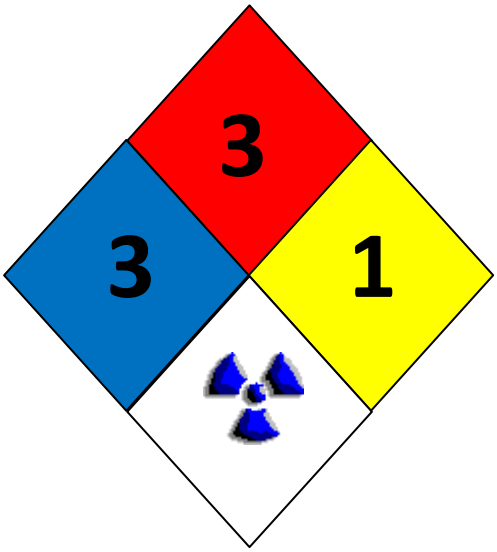
WVU Share Research Facilities

ERB 211 Lab
Chemical Hygiene Plan

211 Engineering Research Building



Emergency Contact:

Weiqiang Ding, Ph.D.
Office: G75D Engineering Sciences Building
Cell Phone: (304) 685-1938

SECTION ONE: EMERGENCY PROCEDURES

1.1 Emergency Contact List

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

9-911 from any campus phone
or **Campus Security** Phone: (304) 293-2677 (293-COPS)

Then Call

Weiqiang Ding, MFCF Manager
Cell Phone: (304) 685-1938 Room: G75D ESB/409 White Hall

or

Harley Hart, MFCF Technician
Cell Phone: (412) 443-1514 Room: G75D ESB/409 White Hall

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

Kenny Claudio, Coordinator of Facilities, Benjamin M. Statler College of Engineering and Mineral Resources
Cell Phone: (304) 216-4858
Office Phone: (304) 293-4091
Room: 373A MRB

Royce Watts, Associate Dean for Administration, Benjamin M. Statler College of Engineering and Mineral Resources
Cell Phone: (304) 288-6762
Office Phone: (304) 293-4124
Room: 377A MRB

For non-emergency assistance please contact:

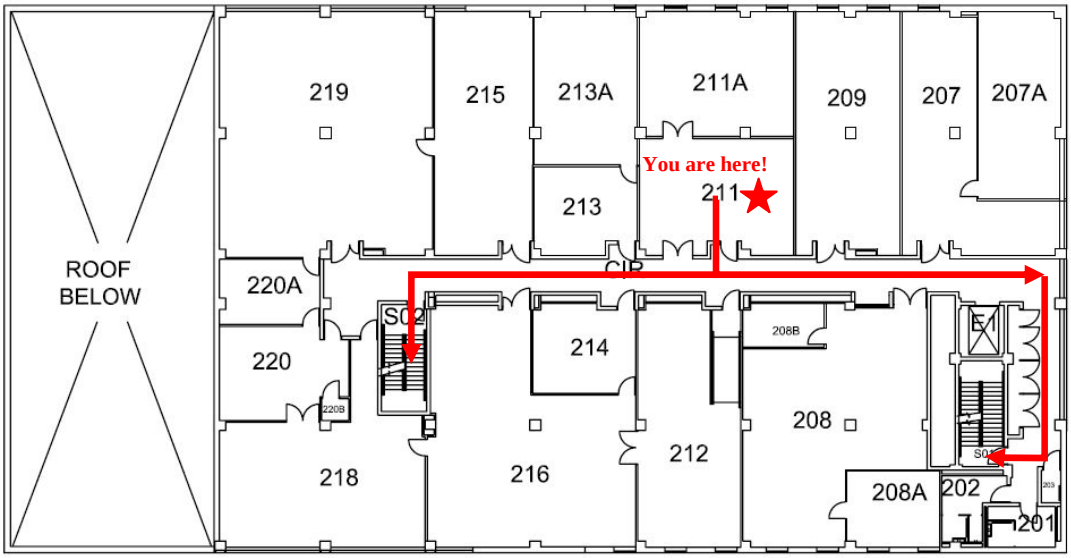
Weiqiang Ding, MFCF Manager
Cell Phone: (304)685-1938 Room: G75D ESB/409 White Hall

or

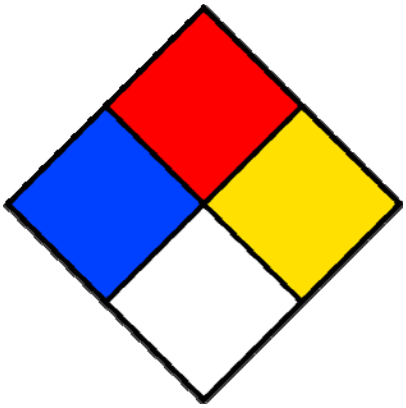
Harley Hart, MFCF Technician
Cell Phone: (412) 443-1514 Room: G75D ESB/409 White Hall

1.2 Evacuation map

On the small scale floor plan of the ERB 211 laboratory, **Fire escape routes are indicated in RED.**



1.3 Safety Diamond



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:

Health (Blue)

4	Danger	May be fatal on short exposure. Specialized protective equipment required
3	Warning	Corrosive or toxic. Avoid skin contact or inhalation
2	Warning	May be harmful if inhaled or absorbed
1	Caution	May be irritating
0		No unusual hazard

Flammability (Red)

4	Danger	Flammable gas or extremely flammable liquid
3	Warning	Flammable liquid flash point below 100° F
2	Caution	Combustible liquid flash point of 100° to 200° F
1		Combustible if heated
0		Not combustible

Reactivity/Instability (Yellow)

4	Danger	Explosive material at room temperature
3	Danger	May be explosive if shocked, heated under confinement or mixed with water
2	Warning	Unstable or may react violently if mixed with water
1	Caution	May react if heated or mixed with water but not violently
0	Stable	Not reactive when mixed with water

Special Symbols (White)

2	Contact	Mild irritant

1.4 Emergency Shutdown Procedure

1.4.1 Bruker XRD Emergency Shutdown Procedure

- If no one is available and the XRD is not acting as expected, the user should do the following:
  - Turn OFF the X-ray
  - Exit the software program
  - Shut down the computer
- If a dangerous situation is evident (smoke, fire, sparks, *etc.*), ONLY if it is safe to do so, the user should press the RED Emergency off button on the control panel located on the side of the XRD instrument to turn OFF power to the entire system and notify the proper emergency personnel. In any case, the user should leave the facility and contact emergency personnel as soon as possible from a safe place.

1.4.2 Digilab FTIR Emergency Shutdown Procedure

- If no one is available and the FTIR is not acting as expected, the user should do the following:
  - Turn OFF the FTS 7000 system power (on the floor under the bench)
  - Turn OFF the UMA 600 microscope power (on the back of the microscope)
  - Exit the *Varian Resolution Pro* program
  - Shut down the computer
- If a dangerous situation is evident (smoke, fire, sparks, *etc.*), ONLY if it is safe to do so, the user should press the green power button on the power strip located on the floor behind the FTIR workbench to turn OFF power to the entire FTIR system and notify the proper emergency personnel. In any case, the user should leave the facility and contact emergency personnel as soon as possible from a safe place.

1.4.3 J.A. Woollam Ellipsometer Emergency Shutdown Procedure

- If no one is available and the ellipsometer is not acting as expected, the user should do the following:
  - Turn OFF the lamp power
  - Shut OFF the EC-400 control box power
  - Exit the WVASE32 program

- Shut down the computer

- If a dangerous situation is evident (smoke, fire, sparks, *etc.*), ONLY if it is safe to do so, the user should press the RED power button on the power strip located in between the ellipsometer and the control boxes on the workbench or unplug the power strip to turn OFF power to the entire system and notify the proper emergency personnel. In any case, the user should leave the facility and contact emergency personnel as soon as possible from a safe place.

SECTION TWO: LAB OVERVIEW

2.1 Description of Lab

- **Purpose of the Lab**

  The purpose of the ERB 211 lab is for performing material characterization with X-ray diffraction technique, Fourier transform infrared spectroscopy technique and ellipsometry technique.

- **Lab Users**

  This lab will be used by the trained users and the staff of the Materials Fabrication and Characterization Facility.

2.2 Facility Description

- **List of Equipments and Descriptions**

1. Bruker Discovery D-8 X-ray Diffractometer

Purpose of the equipment: For determining the crystal structure of solids, powders and thin films; Reciprocal space mapping of crystalline materials and 2D mapping of wafers

Sample Type:

Powders, thin films, or bulk materials

X-ray Source:

Cu-kα1 at 8047.2 eV

Power:

Maximum X-Ray Power of 50 kV and 50 mA and 1 mm slit size

Specification:

360^{o} angular range with a maximum user range of $-100^{o} < 2\theta < 168^{o}$

Accessories:

1 inch hot stage from ambient to $900^{o}C$; 1 inch sample clip stage; 5 inch vacuum stage

2. Digital Lab FTS7000 FTIR and UMA600 Microscope System

Purpose of the equipment: For analyzing chemical composition of micro/macro samples and qualitatively identify molecules and functional groups

Sample Type:

Solid, thin film, powder, liquid

IR Sources:

Tungsten-Halogen Near-IR Source	25,000 – 2,100 cm^{-1}
Mid-IR Duraglow Source	7,900 – 375 cm^{-1}

Beam Splitters:

Near-IR Quartz	20,000 – 2,800 cm^{-1}
Potassium Bromide (KBr)	7,500 – 375 cm^{-1}
Mylar 6.25 μm	470 – 50 cm^{-1}

IR Detectors:

Linearized MCT (Mercury Cadmium Telluride)	10,000 – 450 cm^{-1}	
DTGS (Deuterated Tri-Glycine Sulfate)	10,000 – 150 cm^{-1}	
FPA (Focal Plane Array)	4,000 – 900 cm^{-1}	128×128 pixel 350×350 mm sample area

Accessories:

Variable angle, ATR, Liquid cell, Powder press, photo-acoustic bench accessories

3. J.A. Woollam M-2000 White Light Ellipsometer

Purpose of the equipment: For characterize the film thickness with Angstrom accuracy and determine the optical constant of materials; Stage mapping of thin film uniformity; Multiple layer and surface roughness modeling

Sample Type:

Solid, thin film

Sample Size:

Up to 300 mm in diameter and 20 mm in thickness

Spectrum Range:

245 to 1000 nm (470 wavelengths)

Angle of Incidence:

44^{o} to 90^{o} angle of incidence

Accessory:

Focusing lens attachment

2.3 Assumptions

- **Lab Access**

  Lab access is controlled through the WVU ONITY Lock system at the door.

  As part of the CEMR Electronic Lock policy, the ERB-211 lock has been identified as restricted locks. Access to these locks is only given to lock shop personnel, campus security, emergency services, appropriate CEMR administration, Shared Research Facilities staff and trained 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 these locks:

  Kolin S. Brown
  Lisa Sharpe
  Harley Hart
  Royce Watts

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

- **Restrictions of Access**

  General User: Monday – Friday 8:00 am – 5:00 pm
  Experienced User: unlimited
  SRF Staff: unlimited

Temporary Users: temporary users must be accompanied by a trained user or a staff member at all the time. Temporary users may include summer research participants, visitors or class participants.

MFCF users reserve the time on the instruments via online reservation system (FOM). http://fom.wvu.edu/fom

- **User Agreements**

  Users must sign a user agreement each year in order to gain access to the facility.

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

- **Training Requirements**

  - Users must take general lab safety training, chemical safety training before working in the lab.
  - Copies of the safety presentation slides are located on the Shared Research Facilities website: http://sharedresearchfacilities.wvu.edu/safety/srfSafetyMain.html
  - Users will take instrument operation trainings and then be observed using the tools before granted day-time access to the lab.
  - Experienced users will receive additional trainings and be evaluated before granted unlimited access to the lab.
  - The XRD users must take radiation safety trainings before being granted access to the instrument.

- **Responsibilities**

  The Materials Fabrication and Characterization Facility staff members are responsible for the daily operation and maintenance of the instruments. The building custodians serve the area for trash removal.

- **Dress Code**

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

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

- **Description of Ordering Policies and Procedures for Hazardous Chemicals Used in the Lab**
  - All hazardous chemical orders require prior facility staff's approval before the chemical can be brought into the lab.
  - Use of the chemicals is restricted to users approved by the facility staff.
  - Users cannot store chemicals in the lab.

- **References Available for this Laboratory**
  - Material Safety Data Sheets for chemicals used in the ERB 211 lab are in the Chemical Hygiene Plan, Appendix B.
  - Equipment manuals for the Bruker XRD are stored in the cabinets next to the XRD workbench.
  - Equipment manual for the Digilab FTIR instrument is stored on the workbench of the FTIR instrument.
  - Equipment manual for the J. A. Woollam White Light Ellipsometer is store on the workbench of the Ellipsometer.
  - The Standard Operation Procedures (SOPs) for the FTIR and Ellipsometer are located on the equipment benches, and in the Chemical Hygiene Plan Appendix C.
  - Emergency shutdown procedures are located in the Chemical Hygiene Plan and the SOPs.
  - Instrument usage log books are located on the workbenches in the ERB 211 lab and must be filled out by users for each experiment.

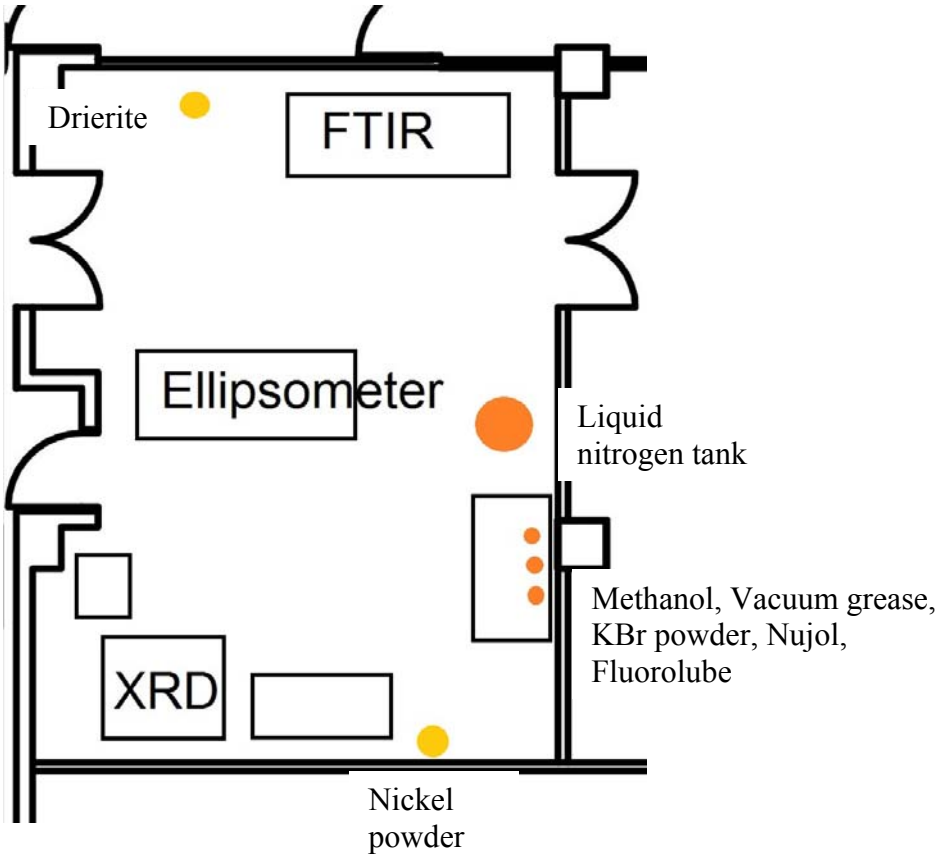
SECTION THREE: CHEMICAL SAFETY

3.1Chemicals and Storage

- **List of All Chemicals**

  Methanol (500 ml squeeze bottle)
  Liquid nitrogen (160L cylinder, 22 psi)
  Nickel powder (500 g)
  Vacuum grease (150 g)
  KBr powder (100 g)
  Nujol (1oz)
  Fluorolube (1 oz)
  Drierite (Anhydrous Calcium Sulfate) (5 lb)

- **Chemical Storage Locations**



3.2 Assumptions

- **Liquid Nitrogen Handling**

1) Wear thermal insulated gloves, insulated apron and face shield whenever handling liquid nitrogen.
2) Must only be done in approved containers with lids (*e.g.*, dewars). Never dispense liquid nitrogen into an unapproved container!
3) Slowly fill the dewar.
4) Lids must be on all the time while transporting the liquid nitrogen transfer dewar.
5) Store the dewar in an upright position. Do not drop, tip, or roll container on its side.
6) Do not try to move the liquid nitrogen cylinder.
7) Never plug, remove, or tamper with any pressure relief device. Under normal conditions, these containers are designed to periodically vent gas.
8) Never adjust, block, plug or attempt to remove or repair anything on a liquid nitrogen cylinder.

- **Accidental Liquid Nitrogen Spill Response**

  - If there is a large spill or rapture of a container, call 9-911, warn others in the building.
  - Notify all people in the lab and evacuate the room! There may be oxygen deficiency in the area of the spill.
  - If there is injury to the body from liquid nitrogen, seek immediate medical assistance.

- **Accidental Liquid Nitrogen Exposure**

  - Users are required to wear appropriate personal protective equipment (PPE) for their safety. If at any piece of PPE becomes damaged or torn, it should be replaced immediately.
  - If liquid is splashed in the eyes, flush with water for at least 15 minutes, Seek immediate medical attention. Call 9-911.

- Skin contact may cause frostbite and burns. Soak affected part in tepid water and seek immediate medical attention. Call 9-911.
- Skin contact is a medical emergency. Lack of prompt medical attention may result in amputation. Call 9-911.

- **List of Personal Protection Equipments available to all personnel in this lab**

Items	Location
Safety Goggles	Sample preparation bench
Face Shield	Sample preparation bench
Insulated Gloves	Sample preparation bench
Insulated Apron	Sample preparation bench
Nitrile Gloves	Sample preparation bench

- **X-ray Radiation Safety**
  - The Bruker XRD equipment is a closed system. Under normal operation, exposure to scattered radiation from the Bruker XRD is extremely low.
  - No personnel dosimetry is required for routine operations. Personally dosimetry is required for persons performing maintenance on the XRD unit.
  - Interlocks are put in place to prevent access and exposure to the primary beam. Bypassing or manipulating the interlocks presents the potential for dangerous exposure.
  - Users should not operate the XRD instrument in a manner other than that specified in the standard operation procedure.
  - Users should not bypass or circumvent the safety interlocks.
  - User should always verify that the beam shutter is closed before reaching into primary beam.
  - Women that are pregnant or suspect that they may be pregnant and wish to avoid all lab exposure should contact the *MFCF staff* in order to make arrangements to get data collected by someone else during the course of their pregnancy.
  - If anyone thinks he/she may have been exposed to the X-ray beam, please seek immediate medical attention. Call 9-911.

- **Water Usage**
  - The heat exchanger water reservoirs of the XRD and FTIR instruments should only be filled with distilled water.
  - The distilled water bottles can be found on the floor next to the XRD chiller.

- **Good General Lab Practice**
  - If it is dirty, clean it up
  - Put all items away immediately after use
  - Keep toolbox organized
  - **Absolutely NO food or drink in the lab**

3.3 Chemical Disposal Procedures

Methanol is used to clean up specimen holder after the experiments. Wipes are used to clean up the methanol residual. The contaminated wipes must be disposed as hazardous waste. Used wipe waste container with lid is kept on the sample preparation workbench to collect contaminated wipes.

The content of the waste container is checked weekly by the SRF MFCF staff and taken to room G55A1 ESB (90 day waste collection site in SRF cleanroom) and placed in the sealed drum for used wipe collection. The SRF staff member dates the tracking form on the drum lid when the first wipes are added. When the drum is full, a hazardous chemical disposal form is filled out and submitted to EH&S for pickup. The EH&S staff person will sign and initial the tracking form when the drum is emptied.

SECTION FOUR: OTHER SAFETY ISSUES AND PROCEDURES

4.1 Definition of Hazard

Users should be aware of additional hazards when working in ERB 211, these are:

- Radiation
- Sharps and broken glass
- High voltages
- Laser

4.2 Assumptions

User should not try to open the control racks of the XRD instrument (high voltage hazards) or the main compartment of the FTIR (laser hazards).

4.3 Safety Procedures

- **Radiation Hazards**
  The XRD instrument generates X-ray radiation while in operation. X-ray is particularly dangerous because it cannot be sensed by a human. The Bruker XRD is a closed X-ray system that generates extremely low scattered radiation under normal operation. Users should not operate the XRD instrument in a manner other than that specified in the standard operation procedure. Users should not bypass or circumvent the safety interlock. User should always verify that the beam shutter is closed before reaching into primary beam. If anyone thinks he/she may have been exposed to the X-ray beam, please seek immediate medical attention. Call 9-911.

- **Sharps and Broken Glass Disposal**
  A broken glass disposal box is kept in the room by the entrance. This box is for the disposing of broken glass or substrates only. MFCF staff members inspect this box weekly. When full, the box is sealed and disposed with the laboratory trash.

- **High Voltage Hazards**

  The XRD equipment utilizes high electrical voltage. Unintentional grounding of an electron beam can produce high spikes of current. Users are permitted to operate the equipment, but not to service. User must follow all operation procedures outlined in the Standard Operation Procedures located in Appendix B. All equipment issues must be brought to a MFCF staff attention. The tool must be disconnected from the power supply before being serviced.

- **Laser Hazards**

  The FTIR has a laser in the main compartment chamber. Laser can cause burns to skin or corneas of eyes. User must follow all the operation procedures outlined in the Standard Operation Procedure located in Appendix B. Users should not open the main compartment of the FTIR system. Only the MFCF staff and service technician can perform service in the main compartment chamber. The tool must be disconnected from the power supply before being serviced.

4.4 Lab-specific Emergency Procedures

- Instrument shut down procedures are to be performed only by the MFCF staff and experienced users who are qualified to do so by the MFCF staff.
- In case of compressed gas emergency, exit the building and notify safety personnel immediately.
- In case of liquid nitrogen spill emergency, exit the building and notify safety personnel immediately.
- In case of fire, evaluate the building and call 9-911.
- In case of unplanned power outage, the instruments will shutdown automatically. See power loss SOP and contact a MFCF staff immediately.
- In case of unplanned water outage, the instruments will shutdown automatically.
- If the emergency button is pressed, refer to the Power Loss SOP and contact a MFCF staff immediately.
- For other lab equipments:
  - Shut off all surge protectors to the computer.
  - Shut power off to all in-use equipments.

4.5 Additional Items

- **Visitors are not permitted in the lab unless authorized by the MFCF staff.**
- Replace wipes and gloves when their supplies are running low.
- Clean nitrile gloves are to be worn at all times when handling samples.
- If you use up any item (*e.g.*, gloves, wipes, sticky tabs, *etc*), notify MFCF staff.

APPENDIX A:
CURRENT CHEMICAL AND GAS INVENTORY

- **Chemicals:**

  Iso-Propanol (500 ml squeeze bottle)

  Methanol (500 ml squeeze bottle)

  Dow Corning Vacuum grease (50 g)

  Nickel powder (500 g)

  KBr powder (100 g)

  Nujol (1 oz)

  Fluorolube (1 oz)

  Drierite (Anhydrous Calcium Sulfate) (5 lb)

- **Gas:**

  Liquid nitrogen (160 L cylinder, 22 psi)

APPENDIX B:
MATERIALS SAFETY DATA SHEET (MSDS)

- Methanol
- Iso-Propanol
- Vacuum grease
- Nickel powder
- KBr powder
- Nujol
- Fluorolube
- Liquid nitrogen
- Drierite (Anhydrous Calcium Sulfate)

APPENDIX C:
EQUIPMENT STANDARD OPERATION PROCEDURES

APPENDIX D:

BENJAMIN M. STATLER
COLLEGE OF ENGINEERING AND MINERAL RESOURCES (CEMR)

CEMICAL HYGIENE PLAN