1. Introduction and Acknowledgements

Safety should always be the first priority when working in the laboratory. This manual highlights the issues specific to the Department of Materials and Metallurgical Engineering at New Mexico Institute of Mining and Technology (NMT). It is a supplement to the general NMT safety manual and will be periodically updated as recommended safety procedures, equipment, and facilities change.

This manual is not comprehensive because no one can foresee all possible outcomes. However, there are many hazards that can be avoided through use of proper precautions as detailed in this manual. Safety is everyone's responsibility. When in doubt, ask for further clarification or instruction. Keep in mind that some of the equipment here has the possibility of causing permanent injury or even death. A moment's inattention can lead to tragedy. We take safety seriously. We expect students, faculty, staff, and all researchers do likewise.

Knowledge is one of the most important factors for safe laboratory work. Protective clothing and safety shielding are not always adequate. They must be supplemented by intelligent use and awareness of the specific situation. For example, the results of many chemical reactions can be anticipated through thermodynamic calculation or consultation with faculty members or experienced students. Electrical safety depends upon knowledgeable use of multimeters and circuit diagrams. In addition to avoiding immediate accidents, precautions must be taken so that the laboratory environment is not booby-trapped for future users. Sloppy wiring or mislabeled chemicals are disasters awaiting unsuspecting fellow researchers.

"One of the most important basic aspects of safety in research is good laboratory housekeeping. This includes the proper storage and handling of chemicals, gas cylinders, electrical equipment and so on. The appearance and organization of our facilities directly affects their safety and productivity as well as departmental reputation. There are two golden rules in developing a safe and productive environment:

1) Whenever you use a lab, it is your responsibility to see that unsafe conditions are corrected immediately;

2) Always leave a laboratory in better condition than you found it.

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If everyone takes this level of personal responsibility, our facilities can only improve."¹

The text of this manual borrows extensively from the safety manuals developed by the departments of Materials Science and Engineering at both Ohio State and Georgia Tech. Sections of this manual were adapted from information available from web sites of other universities and government agencies as indicated. The faculty, staff, and students of the NMT Materials and Metallurgical Engineering Department contributed to the production of this manual and will update information periodically.

2. Five Safety Principles

"Safety in the laboratory requires the same kind of continuing attention and effort that is given to research and teaching. The use of new and/or different techniques, chemicals, and equipment requires careful preparation and planning. Reading, instruction, and supervision may be required, possibly in consultation with other people who have special knowledge or experience. Each individual who works in a laboratory has a responsibility to learn the health and safety hazards associated with the materials to be used or produced, and with the equipment to be employed. It is expected that each person working in departmental labs will apply these principles and be familiar with the information presented in this manual. More specific information and training will be provided on an individual basis by faculty and staff for projects and use of specialized instruments."¹

There are five basic principles for safe laboratory practice: 1. Practice Safety, 2. Concern for the Safety of Others, 3. Understand the Unique Hazards of Your Experiments, 4. Know What to Do in an Emergency, and 5. Report Hazards. Each of these principles is essential and provides guidance for working in a safe manner. These principles have been copied from the Ohio State University Materials Engineering Safety Manual and modified to apply to NMT.¹

(1) Practice Safety

One problem concerning the practice of safety is that it is a subjective matter. For example, some people consider smoking safe while others do not. To have an effective

¹ http://www.matsceng.ohio-state.edu/department/safety/manual/ accessed Nov. 5, 2009

safety program, some common ground rules must be established. This is the main purpose of this Safety Manual. Some of the more basic safety practices that you are expected to follow are:

a) Wear appropriate eye protection whenever working with any potential eye hazards (safety glasses, chemical goggles and face shields)

b) Use a hood for hazardous, volatile, and noxious chemicals.

c) Label an experiment to show its associated dangers and the persons to contact in case of a problem. There should also be an up-to-date notice posted visibly outside each room listing the responsible persons to call in the event of problems in the room.

d) All gas cylinders must be secured, all containers labeled with information about contents, to observe posted signs, such as no smoking, and so on.

It does not end here, because the list is actually endless. Each situation requires its own safety practices, which you are expected to know or find out before doing an experiment.

(2) <u>Be Concerned About The Safety of Others</u>

Your concern for safety must include the people around you. Your experiment must be safely maintained so that everyone in the area is amply protected and warned of inherent dangers. In addition, this principle of looking out for the other person should include the practice of pointing out unsafe procedures to those people committing the unsafe act. This practice could involve something as simple as reminding a friend to wear safety glasses. Another aspect of this second principle involves alerting those around you of an accident. It is your responsibility to alert personnel in the immediate vicinity of a fire or an emergency!

(3) Understand the Hazards Associated with Your Particular Experiment

Prevention is the key to safety. Prior to designing any experiment, using a new piece of equipment, or handling chemicals in the laboratory, it is wise to consider the potential hazards and safety precautions involved in the work. Hazards may include toxic substances, electrical circuits, mechanical equipment, and waste chemicals. Safety precautions should include correct materials storage, proper ventilation, proper grounding of equipment, and training sessions when necessary. Whenever possible, information

about the unique hazards and precautions necessary for any type work should be prepared and made available to everyone working in the lab. Material Safety Data Sheets (MSDS) and equipment manuals are important sources of information. Prior to starting any experiments, an MSDS which includes toxicological information and special handling requirements should be obtained and read for each chemical to be used. MSDS records for all chemicals in the MATE Department are on file in 159 Old Jones, in individual laboratories usually near the entrance, and online resources, e.g., websites of chemical suppliers, national laboratories, and other universities.

(4) Know What to Do in an Emergency

You must be prepared to respond quickly and precisely to an emergency. You must familiarize yourself with the laboratory you are working in, its exits, and its associated safety equipment: eyewash stations, showers, sinks, fire blankets, fire extinguishers, and spill kits. Just a few moments spent learning the locations and use of these pieces of equipment prior to an emergency could save a life. Additional information is provided in this manual.

If the emergency is of an infiltrating nature, such as a fire, gas leak, release of toxic fumes, or radiation leak, the following procedures should be followed:

Alert personnel in the immediate vicinity. Confine the fire or emergency, if possible. Summon aid by calling Campus Police X5555 or Dial 911 Evacuate the building. Report pertinent information to responding emergency personnel.

(5) <u>Report Hazards or Hazardous Conditions</u>

You must report any incidents without delay. Report hazards and hazardous conditions to your supervisor, department chairman, and/or the Hazardous Waste and Safety Officer X5842.

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3. Emergencies

For medical emergency and/or police, call Campus Police, X5555 or 9-911. For fire, pull nearest alarm and call the campus police at X5555. Dialing 9-911 will circumvent the university police, but ambulance response will be slower. In the case of a clear emergency, instruct Campus Police to call the ambulance immediately, rather than waiting for an officer to arrive and assess the situation. Provide adequate information on the phone: name, telephone number, building, room number, condition of any injured individuals (e.g., unconscious, burned, trapped), type of fire, if any. Do not move seriously injured people unless they are in danger of further injury. This section was adapted from the Ohio State Materials Engineering Dept. Safety Manual.¹

A. <u>Medical Emergencies</u>

Immediately provide the minimum necessary first aid to prevent further injury to the victim.

If the injury requires more than a band aid (use this as a general rule of thumb), call Campus Police X5555 and request assistance. Medical help will be sent to you immediately. Be prepared to describe accurately the nature of the accident and your location.

Provide first aid within the scope of your training while waiting for professional help to arrive. It is important that you do not attempt any medical treatments with which you are unfamiliar.

However, there are certain serious injuries in which time is so important that treatment must be started immediately. The proper aid is outlined below according to the type of injury. Report all injuries to your supervisor/advisor after professional help arrives.

If serious medical attention is required, you are expected to call for help by calling Campus Police X5555. Non-emergency victims should go to the Student Health Center.

B. <u>Stoppage of Breathing</u>

For stoppage of breathing (e.g. from electrical shock or asphyxiation), the mouthto-mouth method of resuscitation is far superior to any other known. If the victim is found unconscious on the floor and not breathing, rescue breathing must be started at once, seconds count. Do not waste time looking around for help, yell for help while resuscitating victim.

Training in the techniques of mouth-to-mouth resuscitation and Cardio-Pulmonary Resuscitation (CPR) is available on Campus and several staff, students, and faculty within the department are trained.

C. Severe Bleeding

Severe bleeding can almost always be controlled by firm and direct pressure on the wound with a pad or cloth. The cleaner the cloth, the better; however, in an emergency, a piece of clothing will suffice. In addition:

1. Wrap the injured to avoid shock, and call immediately for medical attention.

2. Raise the bleeding part higher than the rest of the body and continue to apply direct pressure.

3. Keep victim lying down.

4. Never use a tourniquet.

5. If bleeding is profuse, call Campus Police X5555 for emergency assistance and/or transport to the hospital

D. <u>Thermal Burns</u>

1. If the burn is minor, apply ice or cold water.

2. In case of a clothing fire:

a. The victim should drop to the floor and roll. Do NOT run to a safety shower. A fire blanket, if nearby, should be used to smother the flames.

b. After flames are extinguished, deluge the injured under a safety shower, removing any clothing contaminated with chemicals.

c. Keep the water running on the burn for several minutes to remove heat and wash area.

d. Place clean, soaking wet, ice-packed cloths on burned areas, and wrap to avoid shock and exposure.

e. Never use a fire extinguisher on a person with burning clothing.3. Contact Campus Police X5555 for emergency medical support if required.

E. Chemical Burns

1. For chemical burns or splashes, immediately flush with water.

2. Apply a stream of water while removing any clothing that may have been saturated with the chemical.

3. If the splash is in the eye, flush it gently for at least fifteen minutes with clean water. Wash in a direction away from the other eye. Have aid summoned immediately!

4. If the splash is on the body, flood it with plenty of running water for at least 15 minutes. For large scale exposure have someone call Campus Police X5555.

5. A safety shower, hose, or faucet should be used in an emergency.

6. For chemicals spilled over a large area, quickly remove contaminated clothing while using the safety shower; treat as directed under the section thermal burns. No time should be wasted for modesty. Seconds count.

7. If safety goggles are worn during a chemical exposure to the face, leave them on until the surrounding area is thoroughly rinsed, they may be the only thing keeping the chemical out of your eyes.

F. Traumatic Shock

In cases of traumatic shock, or where the nature of the injury is not clear, keep the victim warm, lying down and quiet. Wait until medical assistance arrives before moving the victim. One should treat all injuries as potential shock situations, as they may turn into one. Some common symptoms of shock are cold and clammy skin, paleness, and deliria.

G. <u>Fire</u>

1. Call Campus Police X5555 and report the location of the fire.

2. Pull the hall fire alarm to evacuate the building. These hall fire alarms sound only within the building, and do not alert fire officials. You must call Campus Police X5555.

3. Confine or control the fire, if possible. If in doubt, immediately leave the area and wait for police/fire personnel to respond.

a. Immediately turn off gas supplies and electrical power sources if possible.

b. Use an appropriate extinguisher:

i. CO₂ extinguisher for flammable liquid (Class B) or electrical (Class C) fires.

ii. Solid chemical (NaHCO₃) extinguisher for paper or wood (Class

A), flammable liquid (Class B), or electrical (Class C) fires.

iii. Yellow extinguisher (MgO sand) for metal (Class D) fires.

c. Use common sense. A fire in a beaker may often be extinguished by covering the beaker and depriving the fire of oxygen. Using a fire extinguisher on the same beaker of burning solvent may cause the solvent to splatter, increasing the hazard.

d. If you are absolutely certain that you have extinguished the fire, call Campus Police X5555 and report that the fire is out. Campus Police will still come to assess the damage and to complete a report if necessary.

4. You are expected to use good judgment. Obviously, it may not be necessary to evacuate the building for a small fire in the lab. However, if there is any chance that the fire may endanger others or may cause serious damage, do not hesitate to pull the alarm. Never feel embarrassed about being over-cautious.

5. Immediately after a fire extinguisher has been used, contact Facilities Maintenance to request that the fire extinguisher be exchanged for a new or recharged one.

H. Building Evacuation

As with any other public building, the Fire Code requires that a plan for the evacuation of our building complex be established. Your cooperation with the directives of the evacuation officers and fire wardens is mandatory. The procedure to be followed is as follows:

1. A continuous ringing of the fire bells located in the corridors means everyone is to leave the building.

2. Shut down and secure any laboratory equipment that is in operation.

3. Leave your laboratory. Close but do not lock the door and proceed to the nearest exit.

4. Go to the Emergency Evacuation Assembly point across the street near the Macey Center Sign or the Jones Annex Patio whichever is closer.

5. Permission to re-enter the building will be signaled by Campus Police.

6. Do not loiter in the Jones Hall parking lots as it must be kept clear for access by emergency vehicles.

<u>Physically Disabled Persons</u> have the initial responsibility to request assistance. It is suggested that instructors determine, in advance, if any students require assistance during an emergency. If assistance is requested, the instructor should so advise the class without making any specific individual arrangements. Should the evacuation alarm sound, the instructor should request assistance to move the handicapped person to the nearest exit. Unless specifically requested and considered advisable by those providing the assistance, the movement of a disabled person down a stairway is not recommended. One individual should remain with the handicapped person, if this can be done without unreasonable personal risk, while the others evacuate the building and advise the firemen of the location of the handicapped person so that the evacuation may be completed by the firemen.

Classroom instructors are expected to interrupt class activity and advise students to evacuate the building. Students are obliged to follow emergency procedures.

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In the event of an Earthquake, it is recommended that in the event of one, you should take cover under a sturdy object (like a heavy desk). The idea is to protect yourself from falling debris if the building collapses.

I. <u>Use of Emergency Equipment</u>

Everyone working in NMT labs must know how to use emergency equipment such as fire extinguishers, spill kits, safety showers, and eye wash apparatus. Special training on the proper use of all types of emergency equipment is available by contacting Curtis Verploegh, Safety officer. Know where these items are located in your laboratories. If floor plans are hanging in the hallways of Old Jones.

SAFETY IS THE RESPONSIBILITY OF EVERYONE.

4. Housekeeping

Neatness is required for all quality research. Accept this now or learn through hard experience. It is also a necessary safety precaution. Contamination of food, drink, and smoking materials are potential routes of exposure to hazardous materials. For this reason, food or beverage should not be taken into any laboratory where there is a toxicity hazard. Glassware or utensils that have been used for laboratory operations should never be utilized to prepare or consume food. Laboratory refrigerators and cold rooms should not be used for the storage of foods; separate, clearly labeled equipment should be employed. Smoking is prohibited in our building. It is good practice to wash hands often, even when gloves are being utilized. Avoid the use of solvents for washing; they remove the natural protective oils from the skin and can cause irritation and inflammation. In some cases, the solvents might even aid skin absorption of a toxic chemical.

Work areas need to be kept clean and free from obstructions. Cleanups should follow the completion of any operation or be done at the end of the day. Do not leave a disaster waiting for the next user. Aisles, hallways and stairways must not be used for storage areas. Avoid storing heavy objects on high places from which they could fall. Do not store bottles or equipment on shelves on laboratory benches unless there are restraining lips on the shelves. Storage of bottles on benches is undesirable because of their propensity to be knocked over. Extended storage in hoods is also inadvisable because this practice interferes with the airflow in the hood, clutters up the working space, and increases the amount of material that could become involved in a fire.

All reagents stored in other than their original containers must be labeled clearly as to the contents, date and name of the person storing the solutions. Do not label as "Joe's solution" since this is a significant impediment to waste disposal at a later date. Chemicals stored in the laboratory should be inventoried periodically, and unneeded items should be disposed of. Containers should also be examined for deteriorating labels. The quantity of chemicals stored in the laboratory should be kept as low as possible. Old or outdated solutions should be disposed of as described in the section on Chemical Safety - Disposal. Wastes need to be placed in appropriate receptacles that are properly labeled. Broken glassware, pipettes, and syringes first need to be placed into puncture proof containers.

The individuals along with their faculty advisor should arrange for the removal or safe storage of all hazardous materials which personnel have on hand when they are about to terminate, graduate, or transfer.

5. General Laboratory Practice

1. Working alone is not good laboratory practice.¹ An individual is advised to work only under conditions in which appropriate emergency aid is available when needed. In other words, try and work when others are around to provide help if it is needed. If others are working nearby, let them know where you will be working so that they can occasionally check on you and you can check on them.

2. Eye Protection¹ - In all laboratories where chemicals are used there is the hazard of splashes or dust particles entering the eyes. Pressurized or vacuum vessels may explode or implode sending shrapnel through the lab. While working with electrical wiring there are hazards from molten solder and debris. When testing samples on Instrons or other equipment, pieces can chip and enter the eye. All of these activities, and many

others, require the use of either safety glasses, chemical goggles or face shields. Most lab operations simply require the use of safety glasses. However, when any chemicals are being used at least chemical goggles should be used or in some cases a face shield is required. The appropriate eye protection is generally specified on the Materials Safety Data Sheets (MSDS) found with the chemical, in the lab room, or on file in 159 Old Jones

3. Ear Protection¹ - The healthy ear can detect sounds ranging from 15 to 20,000 hertz. Temporary exposure to high noise levels will produce a temporary hearing loss. Long term exposure to high noise levels produces permanent hearing loss. There appears to be no hearing hazard (although possible psychological effects) to noise exposure below 80 dB. Exposure above 130 dB is hazardous and should be avoided. Ear muffs offer the highest noise attenuation, and are preferred for levels above 95 dB. Ear plugs are more comfortable and are preferred in the 80-95 dB range. If you suspect that a hearing hazard exists then notify the Hazardous Material and Safety Office to have the sound level measured.

4. Respiratory Protection¹ – There should be no need to use respirators as adequate engineering controls should be in place. However, if the need arises to use a respirator, use only respirators provided and/or recommended by faculty or the Hazardous Material and Safety Office. There are many shapes and sizes of respirators and in order to be effective it must be properly fitted. There are also a variety of cartridges available each having a specific application. Respirators should only be used following proper fitting and instruction by trained personnel.

5. Clothing¹ - In situations where splashing or spills may occur it is wise to protect your body with lab coats. Goggles and face shields, splash aprons, and gloves may be needed for chemicals that are corrosive or easily absorb through the skin. Shorts and open-toed shoes are not recommended when working in the lab. Do not work in a laboratory with loose hair or clothing.

6. Protective Clothing and Equipment

Eye protection must be worn at all times in all laboratories where other than purely instrumental studies are being conducted. Ordinary prescription glasses will not provide adequate protection from injury to the eyes. The minimum acceptable protections are hardened glass or plastic safety glasses. Safety goggles or face shields should be utilized where there is a possibility of splashing chemicals, violent reactions or flying particles. Specific goggles need to be worn for protection against laser hazards, and ultraviolet or other intense light sources. Contact lenses should never be worn in the laboratory.

Skin contact is a potential route of exposure to toxic materials. Dermatitis, erythema, burns and absorption of toxic and/or carcinogenic chemicals are some of the consequences of exposing skin to hazardous liquids. Therefore, proper protective gloves need to be worn when working with toxic or corrosive materials or with materials of unknown toxicity. No one glove is suitable for handling all chemicals. Gloves should be selected on the basis of the material being handled and their suitability for the particular laboratory operation. Glove manufacturer's data (e.g., Ansell, Fisher catalog) and the MSDS sheets for the chemicals provide useful information in this regard. Chemical resistance is the most common type of glove evaluation. It is a qualitative and subjective rating and refers to the ability of the material to resist decomposition or disintegration. It does not indicate the chemical protection afforded by the glove. Chemical permeation, on the other hand, does. Permeation is the process by which a hazardous liquid may pass through the glove material to the inside. Because the glove material is not physically destroyed, the individual may not be aware that breakthrough has occurred and that he/she is being exposed. The period of adequate protection, i.e., the breakthrough time plus some interval of time during chemical permeation can only be determined by testing the glove with the chemical to be handled. Select gloves made of materials that have the longest breakthrough times and the lowest penetration rates. As some people are allergic to latex, it is advisable to wear nitrile or cotton gloves underneath latex. A partial list of recommended gloves for handling common chemicals is provided in Table 1.

Clothing including shoes are an important protective barrier. Open-toed shoes or shoes with cut outs offer little or no protection. Wear fully enclosed shoes at all times in the lab. If working with hot materials or a process that may generate hot splatter or sparks, leather shoes or leather shoe covers must be worn as cloth shoes will not provide adequate protection. Do not wear loose, skimpy, or torn clothing. Loose clothing includes items like saris, dangling neckties, baggy pants, over-large lab coats or shirts, dresses and skirts. They may catch on objects or the spills may more easily run under the clothing. Clothing that provides little protection includes items like halter-tops, shorts, skorts, capris, cut offs, tank tops, mini-skirts, and pants with holes or tears. Wear a lab coat or similar protective garment to lessen the contamination of street clothes even if the street clothes are otherwise adequate coverings.

Chemicals	Use	Do not Use
Acetone	Latex	
Carbon Tetrachloride	Nitrile	Latex
Ethyl Alcohol	Latex	Nitrile
Hydrochloric Acid	Any Gloves except	PVA
Hydrofluoric Acid	PVC over any other glove	
Methyl Alcohol	Latex	PVC
n-Hexadecane	Nitrile	Latex
Nitric Acid >70%	Latex	Nitrile, PVA, Rubber, PVC
Nitric Acid <10%	Any Gloves except	PVA
Sulfuric Acid	Latex	PVC
Toluene	Nitrile	Latex

 Table 1. Recommended gloves for handling different chemicals

7. Chemical Safety

In our laboratories, we use a variety of chemicals for electro-polishing, etching and surface treatment and synthesis¹. Some of those chemicals are evaporative, corrosive and/or explosive. For the safety of yourself and others, please deal with them carefully. These general procedures must be adhered to:

1. Never eat or drink in the labs. Do not bring food into labs for storage even if it is temporary.

2. Mark and keep chemicals and etchants in a safe, cool, and stable place. Each container must be marked with chemical, name of person using it and date. Mark bottles that have used chemicals when they are empty.

3. You must handle all chemicals inside a fume hood.

4. Always wear an appropriate mask and safety glasses when working with fine powder, evaporative, corrosive, and explosive chemicals. These chemicals include hydrochloric acid, hydrofluoric acid, n-hexadecane, nitric acid, sulfuric acid, and toluene. The most explosive acid is perchloric acid; you may not handle it unless you have been trained in the procedure for handling perchloric acid.

5. Wear appropriate gloves when working with chemicals. Refer to the MSDS, Table 1, Chemical Suppliers (e.g., Fisher, Cole Palmer) and Glove Manufacturers (e.g.,Ansell), and other safety web sites (Sandia and Los Alamos National Labs) for more complete information. With extremely hazardous materials, check multiple sources as the MSDS may not be up to date!

6. In the event of emergency, call campus police at 5555 or 911. Also contact the professor who is in charge of the particular lab and/or the department chair.

7. Each laboratory/activity should conduct at least an annual survey and dispose of unneeded/expired material. At the end of any project or prior to the departure of an individual, all material should be clearly identified and unneeded/expired material disposed of.

8. Do not pour waste chemicals into the sink. All the chemicals should be separately collected into marked containers. Waste containers need to be compatible with the waste collected, kept closed

More detailed descriptions of chemical safety practices are included on transporting, storage & handling, disposal, and glassware.

Transporting Chemicals

When chemicals are carried by hand, they should be placed in a carrying container or acid carrying bucket to protect against breakage and spillage. When chemicals are transported on a wheeled cart, the cart should be stable under the load and have wheels large enough to negotiate uneven surfaces without tipping or stopping suddenly. Provisions for the safe transport of small quantities of flammable liquids include a) the use of rugged pressure-resistant, non-venting containers, b)storage during

transport in a well-ventilated vehicle, and c) elimination of potential ignition sources. Chemicals should not be carried in open containers in hallways where they may be spilled.

Storage & Handling of Chemicals

Every chemical should have a specific storage space. They should not be stored on counter tops where they can be knocked over or in hoods where they interfere with proper air flow. Flammable liquids should be stored in ventilated storage cabinets. They should not be stored near ignition sources or in areas where accidental contact with strong oxidizing agents is possible. Oxidizing agents include; chromic acid, permanganates, chlorates, perchlorates, and peroxides.

1. All containers must be labeled (including such harmless items as distilled water). The label should contain the proper name of the chemical and, if appropriate, a statement of hazards with the most severe first, precautions, date of purchase or synthesis, and the name of the user.

2. Do not use chemicals from unlabeled containers . The need for adequate labeling extends far beyond the immediate requirements of the individual users, because they may not be present in case of fire or explosion, or when containers are broken or spilled. Also, they may no longer be associated with the laboratory years later when containers have deteriorated or otherwise lost their value. Prior to graduation each person must properly dispose of their waste or unwanted chemicals. All useful chemicals should be reassigned to another person who will assume responsibility. Proper labeling is extremely important as it is impossible to dispose of unlabeled chemicals.

3. Do not pipet by mouth. Never taste or smell any chemical.

4. Clean spills immediately! Small spills may be safely handled by lab personnel familiar with handling precautions for that material. Contact the Hazardous Material Safety Officer for clean up of larger spills. If in doubt of your ability to handle the situation, evacuate the lab, close the door, and call Campus Police X5555 and explain the nature of the emergency.

5. Avoid direct contact with any chemical, what might be considered safe today may eventually be found to be unsafe. The first step in using any chemical should be a review of the Material Safety Data Sheet (MSDS) supplied by the manufacturer. If you don't have an MSDS for a particular chemical, an MSDS for all chemicals used should be on file in the department office. If the MSDS is not available, get a copy online or with the local supplier of the chemical or the manufacturer. Pay specific attention to the potential hazards and safety equipment required for working with the material. Be familiar with the proper emergency procedures recommended for the chemical in case of accidental exposure.

The following are specific examples of potentially hazardous conditions and how to prevent them.

i) Unattended chemical reactions

Take great care in setting up chemical reactions that are to be left unattended for any period of time. Note that unattended operation should be avoided if at all possible. The possible hazards that might arise from failure of a heating mantle (overheating), failure of a water cooling system (hose becoming disconnected or bursting), and failure of an exhaust (if flammable solvents or toxic gases are involved), are obvious points to check before leaving a reaction unattended. Any reaction that is left unattended should be clearly labeled as to the nature of the reaction and its components, the possible hazards (i.e., poisonous vapors), and the name and phone number of the experimenter. A notice describing the nature of the unattended experiment, emergency procedures, and who to contact in case of emergencies should be posted on the outside of the door to the laboratory in which the experiment is being conducted. Before beginning a chemical reaction the experimenter should have an idea of how it will proceed. Thus, ice baths can be ready if it is exothermic, a vent is available if gases are generated, automatic shutdown incorporated in the event of loss of electrical power, cooling water, etc. The experimenter should also notify his/her advisor that the experiment will be running overnight.

ii) Toxic hazards.

Researchers should be aware of the toxic hazards of the materials they are using, and those being used by others in their vicinity. Toxic materials may enter the body through the skin, inhalation, and/or ingestion. Care should be taken to prevent these means of entrance when handling toxic materials. A large number of common substances are acute respiratory hazards and should not be used in a confined area in large amounts. They should be used only in a hood. Some of these include; ammonium hydroxide, carbon monoxide, chlorine, fluorine, hydrochloric acid, hydrogen sulfide, and sulfur dioxide. These may form as by-products of certain reactions. Control of these byproducts should be part of the experimental procedure.

iii) Acids and Bases

Acids and bases are found in most laboratories since there are a variety of applications for them. Three important hazards are associated with acids and bases: chemical burns suffered from spills, inhalation of caustic vapors, and fires or explosions caused by strongly exothermic reactions occurring when strong acids are diluted rapidly. Strong bases may often cause more severe burns than acids as they don't often provide a warning, such as a burning sensation until damage to the skin has already occurred.

1. Always dilute acids by adding them to water and not vice versa.

2. Use dilute acids and bases whenever possible.

3. Keep bottles of strong acids and bases closed when not in use since they can react with moisture in the air to form caustic fumes.

4. If acids or bases are accidentally splashed in the eye or on the skin, flush with water immediately, continue flushing for 15 minutes, and call for help.

5. For any spills of acid or base, refer to the MSDS. A general small acid spill is cleaned up by sprinkling sodium bicarbonate over the area, absorbing with vermiculite or sand, then collecting the remains for proper disposal. Contact the Hazardous Waste and Safety Officer for additional assistance.

iv) Specialized Chemicals: Hydrofluoric Acid and PerchloricAcid

Hydrofluoric Acid

(adapted from http://www.fap.pdx.edu/safety/hydrofluoric_acid/)

Hydrogen fluoride (HF) is a very serious hazard since both its gas and solutions are extremely toxic and it is rapidly absorbed through the skin without immediate warning such as a burning sensation, but causes long term excruciating pain and burns which take a long time to heal. Furthermore, it is a particularly dangerous chemical because it leaches the calcium from the bone underneath the exposure, and can cause an osteoporosis in that portion of bone within a few hours after exposure. In the NMT Materials Engineering Dept., dilute HF (48%) is most often used.

HF must be used inside a functioning fume hood that has been properly calibrated. If using concentrated HF, additional training by supervising faculty is required. To be safe, always use chemical splash goggles preferably with a face shield to provide adequate eye protection. It is recommended that a laboratory coat with chemical splash apron be worn. Only long pants and closed shoes should be worn. Any glove except PVA may be worn when working with dilute HF but a better practice is to wear PVC gloves over top of standard lab gloves for protection against leaks. Gloves that have not been contaminated with HF may be disposed of in common trash. If gloves have been contaminated, they should be removed immediately, hands should be thoroughly washed and checked for contamination.

FIRST AID AND EMERGENCY PROCEDURES for HF

A HF First Aid and Spill kit is kept in Old Jones 175.

Eyewash and Shower Because HF is corrosive and rapidly damages tissue, OSHA requires an eyewash and shower to be nearby and accessible. Each must be tested monthly to ensure it will operate when needed.

Eye exposure: Immediately irrigate eyes at eyewash for at least 15 minutes with copious quantities of water keeping eyelids apart and away from eyeballs. Do not apply calcium gluconate gel to eyes. In all cases of eye exposure seek prompt medical attention.

Skin Exposure: Immediately wash affected area of skin at sink if a small area of hand or forearm has been contaminated or at a drench shower if upper arms, torso, or legs are contaminated. Limit rinsing to 5 minutes so that calcium gluconate gel can be applied to limit the migration of the fluoride ion. Reapply and massage calcium gluconate gel into affected area of skin every 15 minutes. If calcium gluconate gel is not available rinse skin for a minimum of 15 minutes. Remove all contaminated clothing and place in hood or plastic bag. In all cases of skin exposure seek prompt medical attention by going to the emergency room and contact your supervisor and/or department chairman as soon as possible.

Calcium Gluconate Gel: Calcium gluconate gel is a topical antidote for HF skin exposure. Calcium gluconate works by combining with HF to form insoluble calcium fluoride, thus preventing the extraction of calcium from tissues and bones. Calcium gluconate has a limited shelf life and should be stored in a refrigerator if possible and replaced with a fresh supply after its expiration date has passed. Use disposable gloves to apply calcium gluconate gel. Even after applying calcium gluconate, it is essential that a medical evaluation be made.

Ingestion: Drink large amounts of water to dilute. Do not induce vomiting. Several glasses of milk or several ounces of milk of magnesia may be given for their soothing effect. In all cases of ingestion seek prompt medical attention.

Inhalation: Move victim to fresh air. In all cases of overexposure through inhalation seek prompt medical attention.

Safe Work Practices: Do not work alone when you're using HF. Do not eat, smoke, or drink where HF is handled, since the chemical can be swallowed. Wash hands thoroughly after handling HF.

HF Spills: If HF is spilled outside a chemical hood, evacuate the area, close the doors, post the area with a sign to prevent others from entering, and call Campus Police X5555. Small spills of HF inside a chemical fume hood may be cleaned up using lime, soda ash, sodium bicarbonate, or a spill absorbent specified for HF then the waste should be disposed of properly by contacting the Hazardous Waste and Safety Officer X5842 . Organic spill kits that contain Floor-Dri, kitty litter, or sand should not be used because HF reacts with silica to produce silicon tetrafluoride, a toxic gas.

Storage: Store all HF and HF waste in labeled chemically compatible containers (e.g., polyethylene or Teflon). Glass, metal, and ceramic containers are not compatible with HF. HF should never be stored with incompatible chemicals such as ammonia or other alkaline materials. Always place HF on a low protected shelf or other location where it will not be accidentally spilled or knocked over.

Waste: HF waste should be placed in a chemically compatible container with a sealed lid and clearly labeled. Contact the Hazardous Waste and Safety Officer X5842 if you have any questions and for disposal.

<u>Perchloric Acid and Perchlorates</u> : Cold perchloric acid has the properties of a strong acid. When hot it is also a strong oxidizing and dehydrating agent. It becomes unstable with time and will detonate under shock. Perchlorate compounds will often explode from heating, or from contact with flame, by impact, or friction, or spontaneously. Perchloric acid forms explosive compounds with both organic and inorganic chemicals. Because of this, it must be used in a special ventilation hood equipped with water spray and wash down in which no other types of chemical reactions have ever been vented, and which is not lubricated with organic lubricants. It is imperative that no one attempts to store or use perchloric acid or perchlorate compounds without the prior knowledge, instruction, and supervision or approval of your advisor/supervisor. A safety review by the safety officer and selected faculty prior to experimentation is recommended. The information presented is adapted from *http://safety.dri.edu/Hazards/PerchloricAcidGuidelines.pdf*

Perchloric acid is a strong mineral acid commonly used as a laboratory reagent. It is a clear, colorless liquid with no odor. Most perchloric acid is sold as 60%-62% or 70%-72% acid in water.

Hazards: In addition to being a corrosive liquid, while not combustible, under some circumstances perchloric acid may act as an oxidizer and/or present an explosion hazards. Organic materials are especially susceptible to spontaneous combustion if mixed or contacted with perchloric acid. Under some circumstances, perchloric acid vapors form perchlorates in duct work, which are shock sensitive.

Perchloric acid can be a health hazard if inhaled, ingested or splashed on skin or in eyes. To prevent injury, goggles or a face shield over safety glasses, gloves and an apron over lab coat, should be worn when handling perchloric acid. Symptoms of overexposure include irritation and/or burning of the affected area. Inhalation burns are serious and require immediate medical attention. If perchloric acid is ingested, drink approximately 8 oz. of water and seek medical attention. Do not induce vomiting.

Because of its reactivity hazard, perchloric acid digestions of any size must always be performed in a chemical hood. Those that require temperatures above ambient should be conducted in a special perchloric acid hood that is equipped with a wash down system. Hoods used for hot digestions must be labeled "Perchloric Acid Hood Only. Organic Chemicals Prohibited." Solvents must never be used or stored in a designated perchloric acid hood.

Using < 72% Perchloric Acid at Room Temperature

At room temperature, perchloric acid up to concentrations of 72% has properties similar to other strong mineral acids. It is a highly corrosive substance and causes severe burns on contact with the eyes, skin, and mucous membranes. When used under these conditions, perchloric acid reacts as a strong non-oxidizing acid. The following precautions should be taken when using perchloric acid under these conditions:

Substitute with less hazardous chemicals when appropriate. Use dilute solutions (<60%) whenever possible.

Conduct operations involving cold perchloric acid in a properly functioning laboratory (chemical) hood with current certification sticker. If operations are conducted frequently or in large quantities contact EH&S to determine if a specially designed hood dedicated to perchloric acid use is required.

Always use impact-resistant chemical goggles, a face shield, neoprene gloves, and a rubber apron when handling perchloric acid.

When using or storing even dilute perchloric acid solutions avoid contact with strong dehydrating agents (concentrated sulfuric acid, anhydrous phosphorous pentoxide, etc.). These chemicals may concentrate the perchloric acid and make it unstable.

Always transfer perchloric acid over suitable containment in order to catch any spills and afford a ready means of cleanup and disposal.

Perform all operations on chemically resistant surfaces. Avoid contact with cellulose materials such as wood, paper and cotton., which could result in a fire or explosion.

<u>Using Heated <72% Perchloric Acid</u>

When heated to temperatures above 150°C perchloric acid becomes a strong oxidizer and eventually becomes unstable. Concentrated solutions are very dangerous and can react violently with many oxidizable substances, such as paper and wood, and can detonate. Vapors may also contaminate work surfaces or ventilation equipment with perchlorate residues, which may form highly unstable compounds, such as metallic

perchlorates. These compounds may ignite or detonate under certain conditions. The following additional precautions should be followed when heating perchloric acid.

Perchloric acid digestions and other procedures performed at elevated temperatures should be done in a specially designed perchloric acid fume hoods. If procedures involving heated perchloric acid are performed only rarely other accepted methods to capture and contain vapors may be used in place of a perchloric acid hood. If you have been performing perchloric acid digestions in a laboratory hood not designed for perchloric acid use, contact EH&S immediately for an evaluation of perchlorate contamination of the hood.

Lower the fume hood sash as much as possible so that it can function as a physical barrier or use a safety shield to provide splash/splinter protection. Perchloric acid fume hoods should have shatterproof glass.

Never heat perchloric acid in an oil bath or with an open flame. Electric hot plates, electrically or steam-heated sand baths, heating mantles, or steam baths are preferred. Use explosion proof electrical equipment.

Avoid allowing hot perchloric acid to come into contact with any organic materials, including paper or wood, because a fire or explosion can occur. Avoid storing these materials in perchloric acid work hoods. Avoid using greases or hoses that are incompatible with perchloric acid.

iv) Organic Solvents

Many organic solvents possess harmful vapors or pose health hazards because they can be easily absorbed through the skin. Most solvents are quite volatile and the vapors are flammable. Always refer to the MSDS of a solvent before using it to become aware of the hazards, safety precautions, and emergency procedures associated with that specific solvent. Always store them according to the guidelines for storage of flammable liquids. A few examples of the hazards of some common solvents are provided below, but this list is by no means complete.

Acetone Possesses toxic and flammable vapors. Use proper ventilation, safety glasses, and gloves. Store in a flammable liquids storage area.

Methanol Possesses harmful vapors that can cause dizziness, central nervous system depression, and shortness of breath. Severe exposure can lead to coma and eventually death. Less severe exposure can cause blurring of vision, conjunctivitis, headaches, gastrointestinal disturbances, and definite eye lesions. Methanol should be used in a ventilation hood and neoprene gloves should be worn.

Benzene Carcinogenic. Chronic poisoning can occur by inhalation of relatively small amounts over a long time. Can also be absorbed through the skin. Vapors are flammable and it should be stored in a flammable liquids storage area.

Ethers Ethyl ether, isopropyl ether, dioxane, tetrahydrofuran and many other ethers tend to absorb and react with oxygen from the air to form unstable peroxides which may detonate with extreme violence when they become concentrated by evaporation or distillation, when combined with other compounds that give a mixture that can be detonated, or when disturbed by unusual heat, shock or friction (sometimes as little as unscrewing the bottle cap). This class of compounds should be avoided if there is a safer alternative. It is generally recommended that ethers which will form peroxides should be stored in full, airtight, amber glass bottles, preferably in the dark, or in metal containers. Although ethyl ether is frequently stored under refrigeration (explosion proof), there is no evidence that refrigerated storage will prevent formation of peroxides. Furthermore, leaks can result in explosive mixtures even in refrigerators, since the flash point of ethyl ether is -45°C (-49°F).

v) High Energy Oxidizers

Very small amount of strong oxidizers (0.25g) can result in severe explosions and must be handled with the proper protective equipment, such as: protective clothing, leather gloves and face shields. Larger amounts require special procedures involving explosion barriers. Specific procedures are included in the MSDS for the chemical or available by discussion with the Hazardous Materials and Safety Officer.

vi) Powders

All powdered chemicals must be handled carefully per MSDS recommendations. Most ceramic materials are considered inert with the human body however sub-micron particles in the lungs may cause respiratory irritation. Whenever working with fine powders correct respiratory protection is recommended. Cloth/mesh dust masks are not appropriate for work with extremely fine powders. Some powders such as SiO₂, cause lung diseases such as silicosis. BeO and PbO are considered extremely toxic and must be handled with great care. If possible use powders in a hood so as to not contaminate the laboratory. The specific requirements for each powder are generally listed on the MSDS. Some fine powders, notably metals such as Al, are pyrophoric and may explode when dispersed in air thus extra precautions are necessary.

Powders may also irritate the skin thus wearing a lab coat, gloves, long paths, and closed shoes in addition to a proper respirator is required.

vii) Whiskers and Fibers

Because the cancer causing nature of asbestos was discovered, other mineral and ceramic fibers are under suspicion for their health hazards. It is not well known whether this health risk involves a chemical or physical reaction in the body. Fibers and whiskers must be handled with care so that they may not be inhaled or brought into contact with the skin.

viii) Other Materials

Many other compounds have serious hazards associated with them. You should make it a point to learn about the proper handling of the compounds that you use.

Disposal of Chemicals

Do not pour waste chemicals down any drain or in the trash. All the chemicals should be separately collected into marked bottles or cans as appropriate. Waste containers need to be compatible with the waste collected, kept closed unless material is being added, capable of being transported, and appropriately labeled. To the extent feasible, waste should be segregated and not combined. Mixing of different types of waste poses incompatibility dangers and may result in imposition of costly penalties for waste disposal. Containers which have been emptied using normal practices, e.g., pouring, are not considered hazardous except for poisons. These containers should never be reused, and are considered hazardous even when empty. Call NMT Hazardous Waste and Safety Office to dispose of the chemicals.

Contaminated organic solvents such as acetone, alcohol, MEK should never be poured into the sink. These solvents should be put into metal safety cans. If you plan to use a large quantity of organic solvents, you should buy a safety can for your lab.

DO NOT PUT ACIDS IN SOLVENT WASTE CANS. THIS INCLUDES ELECTROPOLISHING ELECTROLYTES AND ETCHANTS.

To dispose of non-organic wastes, put the material in waste collection containers clearly labeled with the word "WASTE" in a conspicuous location and the type of waste being accumulated in the container, e.g., oil, halogenated solvent, hydrochloric acid.

Before the waste is picked-up, the following must be on the label:

1. The name and phone number of an individual who can answer questions concerning the waste.

2. The actual contents of the container. Provide chemical names. Do not use abbreviations, process descriptions, or brand/product names.

3. Contact the Hazardous Material and Safety Office to pick up the waste and dispose of it.

This procedure is to be followed for all inorganic waste including acids and toxic substances. Always label all waste material. It is extremely expensive to identify unknown material. Never mix two chemicals that are to be disposed: they might form explosive or otherwise harmful mixtures.

Follow the guidelines provided in the NMT General Safety Manual.

Glassware and Sharps

- 1. Use only Pyrex or shatterproof glassware.
- 2. Never use cracked or chipped glassware.

3. Insert tubing properly into stoppers (i.e., use lubricants such as a few drops of glycerine and always wear gloves).

4. Broken glass that is contaminated with harmful materials must be disposed of separately.

Broken glass thermometers containing mercury should be treated in the same way as a mercury spill. These should never be thrown in a broken glass container or trash receptacle.

Sharp materials ("sharps") must be placed in special puncture-resistant containers for disposal. Sharps include needles, scalpels, test tubes, pipettes, Petri dishes, and anything that can potentially pierce a plastic bag.

7. General Equipment Safety

1. Before using an instrument or machine, be sure you have been instructed and authorized by the person responsible for the equipment. Become familiar with potential hazards associated with the equipment, emergency shutdown procedures, as well as the operating procedures.

2. Check all electrical connections and mounting bolts before each use.

3. Check that all rotating parts are free to turn, and that there are no mechanical obstructions before starting.

4. Attach an "emergency shutdown note" to any piece of equipment left operating unattended outside normal working hours. This note should contain your phone number and all information that would be required by anyone who might be faced with the need to shut down the equipment.

5. Laboratory equipment is not to be placed in corridors unless it is being picked up by the Property Yard.

Waterlines

All permanent waterlines connecting apparatus to water outlets should be copper tubing with proper metal fittings or high pressure water hoses fitted with proper clamps. The bursting pressure of the hose must safely exceed the highest pressure attainable in the water mains (100psig). It is not recommended to use Tygon or low strength plastic tubing. The tube system must be able to handle >100 psig, even if the water flows at a low pressure and low rate since an obstruction in the hose can subject it to line pressure.

Vacuum Systems

Mechanical vacuum pumps used in laboratories pose common hazards. There are the mechanical hazards associated with any moving parts and the chemical hazards of contaminating the pump oil with volatile substances and subsequently releasing them into the lab. A few guidelines will help in the safe use of these devices. Distillation or concentration operations requiring large concentrations of volatile substances should be performed using a water aspirator. If a vacuum pump is required for lower pressures, the pump must be fitted with a cold trap to condense the volatiles. The output of the pumps should be vented to a hood or alternate exhaust system. The pump oil should also be replaced when it becomes contaminated and disposed of properly as a chemical waste

1. Be certain that your vacuum system has a trap.

2. Use only containers that can withstand evacuation. When possible, tape containers to be evacuated and use a standing shield to guard against implosion.

3. Always close the valve between the vacuum vessel and the pump before shutting off the pump to avoid sucking vacuum oil into the system.

4. All moving belts on mechanical pumps must have a safety cover.

Distillations and Condensers

Superheating and sudden boiling frequently occur when distilling under vacuum. Therefore it is important that the assembly be secure and the heat be distributed evenly (i.e. with a heating mantle or liquid bath). A standing shield should be in place to guard against implosion. An additional thermometer should be inserted near the bottom of the distilling flask to warn of a dangerous exothermic reaction. After finishing a vacuum distillation, cool the system before slowly bleeding in air, since air may induce an explosion in a hot system. Be sure that hoses carrying cooling water are securely attached with hose clamps to prevent accidental floods. Glass joints should be secured with clips available from the stockroom to prevent accidental disconnection or disconnection caused by vapor build up.

Drying Ovens

Electric ovens are often used in laboratories for removing solvents or water from samples and to dry laboratory glassware. These ovens if not properly vented or used in a hood, discharge the volatile substances into the laboratory atmosphere which can accumulate in toxic concentrations. Small amounts of vapor can accumulate inside the oven and mix with the air to form explosive mixtures.

Ovens should not be used to dry any chemical known to possess toxic vapors or that might volatilize and pose an explosion hazard or acute chemical hazard unless special precautions have ovens whose heating elements or temperature controls which may produce sparks are exposed to the interior atmospheres. It is recommended to blow out panels in the rear of a drying oven so that an explosion will not blow the door and contents into the lab. Bimetallic strip or alcohol thermometers rather than mercury thermometers should be used in ovens.

Removal of Organics in Furnaces

When removing binders or other organic substances from ceramic powders prior to sintering, one must observe similar precautions to those discussed for drying ovens. During decomposition, binders breakdown into shorter chain molecules and volatilize from the sample. These decomposition products often contain carbon monoxide as well as other toxic gases. If not properly vented, these gases may pose acute or chronic toxicity hazards to people in the lab and they can also form explosive mixtures when combined with the furnace atmosphere. Prior to burning out any organic material in a furnace one should estimate the chemical composition of possible decomposition products and ensure the heating cycle and furnace atmosphere are properly controlled so that the explosive limits of the by-products are not reached. You must also be trained on use of the furnace and sign the log book so that others know what you are doing in case of an emergency. The CRC Handbook of Chemistry and Physics lists explosion limits for some substances. If in doubt contact the Hazardous Waste and Safety office for additional assistance.

Use of Furnaces

There are a variety of furnaces available in the Materials Engineering Department which can attain temperatures ranging from 500 to 2300°C with unique capabilities. Before using any furnace, be sure that you have been trained on how to operate the furnace and have reviewed safe practice procedures associated with it including required protective equipment. When using any furnace, you are required to sign the log book providing information on what you are heating and how long you plan to use the furnace. It is essential that you sign the log book so that you can be contacted in the event of power failures or emergencies.

8. Gas Cylinders

Compressed gas cylinders are typically charged to 1500-2500 psi. The primary concern with these cylinders is that the valve stems on them are fragile and easily sheared off if the cylinder is handled improperly or not secured during use. With a broken valve stem, the entire tank pressure will be released in a short amount of time through a very small orifice. Under these conditions, the cylinder becomes a rapidly accelerating 300lb rocket, blasting through walls, equipment, and people until it is empty.

All empty cylinders must be stood upright and placed behind chain or wire restraints. Never lay any cylinder on the floor. Use the Facilities Management Web site to have cylinders picked up. Remember, your project is charged a demurrage (~ \$6/cylinder) every month that these cylinders are in your lab.

1. Secure gas cylinders with a strap or chain to a stable object (preferentially a wall or a heavy lab bench), whether or not they are in use. Always leave the cap on when the tank is not being used.

2. Transport gas cylinders with cap on, and use a proper cart. Attempting to rockwalk the cylinder runs the danger of the cylinder slipping and falling.

3. Do not use an open flame near gas cylinders.

4. Never use grease or other lubricants on gauges or connections (This may form explosive mixtures with oxidizing gases).

5. Teflon tape should never be used on regulators for flammable gasses. As the regulator fitting is tightened, the tape can shred and become wedged between the needle

and valve seat for the tank. This may result in a gas leak between the regulator and cylinder fitting which will allow gas to leak into the surrounding area where it represents an explosion hazard.

6. Before using gas in an experiment, be sure there are no leaks in the system. Inspect regulator inlets and cylinder valve outlets for foreign matter; it is essential that the threads aren't damaged so that a tight seal can be maintained.

7. Learn directions for closing and opening valves. (All main valves close clockwise). Before connecting a non-toxic gas cylinder to a system, remove the valve cap and open the valve for an instant to clear the opening of particles or dirt. To turn on a system, open the main cylinder valve completely and open remaining valves successively further from the main cylinder. To shut down a system close the main cylinder first and close remaining valves in the order in which they were opened to avoid storing high pressure in the system.

8. Do not use adaptors to connect regulators. Use only regulators specified for the particular gas. Have all regulators inspected and serviced regularly. Regulators open by turning the handle clockwise, this increases the pressure in the system.

9. Only use regulators, pipes, and fittings specified for the type of gas you will be using. Do not force regulators onto bottles, if they do not fit easily then you are using the wrong regulator. Hydrogen embrittlement may cause hazards such as leaks or ruptures. Contact the supplier of the gas or regulator if advice is needed on the selection of a regulator.

10. Do not locate gas cylinders near heat sources, like furnaces, where they may heat up and explode.

11. Familiarize yourself with the toxic properties and safety hazards of each gas you use. Post any safety information that may pertain to others working in the lab.

12. Store oxygen cylinders and combustible gases separately.

13. All gas cylinders should be labeled to identify their contents. Do not rely on color codes.

14.Cylinders of combustible gases, e.g. CH_4 , H_2 , and O2 should be stored in continuously exhausted areas. Inert gases can act as asphyxiants, so make sure that

adequate ventilation exists when large quantities of N₂, Ar, He and other inert gases are used.

i) Inflammable and Explosive Gases Such as Hydrogen and Methane

Store and use flammable gasses in well-ventilated areas, away from heat or ignition sources.

Wherever possible, copper tubing should be used to transfer gases. Exhaust hydrogen and hydrocarbons should be vented outside into the open air (through copper tubing). Laboratories using these gases must be ventilated with exhaust fans equipped with explosion-proof motors, etc. Rooms containing these gases should have entrance doors marked with signs.

Systems utilizing these gasses must always be kept "tight" and should be examined regularly for leaks.

ii) <u>Oxygen</u>

Care must be taken so that oxygen of high concentration does not come into contact with combustible substances such as oils and cotton. . Do not use an open flame near gas cylinders.

iii) <u>Toxic Gases</u>

The use of toxic gases must be approved by the Hazardous Waste and Safety Officer. and appropriate signs posted in the laboratory.

9. Cryogenics

A cryogenic liquid (Cryogen) includes any liquid with a normal boiling point below -90 °C at atmospheric pressure (101.3 kPa) as defined in NIST 44. Carbon dioxide and nitrous oxide, which have higher boiling points, are sometimes included in this category. Examples of cryogenic liquids are nitrogen (-196 °C, 77 K), oxygen (-183 °C, 90 K), argon (-186 °C, 87 K), hydrogen (-253 °C, 20 K), helium (-269 °C, 4 K), neon (-247 °C, 27 K), krypton (-153 °C, 120 K), xenon (-131 °C, 142 K), carbon dioxide (-78.3 °C, 194 K), nitrous oxide (-89 °C, 184 K) and methane (-162 °C, 111 K).

Eye protection should be worn whenever cryogenic liquids are handled. Where splashing is a possibility, face shields should be used. Appropriate gloves, closed shoes,

and clothing (long pants to protect legs and a long sleeve shirt or lab coat) must be worn. In no case are sandals, flip-flops, or or shorts to be worn. In case of a splash, immediately flood exposed areas and clothing with water. Beware of liquid nitrogen being trapped inside pockets, gloves, cuffs, or folds of clothing as "puddles" of liquid nitrogen can cause serious burns. Avoid wearing watches, rings, bracelets, or other jewelry. Although many gases in the cryogenic range are not toxic (e.g. liquid nitrogen), they are all capable of causing asphyxiation by displacing the oxygen necessary to support life.

It must be emphasized that more injuries arise from burns caused by persons touching objects that are cold than by spills.

Cryogens should be used only in well-ventilated areas. Venting should be provided to avoid quick and violent pressure changes when a cryogenic liquid vaporizes. Handle combustible cryogens such as liquid hydrogen and liquid natural gas (methane) in the same way combustible gases are handled: provide ventilation, keep away from open flames and other ignition sources, prohibit smoking, and vent gases to a safe location. Exposed glass portions of the cryogenic container should be taped to minimize the flying glass hazard if the container should break or implode.

Liquid nitrogen is the most common cryogen used in our department. Additional safety information pertaining to working with liquid nitrogen is given below.

Liquid nitrogen is colorless, odorless, an extremely cold liquid with a boiling point of -196 $^{\circ}$ C (77 K) and a gas under pressure. Liquid nitrogen is a dangerous material due to the possibility of cryogenic burns on contact with the liquid or cold vapors and asphyxiation (suffocation) since cryogenic liquids produce large volumes of gas when they vaporize displacing the oxygen in a confined space. Precautions that must be taken are:

Do not touch frozen objects or the liquid nitrogen. Even though an object does not look cold, it can "burn" you!

Wear goggles whenever pouring or dumping liquid nitrogen.

Wear full length pants without cuffs and long sleeves/lab coat when working with liquid nitrogen. Liquid nitrogen may pool in socks/shoes or pockets/cuffs and cause severe burns.

Use the blue cryogenic gloves to handle any object going into or out of liquid nitrogen and to carry the liquid nitrogen Dewar.

Open the liquid nitrogen valve incrementally so that the flow remains stable.

Do not adjust the DUAL RELIEF DEVICE, which is set to 100 psi. The DUAL RELIEF DEVICE is to vent the expanding gases and prevent an explosion. The bottle will constantly hiss to relieve pressure which is normal.

FIRST AID FOR LIQUID NITROGEN

Inhalation: Persons suffering from lack of oxygen should be moved to fresh air. If victim is not breathing, administer artificial respiration. If breathing is difficult, administer oxygen. Obtain prompt medical attention. Call Campus Police for assistance.

Skin Contact: Remove any clothing that may restrict circulation to frozen area. Do not rub frozen parts as tissue damage may result. As soon as practical, place the affected area in a warm water bath which has a temperature not to exceed 105°F (40°C). Never use dry heat. Call Campus Police X5555 and contact a physician as soon as possible.

Frozen tissue is painless and appears waxy with a possible yellow color. It will become swollen, painful, and prone to infection when thawed. If the frozen part of the body has been thawed, cover the area with dry sterile dressing with a large bulky protective covering, pending medical care. In case of massive exposure, remove clothing while showering with warm water. Call Campus Police X5555 and take to the hospital emergency room.

Eye Contact: For exposure to liquid, immediately warm frostbite area with warm water (not to exceed 105°F).

10. Machine Safety

Typical machine shops have available in working condition drill presses, band saws, milling machines, lathes and others. In the use of all of these devices, eye protection is essential. Appropriate clothing is essential: closed shoes and long pants are needed to protect legs and feet. Beware of any loose personal articles (lab coat, torn clothing, long hair), which may be caught on moving parts of equipment. On a lathe, the rotating positioning wheel can easily catch clothing. Machine shop equipment is completely unforgiving and will draw the body into it, tearing off fingers and limbs. Parts to be drilled or tapped should preferably be clamped down prior to use, or held down firmly. Otherwise the part may be lifted by the drill bit and turned into a propeller blade. A block of sacrificial wood should be used to push parts through the band saw, not the fingers. In all cases, one needs to pay full attention to the equipment and not be distracted during use.

Before using the Materials and Metallurgical Engineering Machine Shop, users must take the departmental training course or demonstrate their ability to the Machine Shop Supervisor.

11. Electrical Safety

High voltage is dangerous, not high current, unless of course the current is running though the human body. For example, if one grabs the two live ends of a SiC heating element which is drawing 20 A at an applied 40V, what determines the current though the grabber's body is the 40 volts and the resistance of the current path through the body. The 20A in the parallel circuit is immaterial.

While electricity is in constant use by the researcher¹, both within and outside the laboratory, significant physical harm or death may result from its misuse. With direct current, a man can detect a "tingling" feeling at 1 mA and the median "let-go" threshold (the current at which he cannot release the conductor) is 76 mA. For 60 Hertz alternating current, the values are 0.4 mA and 16 mA, respectively. Women are more sensitive to the effects of electrical current with approximately 2/3 of the current required to produce the same effect in a man. Higher currents produce respiratory inhibition, then ventricular fibrillation, and ultimately cardiac arrest.

If an electrical hazard is suspected, the device in question should be disconnected immediately and the cause ascertained by a person competent in such matters. Work on electrical devices should be done only after the power has been shut off in such a manner that it cannot be turned on accidentally. Because malfunctioning equipment may contain shorts, merely turning off the equipment is not sufficient to prevent accidents. Equipment should be unplugged before being inspected or the circuit the equipment is wired to deactivated by putting the circuit breaker in the off position or removing the fuse. Equipment wired to a safety switch should be turned off at the safety switch. Internal energy storage devices such as capacitors must be discharged. All electrical wiring and construction must conform to standard safety practice and electrical codes. High voltage equipment must be labeled as such. Switches to turn off all electrical power to the equipment in case of emergency should be prominently labeled.

The following are a list of rules for working with electrical equipment:^{1,2}

1. Assume that all wires are energized with lethal voltages and never assume that a wire is safe to touch even if it is appears insulated. Make sure that a wire is not energized before touching or moving it. Always test before you touch! Every year, workers are injured or killed by circuits they thought were safely turned off. Simply shutting off the power is not enough—hazardous conditions can still exist. Make sure to always TEST BEFORE YOU TOUCH. You may not get a second chance to learn this important lesson. 2. Turn off the power to equipment before inspecting it. Turn off circuit breakers or unplug the equipment. To turn off a safety switch, use your left hand wearing insulating gloves made of leather or heavy cotton or rubber, turn your face away from the box, and pull the handle down. Circuits may discharge violently when being turned on or off and the cover to the junction box may be blown open.

3. Use only tools and equipment with non-conducting handles when working with electrical devices.

4. All current transmitting parts of any electrical devices must be enclosed.

5. When checking an operating circuit, keep one hand either in a pocket or behind your back to avoid making a closed circuit through the body. When debugging malfunctioning equipment, it is good idea to avoid working with two hands if at all possible. If one hand touches a live location while the other hand touches grounded metal the current path will go through the human heart and may cause defibrillation. Shoes are essential because naturally moist feet in contact with the floor or chair make an excellent ground connection

6. Maintain a work space clear of extraneous material such as books, papers, and clothes.

² Sources for information presented in this section include OSHA, Electrial Safety Foundation, and NFPA

7. Never change wiring with circuit plugged into power source.

8. Never plug leads into power source unless they are connected to an established circuit.

9. Avoid contacting circuits with wet hands or wet materials. The fluid in the body acts as an excellent electrical conductor, and the dominant resistance to current flow is the skin. Therefore, avoid working with wet hands, and take precautions against electrical components piercing the skin.

11. Never operate electrical equipment while standing in water.

12. Wet cells should be placed on a piece of non-conducting material.

13. Check circuits for proper grounding with respect to the power source.

14. Do not insert another fuse of larger capacity if an instrument keeps blowing fuses - this is a symptom requiring expert repairs. If a fuse blows, find the cause of the problem before putting in another one.

15. Do not use or store highly flammable solvents near electrical equipment.

16. Multi-strip outlets (cube taps) should not be used in place of permanently installed receptacles. If additional outlets are required have them installed by an electrician.

17. Keep access to electrical panels and disconnect switches clear and unobstructed.

18. Where the possibility of induced voltages or stored electrical energy exists, ground the phase conductors or circuit parts before touching them. Where it could be reasonably anticipated that the conductors or circuit parts being de-energized could contact other exposed energized conductors or circuit parts, apply ground connecting devices rated for the available fault duty.

19. One of the most important safety precautions when building or repairing equipment is to make sure that it is adequately grounded. Electrical work has strict color conventions, the most important of which is that the green wire is ground. This wire should connect at one end to the ground connection from the power supply (e.g. the ground plug on a 110V wall receptacle) and at the other end, intimately to the metal housing. The purpose of this is so that if a live wire makes contact to the chassis, extreme current will pass though the metal, which will immediately blow a fuse or trip the upstream breaker.

Static Electricity and Spark Hazards

Arc Flash Hazards are another danger. According to the Electrical Safety Foundation, "an arc flash is the sudden release of electrical energy through the air when a high voltage gap exists. and there is a breakdown between conductors. An arc flash gives off thermal radiation (heat) and bright, intense light that can cause burns and other injuries. Temperatures have been recorded as high as 35,000°F. Exposure to these extreme temperatures burns the skin directly and ignites the clothing that you are wearing. High–voltage arcs can also produce considerable pressure waves by rapidly heating the air and creating a blast. This pressure burst, or arc blast, can hit a worker with grenade-like force and send metal droplets from melted copper and aluminum electrical components shooting out at speeds up to 700 miles per hour, fast enough for the tiny shrapnel to penetrate your body. An arc flash can occur spontaneously, or can result from inadvertently bridging electrical contacts with a conducting object. Other causes may include dropped tools, the buildup of conductive dust, or corrosion."

Sparks may result in explosions in areas where flammable liquids are being used and therefore proper grounding of equipment and containers is necessary. Some common potential sources of sparks are:

1. The making and breaking of an electrical circuit when the circuit is energized.

- 2. Metal tanks and containers.
- 3. Plastic lab aprons.
- 4. Metal clamps, nipples, or wire used with nonconducting hoses.
- 5. High pressure gas cylinders upon discharge.

12. Radiation Safety for X-ray Diffraction and Spectroscopy

Analytical x-ray machines produce intense beams of ionizing radiation that are used for diffraction and fluorescence studies. The most intense part of a beam is that corresponding to the K emission of the target material and is called characteristic radiation. In addition to the characteristic radiation, a continuous radiation spectrum of low intensity is produced ranging from a very low energy to the maximum kV-peak setting. This is referred to as "bremsstrahlung" or white radiation. When using a diffractometer that is in good working order and being used correctly, the radiation dose to the operator will be zero. However, if something breaks such as safety interlocks or the equipment is not being used property, dangerous exposure can result as is described in the following paragraphs.

X-rays in the range of 15 to 40 keV produced by diffraction machines are readily absorbed in the first 5-10 millimeters of the skin, and do not contribute to a deep dose to the internal organs of the body. However, the eyes, because of the aqueous nature of the tissue, do receive deep dose. Overexposure of lens tissue can lead to the development of lens opacities and cataracts; therefore, safety glasses should be worn when operating x-ray producing equipment (for this application, glass lenses are preferred).

Absorbed doses on the order of 100 rads may produce a reddening of the skin (erythema), which is transitory in nature. Higher doses - 10,000 rads and greater - may produce significant cellular damage resulting in pigment changes and chronic radiation dermatitis. It should be remembered that exposure to erythema doses may not result in immediate skin reddening. The latent (waiting) period may be from several hours to several days.

The equipment operator is responsible for his own safety and the safety of others when using an analytical x-ray machine. The written procedures developed for individual instruments should be sequentially followed. Never put any part of the body in the primary beam. Exposure of any part of the body to the collimated beam for even a few seconds may result in damage to the exposed tissue. Any person lacking knowledge about x-ray equipment should not attempt to make repairs or remedy malfunctions. Always consult the designated representative first. Remember, safety devices and warning systems are not foolproof or fail-safe. A safety device should be used as a back up to minimize the risk of radiation exposure and never as a substitute for proper procedures and good judgment.

<u>Safety Considerations Pertaining to the Operation of the Siemens D500 X-Ray</u> <u>Diffractometer and Kristalloflex 810 X-Ray Generator</u>

There principal safety are two considerations when running the D500 X-Ray diffractometer. The first of these is concern over exposure to ionizing X-Ray radiation. The second is the danger of high-voltage shock from the X-ray generator, tube, or detector. During routine use of this instrument, several separate events would have to go wrong simultaneously for injury to occur through either of these paths. As delivered from the factory, this piece of equipment utilizes 11 separate safety switches to ensure that the operator cannot be exposed to

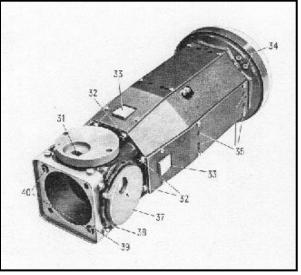


Figure 1 D500 tube stand showing the open shutter indicator as # 32.

energized high voltage leads or an unacceptably high dosage of ionizing radiation.

Preventing exposure to X-Ray radiation from this unit during routine use can be ensured even if the safety circuit fails by making sure that your samples are positioned in the goniometer before the generator is energized, and by making sure that the leaded glass door is never opened while the generator is energized and the shutter is open. When the safety systems are operating correctly, this condition will automatically shut off power to the X-Ray tube, effectively stopping X-Ray production. The status of the window shutter can be easily determined by observing whether the red light at the top of the tube stand is illuminated (Figure 1, # 32 taken from the Siemens D500 Operating Instructions p. 4).

If the light is illuminated, the shutter is open and must be closed before opening the leaded glass door at the front of the instrument. If for some reason the operator is in doubt as to the condition of the shutter (open vs. closed), the close switch for the window shutter (Page 14, Figure 31, # 275 of the Siemens operating manual) should be depressed before attempting to open the leaded glass door.

Under no circumstances should a user attempt to operate this instrument without all of the shielding in place. Likewise, this instrument should never be operated after deliberately disabling one or more of the safety switches present on the body of the instrument. This instrument is safe to operate only when all of the factory shielding is in place, and all 11 safety switches are working properly

Prevention of electrical shock during routine operation of this instrument is as simple as not ever attempting to adjust the cables running into the bottom of the X-Ray tube stand, or the cable, which enters the top of the detector, while the generator or detector circuits are energized. The cables running into the bottom of the tube stand should only be removed by the person responsible for general maintenance of the diffractometer with the generator off and the unit unplugged from the wall. In the event the detector cable becomes fouled around the body of the goniometer, users may GENTLY reposition the cable by manually driving the goniometer to remove cable tension and then GENTLY lifting the cable free of obstruction.

13. General Laser Safety

Lasers present an eye hazard if a person stares into the beam and resists the natural reaction to blink or turn away. Lasers with powers in excess of 500 mW may produce eye or skin damage from diffuse scattered light. Laser warning signs need to be posted for lasers with power in excess of 5 mW. A warning light should be activated when the laser is on. The best practice is to implement engineering controls and fully enclose the laser in a switch protected box or containment system with the operator wearing eye protection as a back up.

Eye protection is required not only from direct impact of the direct beam, but also reflection (diffuse or concentrated) from surfaces. Goggles or safety glasses specifically designed for laser work are needed. Use safety laser goggles or glasses designed specifically for protection from the laser wavelength that you are using. They need to be fitted so that stray light cannot come in from oblique angles. The type of glasses needed depends on the laser type, wavelength, and optical density. Undesirable reflecting surfaces can be rough-finished and painted with flat charcoal black paint.

Direct laser impingement on the skin may cause considerable damage, especially where it is pigmented. A temporary injury to the skin may be painful and treated symptomatically. Injuries to larger areas of the skin are far more serious as they may lead to serious loss of body fluids, toxemia, and systematic infections. Injuries to the skin can result either from thermal injury (temperature elevation in skin tissue) or from a photochemical effect (e.g. "sunburn"). The warmth sensation resulting from absorption of radiation energy normally provides adequate warning for an avoidance reaction to prevent thermal injury of the skin from almost all sources except some high-powered, far infrared lasers.

Potentially toxic vapors, which may result from laser heating of materials, need to be accounted for. Ozone is produced at times from flash lamps and high repetition rate lasers as the beam propagates through air. Ozone is extremely toxic. Proper ventilation is needed when vapors from liquid nitrogen coolants might otherwise starve the room of oxygen.

Laser Classifications

Class IV: >500mW "Danger"

High-power laser with a high risk of injury when the eye or skin is exposed to the direct beam or to diffuse reflections.

Note: Can present fire hazards because of the ability to ignite combustible or flammable materials.

Class IIIb: 5.0-500mW "Danger"

Medium-power laser that can produce eye hazard from exposure to the direct beam.

Note: May produce hazardous diffuse reflections at very close viewing distances. Can also produce minor skin hazard.

Class IIIa:	1.0-5.0mW	"Caution" or "Danger"		
	Prism Coupler	633nm	75mW	Class IIIB
Jones 14	Light Scatterer	532nm	125mW	Class IIIB

Medium-power laser that normally does not produce a hazard if viewed for momentary periods with the unaided eye.

Note: Risk of eye damage increased if viewed for more than 0.25 seconds, or if viewed through collecting optics such as a microscope or telescope.

Class II: 0.4-1.0mW "Caution"

Low power, low risk, visible laser that does not normally present a hazard due to the eye's natural aversion response to bright light.

Note: Directly viewing a Class II laser for extended periods of time can cause eye damage.

Jones 142 AFM 670nm 1mW Class II

Class I: <0.4mW

Laser or laser system that does not emit hazardous laser radiation under normal operating conditions.

Protective measures:

Avoid exposure to direct laser beam!

Protective eyewear should be worn, but it must be designed for specific radiation from laser (this is only secondary protection and not to be used for direct viewing of beams!)

Gloves and lab coats may protect against some skin burns.

Enclosures and/or safety curtains must be in place around laser work area to prevent stray beams. Preferably, safety door interlocks with a "laser on" warning lamp should be used with rooms where lasers are being used extensively.

14. Mechanical Testing using the Instron 1122 Testing Machine

The Instron 1122 is a screw driven mechanical testing machine with a 1000 lbf capacity capable of tension, compression, and fatigue testing. Be aware that large amounts of energy stored in a specimen during deformation may be released suddenly when fracture occurs; fragments may be ejected. Safety glasses must be worn at all times in the area. This equipment can cause injury if not operated carefully. Contact Dr. Majumdar for training to use this machine.

1. Keep hands and loose clothing/jewelery away from the machine when operating.

2. Make sure safety shields are in place to prevent flying debris when furnace is not in use.

3. Wear appropriate eye protection when using this equipment.

4. Never work alone.

5. Always protect yourself and the equipment. Leave the mechanical limit stops as set.

6. Stay CLEAR OF MOVING PARTS. In particular, keep your hands away from the test fixtures when the crosshead is moving.

7. Improper use of the equipment may cause damage and IS HAZARDOUS.

8. Use the fixtures as instructed and within specified limits. If in doubt, ask for assistance.

9. Report any hazards or injuries immediately to the personnel in charge of the lab.

10. In the event of emergency in this room, please contact Dr. B. S. Majumdar, Department Chair, or Campus Police X5555.

15. Safety Associated with Preparation of Samples for Microscopy: Mounting, Grinding, Polishing, and Etching

Everyone is required to read the Buehler Summet Laboratory Safety Guidelines located in Appendix A of this Manual to familiarize themselves with safe laboratory practice associated with the preparation of metal and ceramic samples for microscopy.

For polishing and grinding in Jones 150, safety glasses are required with a recommendation that long sleeves, gloves, long pants and closed shoes be worn. The compounds used for polishing and grinding grit may cause skin irritation thus covering exposed skin is highly recommended. If you have skin that is easily irritated or known allergies to silica, alumina, silicon carbide, or other polishing materials, then gloves, long sleeves, long pants, and closed shoes are required. When etching samples, safety glasses or face shield, gloves appropriate for the etch, lab coat with long sleeves, long pants, and closed shoes are required.

16. Emergency Call List

The Hazardous Waste and Safety Office should be called with all safety concerns. Examples include suspect toxic gases, unidentified vapors or odors, chemical spills, biological hazards, asbestos hazards, radon or radiation hazards, water or soil contamination, or any situation that may require analysis. Unless life or property is in imminent danger, do not call the Socorro fire department or Socorro Hazardous Materials Team. An assessment will be made by the NM Tech Hazardous Waste and Safety Office, and they will make the decision. Individuals of this department can be reached, day or night. Attempt to contact personnel in the following order:

APPENDIX A

The Buehler Sum-Met document is another great resource for learning how to prepare samples for optical microscopy. Please visit the Buehler Web Site:

http://www.buehler-asia.com/brochure/download_Buehler-Sum-Met-English.pdf