to any numbers above, then contact:

Kenny Claudio
Cell Phone: **(304) 216-4858**
Office Phone: **(304) 293-4091**
Room**: 373A MRB**

Royce Watts,
Cell Phone: **(304) 288-6762**
Office Phone: **(304) 293-4124**
Room: **377A MRB**

For non-emergency assistance please contact:

Marcela Redigolo, Electron Microscopy Manager
Cell Phone: **(304) 680-3007**
Office Phone: **(304) 293-9973**
Room: **G75D ESB**

SECTION 1.2: EMERGENCY EVACUATION PROCEDURES

In case of Fire or Chemical Spill all users should evacuate the laboratory immediately!

- Use the shortest, unobstructed path to the exterior of the building.

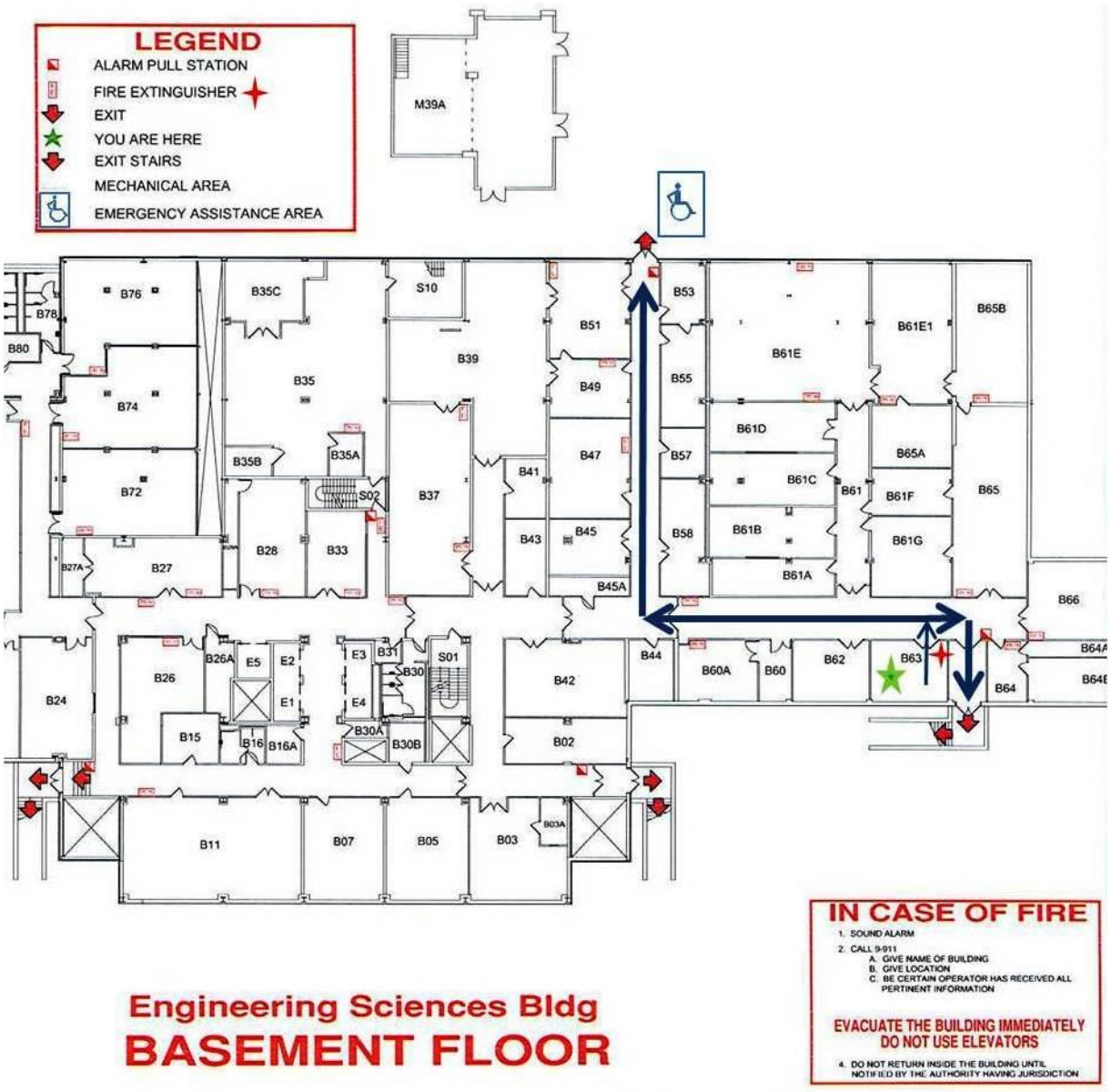


Figure 1: TEM Lab Evacuation Plan (room B63 in the basement of the Engineering Sciences Building. Closest exits marked with dark blue arrows

- Evacuate the building by pulling the fire alarm pull station when exiting the building. All basement exits have a fire alarm pull station.
- Call the emergency contacts.

SECTION 1.3: SHUTDOWN PROCEDURES

If a dangerous situation is evident (smoke, fire, sparks, etc.), ONLY if it is safe to do so, should a user attempt to shut down a system. The user should then notify all other persons in the lab to evacuate immediately. After evacuation, a user should contact proper emergency personnel from a safe place.

If no one is available and a machine is not acting as expected then the user should attempt to put the machine in its default mode; do not leave the machine running in an abnormal state! If the machine cannot be placed in its default mode, the user should stay by the tool and contact one of the WVU Shared Facilities Staff Members. If it becomes necessary to leave the tool then the user should leave a large, legible note on the machine stating that the tool is down, and the user's contact number.

Listed below are the procedures to place the TEM into a safe default mode, or to shut down. Shutdown procedures may also be found on the cover of the microscope's logbook or in the Standard Operating Procedure found in Appendix A.

To place in default mode:

- **Turn the filament OFF on the TEMCON software.**
- **Turn HT OFF on the TEMCON software.**

To shutdown (this emergency shutdown will turn OFF power to the entire microscope):

- **Press the EM STOP (Fig. 2) switch, located inside the microscope panel under the left side of the microscope. The panel has the name of the microscope in the front. Just press the panel once for it to open and you can find the EM shut off button inside.**

OR

- **Turn OFF the electrical breaker box, located at the wall to the left side of the microscope.**

Figure 2: Photo of the emergency (EM) button for the transmission electron microscope.

SECTION 1.4: EMERGENCY RESPONSE EQUIPMENT

First Aid kit is located inside the lab, **B63**, attached to the wall by the entrance door.

The nearest **AED** (Emergency Defibrillator) is located in the **MRB Atrium**. A second AED can be found on the 1st floor lobby of the Engineering Sciences Building (ESB).

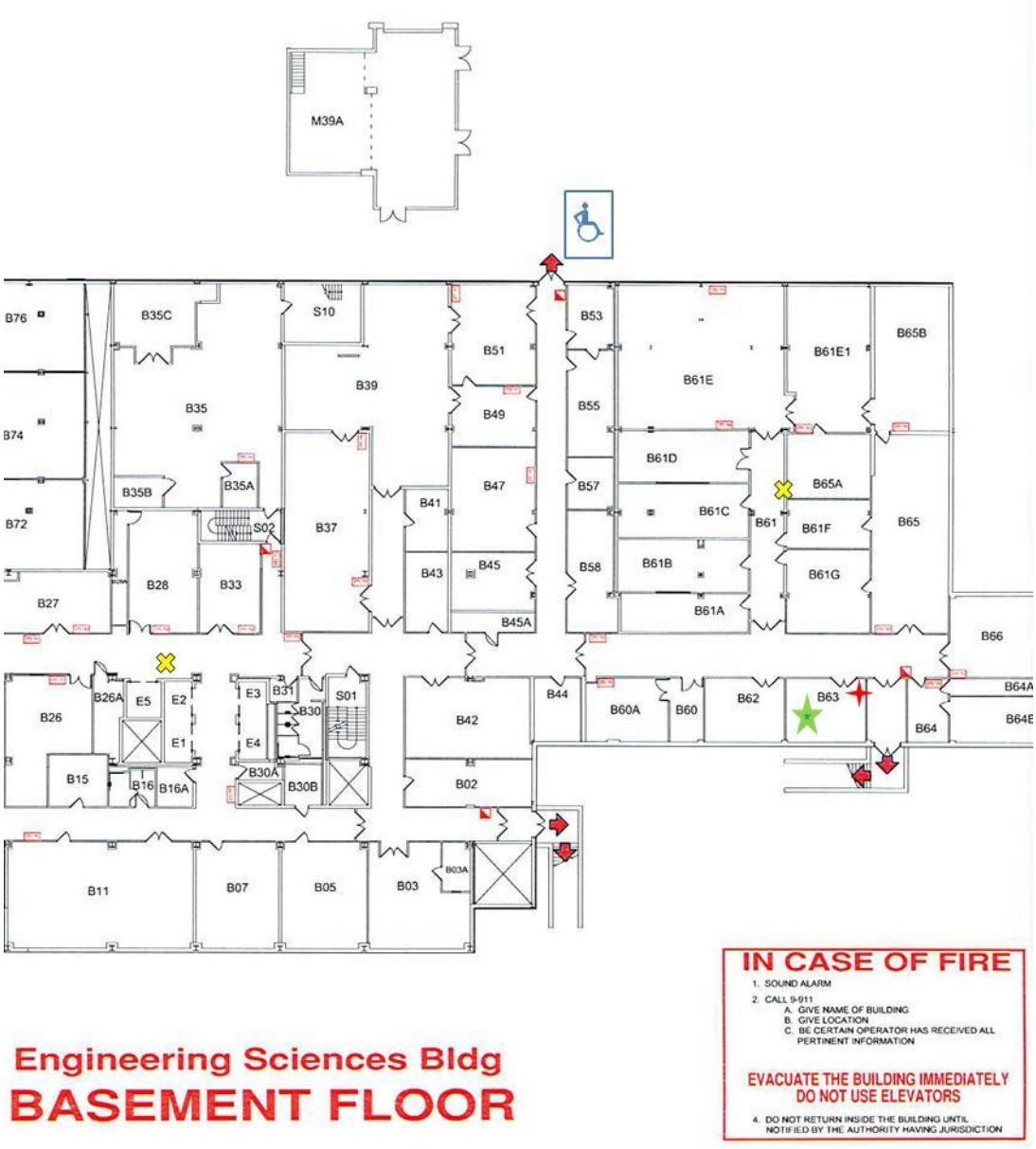


Figure3: Location of the safety showers (✖), eyewash stations (✖) and TEM lab fire extinguisher (✦).

Safety shower is located in the ESB basement, inside the B61 area (in the hall after the glass doors). Another s**afety shower** but with **eyewash station** is located in the ESB basement, by the freight elevators. Figure3 indicates the location of these safety showers and eyewash station using ✖ symbol.

An additional **safety shower** and **eyewash station** is located in the corridor outside the cleanroom (G75) in the ground floor of the Engineering Sciences Building (ESB).

The **Chemical Hygiene Plan** and **MSDS**s are located inside the lab by the entrance door, **B63 ESB**. The lab also has a fire extinguisher, type ABC, indicated in figure3 by the cross ✦.

All basement exits count with a fire alarm pull station.

SECTION 2: LAB OVERVIEW AND DESCRIPTION

The Transmission Electron Microscope Laboratory (TEM Lab) is part of the Electron Microscopy Facility, which is one of the four WVU Shared Research Facilities. This microscope is the main instrument in the TEM Lab, and provides high resolution and high magnification imaging, and electron diffraction capability for the structural analysis, and energy dispersive X-ray spectroscopy for elemental analysis (EDX).

As part of the WVU Shared Research Facilities, the TEM lab provides student and postdoctoral researchers with the opportunity to learn how to use cutting-edge materials science and engineering equipment. The facility is open to all researchers, including researchers at government laboratories and industries.

The transmission electron microscope requires Nitrogen gas and liquid Nitrogen for its daily operation. The Nitrogen gas is used to purge the specimen chamber during insertion of the sample into the microscope. The liquid Nitrogen is used to refrigerate the INCA EDX detector and to help prevent contamination of the sample by refrigerating the ACD (anti-contamination device).

The microscope also counts on water refrigeration of the main lenses and other parts. The Haskris chiller responsible for supplying and collecting the water is located in another room (B-64A) to avoid vibrations and noise that could deteriorate the high resolution data collected with the microscope. The supply and return connections for the water can be found in the back wall of the lab, clearly labeled and with gauge indicators of the water pressures in the lines.

Vacuum pumps are used in the lab to keep the required vacuum levels of the microscope column and chambers. One small bottle of pump oil is located in the lab, inside the cabinet and only the manufacturer JEOL does the change of oil during their scheduled maintenance periods or if required in any other moment. MSDS for the pump oil can be found together with the other MSDSs, in Appendix B.

Users can load their samples into one of the two specimen holders available. There is a desk in the lab that is used solely for this purpose. No sample preparation is done in this lab, thus there are no other chemicals located in this laboratory.

Figure 4 shows the approximate layout of the TEM lab, while figure5 shows the scheme of the water connections on the back wall of the lab.

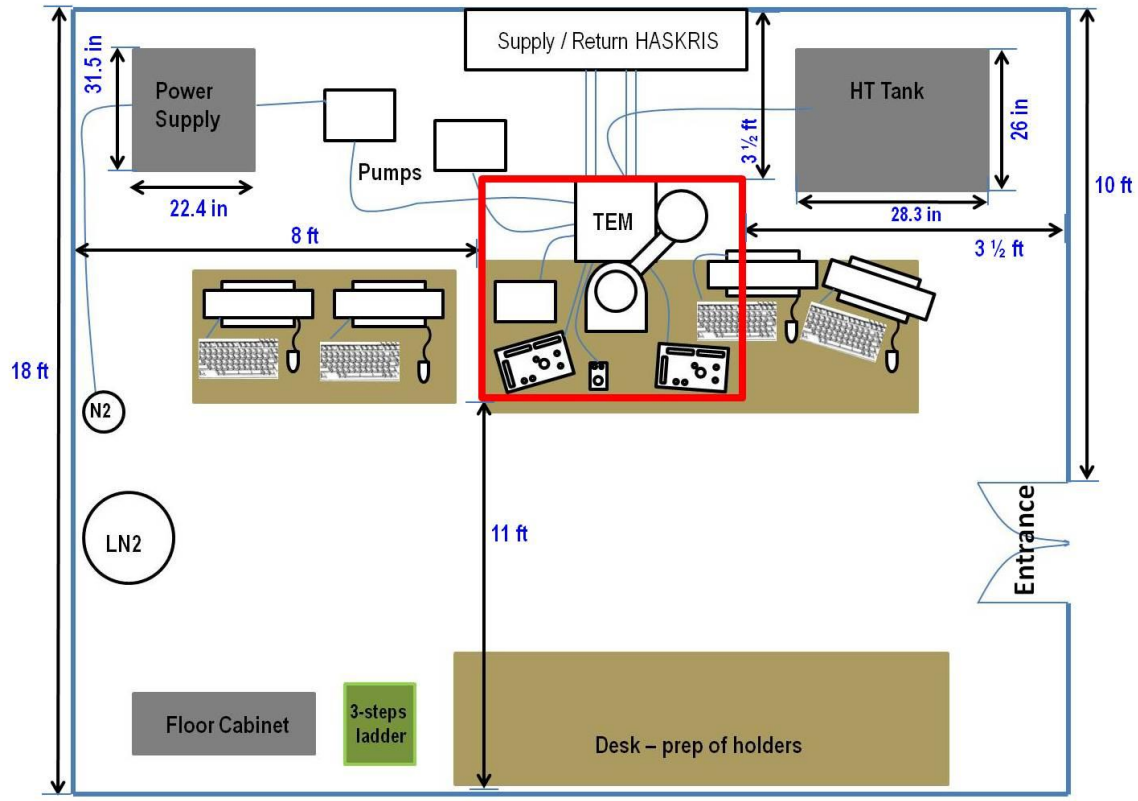


Figure4: Approximate Layout of the TEM lab.

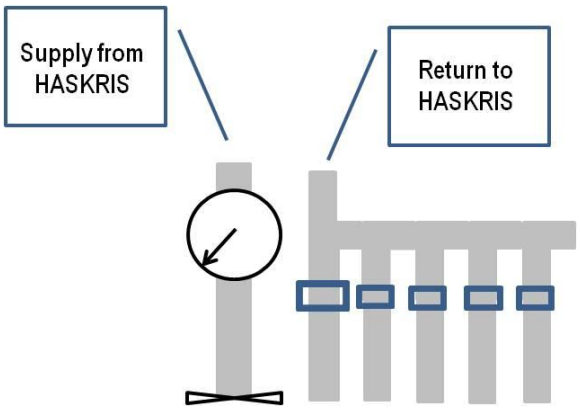


Figure5: Scheme of the water connections (supply and return) on the wall of the lab B63, receiving and sending water to the chiller located in another lab (B64A).

SECTION 2.1: LABORATORY ACCESS

Access to the WVU Shared Research Facilities is controlled through the WVU ONITY Lock system. The door to the TEM lab, B63 ESB, has one ONITY lock.

As part of the CEMR Electronic Lock policy, the TEM lab lock, B63 ESB, has been identified as a restricted lock. Access to this lock is only given to lock shop personnel, campus security, emergency services, appropriate CEMR Administration, Shared Research Facilities staff, and TEM users. A version of the CEMR Electronic Lock policy may be found on the website http://www.its.cemr.wvu.edu/cardlocks/index.php or a full, written version may be obtained upon request to the Dean's office.

The following persons are the official operators of this lock:

- Kolin S. Brown
- Lisa Sharpe

In addition, the WVU Lock Shop and WVU Card Services also have capabilities to add students, staff and faculty to this lock.

TEM users are only given access to the TEM laboratory during normal working hours after they have completed all required safety trainings and have a signed user agreement on file with the SRF Electron Microscopy Facility Manager.

The normal working hours of the TEM lab are 8 AM to 5 PM, Monday to Friday. After-hours are 5 PM to 8 AM, Monday to Friday, and full time during weekends and holidays.

SECTION 2.2: REQUIRED SAFETY TRAINING

All TEM users must complete the following three safety and equipment trainings to receive access to the facility during normal working hours.

- SRF General Lab Safety Training
- SRF Chemical Safety Training
- TEM Training

Copies of the safety presentation slides are located on the Shared Research Facilities website: http://sharedresearchfacilities.wvu.edu/safety/srfSafetyMain.html

SECTION 2.3: USER AGREEMENTS

All TEM users must have a user agreement on file with the WVU SRF Electron Microscopy Facility Manager. User agreements must be signed by the user, the user's advisor/supervisor and the user's department chair, when appropriate.

Copies of the User Agreements are located on the Shared Research Facilities website:
http://sharedresearchfacilities.wvu.edu/forms/srfForms.html

SECTION 2.4: DRESS CODE

The following dress code is required for all users entering the TEM Lab:

1. No shorts, legs must be fully covered
2. No sandals or open toed shoes, feet must be fully covered

Users operating the TEM must wear proper protection gear when working with the liquid nitrogen:

- Insulated gloves
- Insulated apron
- Full face shield

SECTION 2.5: AFTER HOUR ACCESS

After hour access to the TEM Lab is given upon request by the WVU Shared Research Facilities Electron Microscopy Facility Manager. The manager will use her own discretion to grant access when a user has demonstrated that he or she can work alone safely and handle an emergency.

SECTION 2.6: TEMPORARY USER SUPERVISION

Temporary users in the TEM lab must be accompanied by a trained TEM user or staff member at all times. Temporary users may include summer research participants, visitors or class participants.

The following chemicals are always present in the TEM Lab:

- Nitrogen Gas
- Liquid Nitrogen
- Sulfur Hexafluoride

A current chemical inventory may be found in Appendix B. This inventory is updated yearly.

SECTION 3: LABORATORY HAZARDS

Users should be aware of additional hazards when working in the TEM lab, these are:

- High voltages
- Hazardous gas
- Cryogenics

SECTION 3.1: HIGH VOLTAGE HAZARDS

The following instrument inside the TEM lab use High voltages:

- Transmission Electron Microscope (TEM)

All TEM lab users are given proper instruction on equipment operation before given authorization to use the tool. Users must follow all operational procedures outlined in the Standard Operating Procedures located in Appendix B.

WARNING: Any attempt to bypass any safety interlocks on any piece of equipment in the TEM lab may result in severe shock or electrocution.

SECTION 3.2: HAZARDOUS GAS

The following gasses are typically used by equipment inside the TEM lab:

- UHP Nitrogen
- Sulfur Hexafluoride

All cylinders in use are mounted securely against a wall. All cylinders are transported only by the company providing them to us. The company personnel come into the lab to deliver the full cylinder and collect the empty one. During this time, they connect the regulators and attach cylinders to the wall.

SECTION 3.2.1: UHP NITROGEN

Nitrogen is defined as a simple asphyxiant. It is a nontoxic gas but the liberation of a large amount in a confined area could displace the amount of oxygen in air necessary to support life.

Users must follow all equipment operational procedures outlined in the Standard Operating Procedures located in Appendix B, in the equipment logbook and during equipment trainings. Bypassing any safety interlock may expose the users to the gas.

Users should not attempt to change the gas bottle or disconnect any gas line connected to the cylinder. A SRF staff member should be contacted if the cylinder needs to be changed.

Prompt medical attention is mandatory in all cases of overexposure to nitrogen. Quick removal to uncontaminated area is most important.

SECTION 3.2.2: SULFUR HEXAFLUORIDE

Sulfur hexafluoride is defined as a simple asphyxiant. Also, contact with rapidly expanding gas may cause burns or frostbite.

This gas is solely used for the annual, main maintenance of the TEM. The entire procedure is done uniquely by JEOL engineers. No user of the TEM lab is allowed to interact with the sulfur hexafluoride cylinder.

SECTION 3.3: LIQUID NITROGEN HANDLING

Liquid nitrogen (LN2) is inert, colorless, odorless, non-corrosive, non-flammable, tasteless, extremely cold, and has no warning properties. Special care must be taken by personnel who handle or work in areas where liquid nitrogen is used. The hazards associated with LN2 include:

- Over-pressurization and explosion due to LN2 vaporizing to nitrogen gas (700x expansion ratio) in unvented containers.
- Severe burns caused by exposure to cold temperatures.
- Asphyxiation due to displacement of oxygen in the air in confined work areas.

Humans cannot reliably detect the presence of nitrogen. Liquid nitrogen has a 700x expansion ratio which may create physical hazards and injuries from the explosion of unvented containers. Extensive tissue damage or burns can result from exposure to LN2 or cold nitrogen vapors.

Asphyxiation may result from the displacement of oxygen in the air with nitrogen to levels where there is insufficient oxygen to support life. Inhalation of oxygen deficient air can cause dizziness, nausea, vomiting, loss of consciousness, and death.

Users of the TEM lab are trained on how to properly transfer liquid nitrogen from its main cylinder to a proper cryo-dewar available in the lab. For this transfer, users must always wear

the proper personnel protective gear (PPE) described in Section 2.4. The dewar should never be sealed during any time.

WARNING: Do not seal the cryo-dewar with liquid nitrogen inside! Sealing the liquid nitrogen in the dewar may cause it to explode.

Users should not have to make adjustments to any regulator or to change any cylinders. The TEM users are to follow all equipment operational procedures outlined in the Standard Operating Procedures located in Appendix B, in the equipment logbook and during equipment trainings.

The TEM lab is not considered a confined work area. Proper ventilation is present to avoid accumulation of nitrogen gas. Still, during users training, all hazards related to liquid nitrogen are mentioned and explained.

Users are also instructed to never plug, remove, or tamper with any pressure relief device from the liquid nitrogen tank. Under normal conditions, these cylinders are designed to periodically vent gas.

SECTION 3.3.1: PERSONAL PROTECTIVE EQUIPMENT – LIQUID NITROGEN

The following personal protective equipment is required when handling or using LN2:

- **Water proof thermal insulated gloves** (e.g., cryo-gloves):- Hands must be protected with water proof thermal insulated gloves that can be quickly removed if LN2 is spilled on them. Insulated gloves are not intended for submersing hands into LN2.

- **Cryo-aprons**:- Body must be protected with pants and closed-toe shoes. Thermal insulated aprons should be available.

- **Safety goggles**:- Eyes are most sensitive to the extreme cold of LN2 and its vapors. Over-pressurization may result in the explosion of improperly vented equipment. Chemical splash goggles must be utilized when handling LN2.

- **Full face shield**:- Face must be protected against splashes and spills of liquid nitrogen by a face shield.

SECTION 3.3.2: ACCIDENTAL EXPOSURE

Users are required to wear appropriate personal protective equipment (PPE) for their safety. If at any time a piece of PPE becomes damaged or torn, it should be replaced immediately. If a

user has been exposed to liquid nitrogen, the following procedures should be followed immediately.

For frostbite exposure:

1. Remove all contaminated clothing and gloves, and any clothing item that may restrict circulation to the frozen area.
2. Do not rub frozen parts, as tissue damage may result.
3. Place the affected area in a warm water bath that has a temperature not exceeding 105°F (40°C).
4. Never use dry heat!!

In case of MEDCIAL EMERGENCY contact:

9-911 from any campus phone
or
Campus Security
Phone: **(304)293-3136 (293-COPS)**

If going to the hospital for medical assistance, make sure you:

- Take a copy of the MSDS with you
- Inform medical personnel you were exposed to liquid nitrogen.

SECTION 3.3.3: LIQUID NITROGEN SPILL RESPONSE

Personal protective equipment should be worn at all times. If a significant quantity of liquid nitrogen has escaped, or been spilt, the area affected will not contain adequate oxygen to support life. The area should be immediately evacuated and the following personnel should be contacted:

Environmental Health & Safety
Phone: **(304)293-3792**

Then call,

Marcela Redigolo, Electron Microscopy Facility Manager
Cell Phone: **(304) 680-3007**
Office Phone: **(304) 293-9973**
Room: **G75D ESB**

Or

Kolin Brown, Cleanroom Manager
Cell Phone: **(304) 366-6551**
Office Phone: **(304) 293-9683**
Room: **G75D ESB**

If no one responds to any numbers above, then contact:

Kenny Claudio
Cell Phone: **(304) 216-4858**
Office Phone: **(304) 293-4091**
Room**: 373A MRB**

Royce Watts,
Cell Phone: **(304) 288-6762**
Office Phone: **(304) 293-4124**
Room: **377A MRB**

If a person is showing symptoms of mild or severe asphyxia, they should be moved to an area with fresh air. If they are not conscious, security should be called immediately and resuscitation started by a qualified first aid officer or physician.

Cold burns from liquid nitrogen should be immediately and continually flushed with tepid or unheated tap water.

Professional medical advice should always be sought urgently for significant cold burns and asphyxia. Persons affected may need urgent medical treatment at a hospital.

SECTION 4: VACUUM PUMP OIL

The TEM requires a certain level of vacuum to be kept inside its column and chambers. Oil is used in the instrument's vacuum pumps. Only authorized personnel are allowed to change the vacuum pumps oil. Due to contract, at the present moment, only the engineers of the manufacturer of the microscope, JEOL, are authorized to do such procedure.

The lab has one small container of vacuum pump oil stored inside the lab's floor cabinet. In case of maintenance, all used oil must be captured in an appropriately chosen waste jar, which is properly labeled with the words USED OIL. Once the service is completed, the waste is taken to room G55A1 ESB, which serves as the hazardous waste collection point for the WVU SRF Cleanroom. The label is dated and signed by a WVU SRF staff member.

A hazardous chemical disposal form is filled out and submitted to EH&S for pickup. A copy of the hazardous chemical disposal form can be found on the WVU EH&S website, http://ehs.wvu.edu/forms.

APPENDIX A

STANDARD OPERATING PROCEDURE

TRANSMISSION ELECTRON MICROSOPE

TEM LAB – room B63 ESB

APPENDIX B

CHEMICAL INVENTORY AND MATERIAL SAFETY DATA SHEET (MSDS)

TEM LAB – room B63 ESB