



# **Lab Safety and Procedures**

## **SMIF Safety Policy**

**The Management and Staff of the Duke University Shared Materials Instrumentation Facility (SMIF) have implemented all reasonable measures to ensure that the facility provides a safe working environment. It is the responsibility of all users and staff to act in a professional, courteous, and safe manner at all times while in the facility.**

**Users violating the operating and safety rules of the facility or endangering the safety of themselves or other users, will be denied further access to the laboratory**

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# 1.0 General Procedures

## 1.1 Location

The SMIF clean room and characterization lab facilities are housed on the first floor of the west wing of the Fitzpatrick CIEMAS building.

## 1.2 Lab Access and Equipment Use Certification

Access forms, equipment operating procedures, equipment certification checklists, and this safety manual can be downloaded from the SMIF web site: <http://smif.lab.duke.edu>

To gain access to the SMIF facilities and equipment, users must follow the steps shown below. Access to the SMIF labs is by card entry using Duke ID cards. *Users must be certified by a SMIF staff member in order to use any equipment on their own.*

### 1) SMIF Access

- a. Download and review the SMIF Lab Safety and Procedures Manual (This document). Users will be asked for their signature indicating that they have read and understood the Safety and Procedures Manual.
- b. Complete the appropriate SMIF Access form, have it signed by your advisor/manager, and return it to SMIF.
- c. Sign-up for the appropriate safety and procedures classes:
  - i. All users must complete the Lab Safety and Procedures class before access to the SMIF facilities is granted
  - ii. Cleanroom users must also complete the Cleanroom Safety and Procedures class before access to the clean room is granted  
Note: Biological bay users must also complete the Biological Hood and Safety training before use of biological materials is allowed
  - iii. Chemical hood users must also complete the Wet Hood and Chemical Safety class before use of chemicals is allowed

### 2) Equipment Training

Users can receive training on a specific tool in one of three ways:

- a. Train under a certified user from their own group (SMIF labor costs are not charged)
- b. Sign up for one of the equipment training sessions offered by SMIF. (SMIF labor costs will be divided between the participants)
- c. Contact SMIF to schedule individual training (SMIF labor costs are charged at full rate)

### 3) Equipment Certification

After training is completed, contact SMIF to arrange certification. A SMIF staff member must certify users on each particular tool before that user is allowed to schedule time on the tool and use the tool without supervision.

Certification is done via a checklist system that verifies the user is capable of properly and safely using the tool. You may download the certification checklists and request certification using the SMIF web site.

### 1.3 Lab Etiquette

- **Be considerate of other users**
- **Food and Drink are not allowed in any of the Equipment Labs or in the Clean Room.** Food and drink are only allowed in the seating area at the end of the main hallway.
- **Clean-up your work areas after you are done**
- Users have an obligation to one another and to the laboratory to minimize interference with other users and to maintain the quality of the laboratory and its equipment.
- Do not store samples, tools, books, materials, etc. in the lab except where designated by the SMIF staff. (Per section 4.2) If additional storage space is required, you must contact the SMIF staff first.
- **Notify SMIF staff of any issues or questions** via email to [smif@pratt.duke.edu](mailto:smif@pratt.duke.edu)

### 1.4 Hours of Operation

- The SMIF facilities are open to certified users during the following times:
  - Clean Room Lab: 8am to 8pm 7 days/week
  - Characterization Labs: 24 hours/day\* 7 days/week

\*The acid and solvent wet hoods in the Offline/Sample Prep lab can only be used from the hours of 8am to 8pm.

Per the above schedule, clean room users must exit the clean room prior to 8pm, and card access is not activated until 8am. After 8pm the air handling and exhaust units are stepped down to conserve energy and reduce operating costs of the facility. During this time, it is potentially unsafe to be in the clean room.
- After certification, users may work alone in the labs provided the following:
  - They have been certified on all equipment that they will be using
  - The work performed must be *Routine* for the user
  - The user must understand the *Risks*
  - The user follows all safety and laboratory procedures

***If using chemicals, there MUST be another person present in the lab. You may not wet etch, mix chemicals, or dispose of waste without another user in the area*** (See section 6.5.9 regarding the buddy system for chemical usage).

### 1.5 Computer Abuse

Computer abuse is a violation of university policy, and may subject the abuser to various disciplinary actions from SMIF management, the campus judicial system, and legal authorities. Abuses of the SMIF computers will have the same results as violations of

SMIF safety rules ranging from denial of access to the computers for a period of time to permanent exclusion from the facility. Computer abuse includes, but is not limited to:

- Using SMIF computers without proper authorization, or for unauthorized purposes, including using or attempting to use an account not issued to you
- Tampering with or obstructing the operation of the SMIF computer
- Inspecting, modifying, distributing, or copying software or other data (whether this is system software, data, or files of another user) without authorization
- Downloading software onto SMIF computers without authorization from SMIF management

## **1.6 User Communication**

All users must supply SMIF with a functional e-mail address that they check regularly for messages. E-mail will be the primary mechanism for notification of users of equipment and laboratory status information. Please notify SMIF staff if there is any change to your contact information that was given in your access form.

## **2.0 Alarms and Evacuations**

### **2.1 Toxic Gas Monitoring System**

The SMIF Facility has installed a sophisticated Toxic Gas Monitoring (TGM) system. This system senses the presence of dangerous gases and vapors and then annunciates the appropriate alarms and automatically shuts down the gas source. It is integrated with the CIEMAS building and fire alarm system.

### **2.2 Manual Alarm Buttons**

Manual alarm buttons are located at all emergency exit doors in the clean room, as well as at each exit door in the characterization labs. These are blue boxes mounted on the wall with a push button in the middle. In the event of an emergency that is not immediately detected by the toxic gas monitoring system (such as a large chemical spill), users should immediately evacuate the area and press the manual alarm button on their way out the door.

### **2.3 Alarms Types and Evacuation Response**

There are two types of alarms within the SMIF facilities:

#### **Flashing blue light with a continuous siren (TGM alarm)**

This is an indication of a local alarm caused by a detection event from the TGM system or from a manual alarm button activation. Upon hearing or seeing this alarm, you should immediately evacuate the area(s) where the alarm is sounding. If you have knowledge of the cause of the alarm or any other helpful information you should proceed to the SMIF office area and notify one of the SMIF staff members.

**Flashing white light with intermittent horn (Fire/Building alarm)**

This is an indication of a building wide alarm caused by the building fire alarm system or a high level detection event from the TGM system. Note that for the case of a high level detection event from the TGM system, both the TGM alarm and the fire/building alarm will annunciate simultaneously. Upon hearing or seeing this alarm, you should immediately evacuate the CIEMAS building and gather outside in the area between CIEMAS and Hudson Hall. If you have knowledge of the cause of the alarm or any other helpful information you should locate a SMIF staff member in the evacuation area to relay this information.

*Note to clean room users:* If an alarm sounds that requires you to exit the clean room you should immediately exit through the closest door (for example, at the end of the bay where you are working). You should not try to make your way to the gowning room and/or take the time to remove your clean room garment. Once safely outside the clean room or building, you may remove your garment. Once the alarm condition is over, please return your dirty garment to the pre-gowning area. (Do not take or wear it back inside the gowning room or clean room). You may get a new garment when you enter the gowning room.

**2.4 Summary**

<b>Intermittent Horn with Flashing White Light:</b>	<b>Evacuate the CIEMAS building</b>
<b>Continuous Siren with Flashing Blue Light:</b>	<b>Evacuate the affected area</b>

**3.0 Equipment Use and Availability**

**3.1 Certified Users**

Access to the laboratories does not of itself permit use of any particular instrument. You must receive training and then be certified by SMIF staff to use any piece of equipment.

Much of the equipment in the facility is highly complex and delicate. We view hands-on access as an important part of the educational process. Each instrument necessarily has rules and operational procedures that are set by the staff to assure the safe and continued operation of the instrument. Violation of these procedures or carelessness in operation can result in damage to the equipment, downtime and considerable expense.

Consequently, careless or damaging use of equipment will result in suspension of user privileges, either for a specific instrument or the facility as a whole.

Operating procedures for each tool can be found on the SMIF web site, and will be used as the basis for training on a given tool. Users will be expected to understand and be able to fully follow the operating procedure in order to be certified.

A few general policies for equipment usage are:

- Know where equipment operating procedures and manuals are located
- Do not touch or adjust any controls or settings that were not explained to you during training
- Leave all equipment in the appropriate state for the next user (as instructed during training)
- You must contact SMIF staff with any requests to modify or move equipment

## 3.2 Equipment Operations

SMIF uses an on-line login system on the SMIF web site to record usage in the clean room and on characterization equipment. Users will be trained on using the login system as part of the certification process for gaining access to a tool or lab. As this login system uses the billing information provided on your access form, **please notify SMIF immediately via e-mail if your billing or contact information changes.**

## 3.3 Equipment Use Charges

One of the fundamental reasons for the creation of the Shared Materials Instrumentation Facility was to enable access for Duke University researchers to equipment that they do not have the resources to maintain and operate on their own. As with other university-based shared facilities, hourly user fees are charged as a means of recovering the direct costs associated with operating such a facility. These fees are reviewed by the Office of Sponsored Programs and approved by Duke's central administration.

**All Users** (Duke, non-Duke university, federal, and industry) are charged hourly equipment or lab use fees. These fees are used to recover direct operating costs associated with the equipment and the laboratory (equipment and lab supplies, maintenance costs, labor to keep the tools running, etc.) Academic and non-academic users are charged at different rates. The current fees for each tool or lab are given on the SMIF web site.

## 3.4 Equipment Scheduling

All SMIF equipment is reserved through a web based calendar system accessible off the main SMIF web page at <http://smif.lab.duke.edu>. Users should understand the scheduling system and use it reserve time slots on instruments. Equipment is generally reserved on a first-come basis. You will be notified if there are certain sign-up restrictions on particular tools due to the level of usage. You must schedule time on equipment in advance.

Certified users are given a login and password that allows them to log into the calendar reservation system and schedule time on the tools for which they are certified. Other people may view the tool schedule without a login, but do not have access to schedule time.

### 3.5 Equipment Problems

Problems with equipment malfunctions, breakage, etc. should be reported to the SMIF staff. **Do not try to fix or adjust anything yourself.** This equipment is very expensive and much of it is very delicate. Considerable damage can be done at a great cost of both money and downtime by careless attempts to fix things.

**Do not call the staff at home in the evenings or on weekends about minor problems with the equipment or your process or measurements.** Send an e-mail to smif@pratt.duke.edu and the SMIF staff will address the problem the next morning. Obviously, major problems like fire or smoke, or equipment alarms should be reported immediately using the emergency contact information posted near the phones in each lab.

Any emergency involving injuries, fire, chemical spills, etc., should be first reported to the Duke Police dispatcher by dialing 911 from any Duke phone. They in turn will contact the proper response organizations (e.g., the Duke Occupational and Environmental Safety Office (OESO)). There is always someone on call from Duke OESO.

## 4.0 General Laboratory Practices

### 4.1 Visitors

You are responsible for the actions and safety of any visitor that you bring into the SMIF facilities. A “visitor” is considered to be anyone who has not completed the SMIF Safety and Procedures training class, whether or not that person is affiliated with Duke University.

**No visitors are allowed to be taken inside the clean room lab without prior approval from SMIF management.**

You may take visitors inside the characterization labs, but you are encouraged to notify SMIF management first.

### 4.2 User Storage

A limited amount of storage space, in the form of small bins is available for users in the clean room lab. These should be used for keeping only currently needed substrates, masks, etc. Clean room users will be assigned a storage bin. These bins can be removed from the storage area within the clean room while using the lab, but must be returned to their storage area before you leave the clean room.

**No chemicals of any kind may be stored in the User Storage areas.** Chemicals are to be stored only in the designated chemical cabinets.

There are no user storage areas outside of the clean room. All users should remove samples and other items from these areas when they are done and store them in their own labs.

### **4.3 Phones**

Phones are provided in the SMIF labs for the use of staff and users. The facility phones are not a substitute for office phones for users. Users should not routinely make or receive calls in the SMIF labs. SMIF staff will not routinely take messages for SMIF users.

Phone numbers for the SMIF staff are posted by each of the lab phones. Please only use the after hour numbers in case of equipment or facility emergencies.

**For immediate response to emergencies involving safety (e.g., injury, chemical spill, etc.):**

**Dial 911**

**to reach the Duke Police dispatcher. They in turn will contact the proper response organizations (e.g., the Duke Occupational and Environmental Safety Office (OESO)).**

### **4.4 Buddy System**

A “Buddy System” is in place for all chemical use in the SMIF facilities. (See section 6.5.9) For the buddy system, a knowledgeable user must be in the lab to assist you in an emergency. Additional procedures may apply to specific instruments. You will be advised of these when you are trained on each instrument.

## **5.0 Clean Room Practices**

### **5.1 Protocol for Contamination Control**

The primary limitation to clean room cleanliness is the people using the clean room. Strict adherence to our rules and common sense will allow us to maintain a level of cleanliness adequate for the types of work done in the SMIF clean room

**You are the biggest source of contamination in the clean room. Your clothes, your feet, your skin, and your hair produce particulates that may compromise your research goals.**

## Clean Room Rules and Restrictions

- **Do not** bring anything into the clean room that is not absolutely necessary for the work you are doing.
  - You should leave all coats and jackets outside the clean room on the hallway coat rack
  - Backpacks, purses and other items can be securely stored in the lockers located in the pre-gowning entrance room. These lockers are for storing items only while you are in the cleanroom, and must be emptied when you leave the cleanroom.
- **Items that are not allowed in the gowning room or clean room include:**
  - Food, drinks, or gum
  - Paper (special clean room paper lab-books can be purchased from SMIF for use in the clean room)
  - Cardboard or any other packing material
  - Pencils, erasers, or felt-tip markers
- **You must clean off any items** (sample carriers, equipment, parts, tools, etc) before bringing them into the clean room. Wipes and cleaning solution are located in the gowning room for this purpose.
- **Activities that are not allowed in the gowning room or clean room include:**
  - **Do not** wear make-up.
  - **Do not** comb your hair in the clean room or gowning room.
  - **Do not** run or engage in “horseplay”

## 5.2 Clean Room Attire

- Prior to entering the gowning area of the clean room you should ensure the following:
  - **Do not** wear dirty clothes; particularly muddy boots or shoes into the clean room
  - Bare legs should be covered.
  - Socks or nylons should be worn along with closed toe shoes that completely cover the foot.
- **Entrance and Donning Sequence**
  - Enter into the pre-gown area
  - Put on a pair of disposable shoe covers
  - Log in for clean room usage using the sign-in computer
    - *Note: Each user must independently log into the clean room using the sign-in computer. Users that enter the clean room without logging in will have their access revoked.*
  - Use your Duke card to enter the gowning area
  - Immediately put on a pair of Latex or Nitrile gloves
  - Put on your clean room garment, dressing from the head down:
    - Cleanroom hood with mask
    - Cleanroom coverall
      - Take care not to drag the legs on the floor.
      - Be sure that hood is tucked into Coverall

- Fasten the zipper completely up to the neck
    - Fasten snaps and all buttons on the suit
  - Cleanroom booties
    - Snap to the coverall and/or adjust the top of the shoe to be snug around the calf.
  - Safety glasses or goggles
- Attach your identification badge to the outside of the coverall
- Walk across the second tacky mat before entering the cleanroom
- **Exit and Doffing Sequence**
  - When exiting the cleanroom and still within the gowning room, remove the garments as follows (bottom up):
    - Remove booties and snap to bottom of leg of coverall
    - Remove coverall and hang on your designated hook
    - Remove hood and snap to top of bunny suit
  - When garments require cleaning, place the garment in the laundry container
  - When exiting the gowning room and still within the pre-gowning area, remove your gloves and disposable shoe covers and discard them in the waste container.

## 5.3 Clean Room Services

Compressed air, laboratory vacuum, house (dry) nitrogen, and de-ionized (DI) water are supplied in the clean room.

**Compressed air** is used only for pneumatic valves on equipment.

**Laboratory vacuum** is used only for spinners and chucks.

**House nitrogen** is clean, dry nitrogen obtained from the boil-off of liquid nitrogen in a storage tank. It is used for venting vacuum systems, for vacuum pump purges, and for N<sub>2</sub> guns (blowing off or drying substrates)

**De-ionized water** is available in the chemical benches. Please do not leave DI faucets or cascade rinse tanks running unnecessarily.

## 6.0 Personal Chemical Safety

### 6.1 Safety Overview and Philosophy

Safety is the primary concern in all SMIF laboratory activities. All operations must be undertaken with the safety of both the individual user and other users as the primary consideration. **Operating safely is more important than getting your project done.** As a general rule, anyone violating any safety rule or otherwise compromising his or her personal safety or the safety of others will be denied access to the laboratory.

All users should read through the **Duke Laboratory Safety manual** (specifically Sections 2 and 3 on Chemical and Fire Safety) and complete the on-line laboratory safety

training offered by the Duke Occupational and Environmental Safety Office at [www.safety.duke.edu](http://www.safety.duke.edu).

**Many chemicals and gasses used in the SMIF labs are extremely hazardous. They can cause severe and permanent damage to human tissue, resulting in serious injury or even death. It is each user's responsibility to be alert and cautious when using these chemicals and gases and to avoid all contact with them. Following the safety procedures will minimize your risk of injury.**

For the most part, rules on chemical use are formulated on the basis of basic chemical knowledge, the properties of individual chemicals, and common sense. In addition, a large volume of state and federal law covers chemical use in the workplace and disposal of waste. **In spite of rules and staff supervision, primary responsibility for safety rests with the individual user.** A responsible, considerate user with an understanding of basic chemistry, common sense, and an instinct for self-preservation will have little trouble with our chemical rules or chemical safety.

Your safety in the laboratory is determined not only by your actions but by the actions of those around you. Since the staff is in the laboratory only a portion of the time the facility is open, the users are often in the best position to observe the behavior of others. You are encouraged to point out rule violations immediately to the offenders, as well as to the staff at the first available opportunity. The access of everyone to the facility depends on maintaining a safe working environment.

We also welcome your suggestions regarding safety and laboratory procedures. **Please notify SMIF staff immediately of any safety concerns.**

## 6.2 Laboratory Hazards

Hazards in the laboratory fall into two general categories; compressed gases and wet chemicals.

### Compressed Gases

The facility uses a variety of compressed gases, some of which are **toxic, highly toxic, corrosive, flammable, or explosive**. The use of these gases is thus strictly regulated. An accident with any of these could be catastrophic. These hazards, however, can and have been minimized by the proper use of engineering controls, such as the use of proper equipment, proper confinement, ventilation, sensors, purges, safety valves, etc., and by procedural controls implemented by the staff. These will be discussed more in the section on gas safety.

### Wet Chemicals

The second, more troublesome category of hazard, concerns wet chemicals, i.e. the acids, bases and solvents commonly used in etching, cleaning and lithography. These are "hands on" hazards and, in a multi-use facility like ours, are harder to control by engineering. The chemicals we commonly use in this facility can cause severe burns,

tissue damage, organ damage, asphyxiation, and genetic damage if improperly used. These chemicals can enter the body by inhalation, ingestion, or absorption (either directly through the skin or through gloves) and may have either long or short-term health consequences. In addition, improper use of solvents can result in a major fire. These concerns will be covered more in a later section. **Users are expected to treat all chemicals with appropriate respect, and to be aware of all possible reactions that may be created, either intentionally or by accident.**

### 6.3 Sources of Chemical Information

The **Material Safety Data Sheet (MSDS)** is a convenient, condensed source for information on the properties of any chemical. The **MSDS** is a federally mandated document that must be supplied to you by the manufacturer or distributor of a chemical. It contains in summary form, the chemical composition, the physical and chemical properties, toxicology data, and instructions for handling, spill control, and waste disposal. **You must read the MSDS for any chemical you use in SMIF.**

Safety data sheets for all chemicals approved for use in SMIF are available in notebooks located in the seating area at the end of the main hallway.

### 6.4 Terminology

The following terms are often encountered when reading about the properties of chemicals and the toxicity of chemicals. Simple definitions are included here to help you understand the properties of common chemicals when referring to the MSDS or other references. This is not intended to be a complete reference on Toxicology or Chemical Safety.

#### 6.4.1 Chemical Properties Terms

- **Corrosive** chemicals are those that cause visible destruction of, or irreversible alterations in, living tissue by chemical action at the site of contact. Examples of corrosive chemicals include strong acids and strong bases.
- **Flammable** materials include aerosols, gases, liquids, and solids. Flammable gases can form a flammable mixture with air. Flammable liquids refer to any liquid having a flashpoint below 100°F.
  - **Flash point** is the minimum temperature of a liquid at which it gives off sufficient vapor to form an ignitable mixture with air. Liquids with a flash point near room temperature can be ignited very easily during use.
- **Pyrophoric** chemicals spontaneously ignite in air. No source of ignition (spark) is needed. They react spontaneously when exposed to oxygen. Silane is an example of a pyrophoric gas.
- **Exothermic Reaction** is a reaction that produces heat (releases energy).

#### 6.4.2 Types of Exposure

- **Acute Exposure** as used in toxicology refers to a short-term exposure. It has nothing to do with either the severity of the exposure or the severity of the effect. The type of exposure occurring during an accidental chemical spill is properly described as an acute exposure.
- **Chronic Exposure** as used in toxicology refers to a long-term exposure. Again, it has nothing to do with the severity of the exposure, the severity of the consequences, or the duration of the consequences. Chronic exposures can be the result of chemicals in the workplace, the home, or the environment. Chronic exposures are usually the result of carelessness, ignorance, or neglect, and not the result of an accident.
- **Local Exposure** refers to exposure limited to a small area of skin or mucous membrane.
- **Systemic Exposure** means exposure of the whole body or system, through absorption, ingestion, or inhalation.

### 6.4.3 Types of Effects

- **Acute Effects** refers to the duration of the symptoms. Acute means symptoms lasting a few hours or days. Again, it has nothing to do with the severity of the effects.
- **Chronic Effects** are long-term effects, manifested by prolonged duration and continuing injury.
- **Local Effects** occur in a small area, at the place of contact.
- **Systemic Effects** occur throughout the body, or at least away from the point of contact.
- **Allergies and Hypersensitivity** are reactions by particular individuals to particular chemicals, caused by heredity or prior overexposure. Hypersensitive individuals should avoid exposure to the offending agents.

### 6.4.4 Exposure Levels

- **TLV-TWA (Threshold Limit Value -Time-Weighted Average)** - This is the (averaged) level to which you can be exposed 8 hours a day, 5 days a week for a working lifetime, without adverse health effects. These levels are set by ACGIH (American Conference of Governmental Industrial Hygienists). This level is most relevant to chronic (long term) exposure to chemicals in the work place.
- **IDLH (Immediately Dangerous to Life and Health)** - This level represents any condition that poses an immediate threat of loss of life; may result in irreversible or immediate-severe health effects; or may result in eye damage; irritation or other conditions that could impair unaided escape. It is the value most appropriate to sudden, one time accidental exposures.
- **STEL - Short Term Exposure Limit-** Maximum concentration to which you can be exposed for 15 minutes, up to 4 times a day without adverse effects.
- **PEL - Permissible Exposure Limit-** The statutory equivalent of TLV.
- **LD50** - The dose at which 50 % of those exposed will die. Separate levels apply to various modes of exposure (inhalation, dermal, etc.)

## 6.4.5 Toxic Effects

- **Carcinogen-** A substance producing or inciting cancerous growth.
- **Mutagen-** Capable of inducing mutations.
- **Teratogen-** A substance causing damage or death to a fetus.

## 6.5 Chemical Use

**Users must attend the Wet Hood and Chemical Safety class and be trained and certified on chemical hood usage before using the chemical hoods. Chemicals are to be used ONLY in the exhausted chemical hoods.**

### 6.5.1 Chemical Authorization

Only specifically authorized chemicals may be used in the SMIF laboratories. Many standard chemicals have been pre-authorized.

**No other chemicals may be brought into the facility without the specific authorization of SMIF.** SMIF may issue approval based on a review of the **MSDS** and other relevant information provided by the user. Approval of new chemicals is not guaranteed, as certain chemicals may not be allowed if they are too hazardous or are incompatible with other uses of the laboratory.

You should obtain approval **before** buying or ordering a chemical. **Do not show up with chemical in hand expecting instant approval.** Chemicals must not be brought and left in the facility awaiting approval.

**Once approved by SMIF staff, all chemicals must be labeled with the following:**

- Name of chemical as it appears on the MSDS
- Physical and health hazards of the chemical
- Contact information for the main owner/user of the chemical when applicable

Once a chemical is approved, SMIF will contact you to verify proper storage, use, and disposal of the chemical.

### 6.5.2 Chemical Practice

A little bit of common sense and courtesy when using chemicals will make the laboratory safer and make it easier for the staff to monitor chemical use.

- You must clearly and legibly label all chemical containers (beakers, etc) with your name, the chemical name, hazard class (corrosive, flammable, etc) and date if that container will be left in the hood for any length of time without you immediately present at the hood.

- **Do not leave any unlabeled chemical containers in the hoods, even for “just a minute”.**
  - Chemicals must be properly disposed of as soon as you are finished.
- These procedures are necessary to allow us to identify chemicals left behind, spilled, or forgotten.

Since we have a limited amount of chemical hood space, cooperation by users is required. If there is more than one user working in a hood, the first user in the hood has the option to exclude others from the area while he/she is working, for either safety or process reasons.

### 6.5.3 Lab-ware

SMIF provides glassware and plastic ware for all to use in the laboratory. You are not authorized to appropriate a private stash for your own use. Users are encouraged to purchase their own lab-ware for their private use if cross-contamination is a concern. All lab-ware should be thoroughly cleaned and rinsed after use and properly stored. Private use lab-ware should be stored in your individual user storage.

Do not dispose of glass or contaminated materials in the regular trash. Contact SMIF staff if there are any questions regarding proper disposal of materials. Glass should be disposed of in proper broken glass containers.

### 6.5.4 Chemical Hood Operation and Rules

**All chemical operations are to be done in the chemical benches.** You should perform your work well inside the hood, away from the front edge. However, **you should never stick your head inside a chemical hood in order to reach something at the back of the hood.** If you are using the hoods properly, you should not be able to smell chemicals outside the hood.

**You are expected to clean up after yourself.** Do not leave beakers, wipes, etc., or chemical or liquid residue in the wet hoods. The work surface should be clean and dry when you start, and clean and dry when you leave. The next user has no idea if that puddle of clear liquid is HF or just water.

#### **Keep solvents and acids separated.**

- Acids are to be used **ONLY** in the white color polypropylene chemical hoods.
- Solvents are to be used **ONLY** in the stainless steel chemical hoods. Water is not allowed in these hoods.

#### **Photolithography chemicals are to be used only in the clean room Photo Bay areas**

- Photoresist and spin on organics are to be applied only in the designated spin modules within the spin coating hoods
- Developer solutions are only to be used in the developer (base) chemical hood

**Understand the proper disposal of any chemical with which you are working.** (See section 6.7).

### 6.5.5 Hot Plates

While you may think hotplates are mundane, they can in fact be very dangerous when used in chemical hoods. Fires and melt down of the plastic hoods are both significant concerns. The following rules apply to hotplate use:

- **Hot plates used for heating chemicals must be attended.** You must be in the laboratory and near the hot plate whenever it is powered on.
- You may not heat solvents with flashpoints of <130 F.

### 6.5.6 Personal Protective Equipment

When using chemicals, users must minimally wear the following items:

#### Clean Room Laboratory

For Acid or Developer Use	For Solvent or Spin Coating Use
In addition to the standard clean room attire, the following items are to be worn: <ul style="list-style-type: none"><li>• Chemical Apron</li><li>• Chemical Face Shield</li><li>• Chemical Gloves</li></ul>	Standard clean room attire <i>including gloves and goggles</i>

#### Offline/Sample Preparation Laboratory

For Acid Use	For Solvent Use ONLY
<ul style="list-style-type: none"><li>• Chemical Apron</li><li>• Chemical Face Shield</li><li>• Chemical Gloves</li></ul>	<ul style="list-style-type: none"><li>• Goggles or Chemical Face Shield</li><li>• Lab gloves (latex or nitrile)</li><li>• Lab coat (optional)</li></ul>

- Check chemical gloves for holes.
- Wash gloves when contaminated.
- Wash gloves before removal.
- Wear gloves to open chemical cabinets.
- Wash and remove gloves before touching anything else (door knobs, notebooks, phone, microscopes, etc.)

The aprons, face shields and chemical gloves are to be worn **only** at the hoods. They must be removed when using other equipment in the laboratory.

### 6.5.7 Chemical Supplies and Storage

SMIF stocks and supplies the chemicals commonly required for processing in the facility. Users are neither allowed to bring in their own stocks of these chemicals or to appropriate as a private stash any of these chemicals. We do not have room for people to have private bottles of commonly used chemicals.

We have very limited chemical storage for user specific chemicals. You may also not store custom solutions without staff approval. Mix only as much of these as you need each day. All special chemicals and solutions must be labeled with the arrival date and the owner's name if appropriate. Chemicals older than 1 year will be periodically disposed of without question.

Working stocks of chemicals are kept in the chemical cabinets near each chemical area. Users must not open a new bottle until the old one is empty. When you empty a chemical bottle, do not return it to the chemical storage cabinet.

- Empty acid bottles should be thoroughly rinsed out with water, and the empty bottle left in the chemical hood for disposal by SMIF staff. (Rinse water may be safely disposed in the normal drain).
- Empty solvent bottles should be placed in the solvent chemical hood with the cap off and allowed to dry. SMIF staff will then properly dispose of the empty bottle.

### 6.5.8 Pregnancy

Users who believe themselves to be pregnant should discuss laboratory use with the SMIF director as soon as possible. This need not severely restrict laboratory use but should nonetheless be discussed. (See discussion of solvents in photoresist in section 6.6.5.)

### 6.5.9 Buddy System for Chemical Usage

A buddy system applies to the use of wet chemicals in any of the SMIF facilities. The buddy system requires that another knowledgeable user be in the laboratory, aware of your situation, and close enough to be of assistance if you have an accident. He or she need not be constantly at your side, just available and aware. **This is especially important for after-hours and weekend usage.**

A limited exception to this rule is made for the use of lithography chemicals. You may spin resist and develop patterns without a buddy present. It is still recommended, however, that even in these cases, another person be available to assist you in the laboratory.

### 6.5.10 Contact Lenses

In the past, it was not considered good laboratory practice to wear contacts in any laboratory. Recently the American Chemical Society has changed its recommendation,

and SMIF has adopted its practice. Contact lenses may be worn in the laboratory, provided that appropriate supplemental eye protection is worn when actually using chemicals. In SMIF, this would be the full-face shield when using acids and goggles or face shield when using solvents and lithography chemicals.

### 6.5.11 Emergency Response Equipment

**Spill kits and spill control pillows** are available in the laboratory for absorbing liquid chemical spills. They are located near the chemical hoods.

**Emergency showers and eyewashes** are located near all chemical hoods in the clean room and offline/sample preparation lab. Most chemical burns, particularly in the eyes, should be washed for 20 minutes before seeking further medical attention.

**First Aid Kits** are located in the clean room on the main aisle and in the offline/sample preparation lab.

**Calcium Gluconate Gel** is available in the first aid kits for application to HF burns. This should be applied promptly, but is not a substitute for prompt medical attention.

### 6.5.12 Chemical Accidents

The following actions should be taken in the event of a chemical spill:

#### **Minor Spill (< 1 liter)**

- Alert people in the immediate area of the spill
- Avoid breathing vapors from the spill
- Acids: Confine liquid to a small area using spill pillows found in the acid spill kit
- Solvents: Use wipes or absorber found in solvent spill kit
- Developer: Use spill pillows found in the acid spill kit
- Notify SMIF immediately by calling emergency numbers posted near the phone
- Wait for SMIF personnel to arrive. SMIF personnel will complete the clean-up of the affected area.

#### **Major Spill (> 1 liter)**

- Attend to injured or contaminated persons and remove them from exposure
- Press the closest manual alarm button (blue box) and evacuate the area
- Make yourself available to the SMIF staff and/or emergency responders and be prepared to tell the following:
  - What chemical(s) are involved
  - How much was spilled
  - Where the spill is located
  - Nature of any injuries

All accidents involving chemicals and all accidents involving personal injury must be reported to the SMIF management in writing (e-mail) as soon as possible after the incident. Explanations should include the nature of the event, the procedures being followed or not followed at the time, and suggested actions for preventing future similar

incidents. All injuries should also be reported within 24 hours to Workers' Compensation, via a form available on the HR web site:  
<http://www.hr.duke.edu/forms/injury.html>.

### 6.5.13 Reminders and Final Checklist

Common sense is the most valuable aid you have in working with chemicals. If you are unsure about any aspects related to the chemicals you will be using, do not use them before asking the SMIF staff for assistance. It is impossible to enumerate all the rules and cautions applicable to chemical use, but here are a few more to consider:

- Always pour chemicals slowly to avoid splashing
- When mixing, always pour acid into water – never pour water into acid
- Completely rinse any used glassware or Teflon containers
- Perform all chemical operations carefully; especially moving, mixing, and pouring
- Open chemical containers must remain under the hoods. Move them around inside, not outside
- Keep your head outside the hood enclosure at all times
- Don't sit down at the hood, it puts your face directly in the fume path
- Finish open chemical bottles before opening new ones
- Don't use N<sub>2</sub> blowguns near open chemicals
- Don't use plastic beakers on hot plates
- Clean up after yourself and be careful

## 6.6 Specific Chemical Hazards

### 6.6.1 Acetone and Flammable Solvents

**Acetone** is widely used throughout the facility. It is a very flammable solvent with a low flash point, (i.e. it can be ignited at a low ambient temperature). Because of this it presents a significant fire hazard. A spill of a gallon bottle of acetone could cause a catastrophic fire or explosion. It should not be transported except in chemical buckets. Solvents should also be handled with care in the hoods and not used near hot plates. Spilled solvent can be ignited by the hot plates. Spilled solvents can react explosively with chemical oxidizers present, e.g., peroxides or nitric acid. Spilled solvents should be contained immediately with spill control pillows.

### 6.6.2 Hydrofluoric Acid

**Hydrofluoric acid, HF**, presents a significant hazard for personal injury. It is used in the lab in its pure form, diluted, and as the active component of BOE, Buffered Oxide Etch. It is used for etching silicon dioxide and particularly for stripping the native oxide prior to further processing. HF, however, is a very hazardous chemical, much more so than any of the other acids. At the concentrations used in the laboratory, an HF "burn" is initially painless. The acid however will silently eat away at your flesh and the damage will penetrate deeper and deeper, until it comes to the bone. This can result in irreversible tissue damage, and **at some point, if left untreated, you may die.**

Simple washing of an HF splash is not sufficient to prevent damage. If you suspect you have been exposed to HF, you should immediately do the following:

1. Rinse off the exposed area with water (e.g., the safety shower)
2. Apply Calcium Gluconate Gel to the exposed area
3. Call 911 and request immediate medical assistance. Be sure that medical personnel know that it is an HF burn and know that it requires specific treatment different from a common acid burn.

Using safe practices to prevent exposure to HF are of utmost importance. Be absolutely certain that you don't get it on you by being very careful and wearing full chemical protection gear.

HF etches silicon dioxide very well. Therefore, it also etches glass. It must not be kept in a glass bottle, used in a glass beaker or disposed in a glass waste bottle. Plastic lab-ware is available for this purpose. HF, like all other chemicals, must only be used in the chemical hoods.

### 6.6.3 Piranha

Liquid **piranha** is a common name applied to a mixture of Hydrogen Peroxide and Sulfuric Acid. It is extremely aggressive toward carbonaceous materials (e.g. flesh and photoresist residue, equally). It also removes heavy metal contamination. It is used by some users for cleaning wafers.

### 6.6.4 Chlorinated Solvents

Chlorinated solvents (e.g., **chlorobenzene, trichloroethylene, and methylene chloride**) are used in various resist processes. They are particularly bad for you, causing cancer, organ damage, etc. They should not be mixed with normal solvents in waste bottles. There are separate waste bottles for chlorinated solvents. As with most solvents, they can be readily absorbed through the skin.

### 6.6.5 Photoresists (Glycol Ethers)

Commercial photoresists and electron beam resists are dispersed in a variety of solvents. The composition of these mixtures is generally not disclosed on the bottle; you must look on the MSDS for it. One family of chemicals, the **glycol ethers**, commonly used in photoresists, masquerades under a variety of names. Members of this family of chemicals have been shown to be teratogens and have other effects on reproduction in laboratory animals. A number of recent studies have found evidence that these chemicals can lead to miscarriage and other reproductive effects.

The liquid and vapor are eye and respiratory tract irritants and may cause kidney damage, narcosis, and paralysis (in simple terms, it damages your kidneys, eyes, lungs and brains). Primary routes of exposure are inhalation, skin absorption, and skin and eye contact with vapors.

As with all chemicals, these are only the effects we know about. These experimental laboratory exposures were large amounts but nonetheless it is prudent to be careful with these solvents. **Don't be sloppy with photoresist.** If being used properly, you should not be able to smell the photoresist.

### 6.6.6 Peroxides

All peroxides are highly oxidizing materials. Considerable energy can be released in their reactions with common materials. Some peroxide compounds are unstable, and can explode. We have **hydrogen peroxide** in the facility. Extreme care should be used in mixing solutions containing peroxides. Peroxides are incompatible with all forms of organic solvents and flammable materials.

## 6.7 Chemical Waste

### **All chemical waste must be poured into the designated drain or container**

All chemical waste is to be collected and neutralized or consolidated, bottled and sent out as regulated chemical waste. Waste bottles are collected by the Duke OESO, repackaged, and taken away by a licensed waste hauler. The waste is burned, neutralized, or buried in licensed facilities, in accordance with EPA rules.

Each chemical hood has a drain or container for disposing of the chemicals that are allowed in that hood:

- Acid and developer hoods: Use the acid waste drains. This waste is collected in the acid waste neutralization system located in the basement.
- Solvent hoods:
  - Non chlorinated solvents: Use the solvent waste drain. This waste is collected in containers below the hood. Full containers are taken by SMIF staff and transferred to an OESO solvent waste container and then taken to an OESO collection point.
  - Chlorinated solvents (any solvent with the word “chloro” in its name): Use the designated waste container located in the hood. Full containers are collected by SMIF staff and taken to an OESO collection point.
- Spin Coat hoods: Solvents are collected the same as solvent hoods. However, photoresist and other spin on organics should never be poured down the waste drain, but rather collected in a designated container.

Solid materials that are contaminated with chemical waste (such as aluminum foil, wipes, dispensers, etc.) should be packed into a zip lock bag and properly labeled with the type of waste, your name, and date. The waste bag should be completely sealed and placed in the appropriate contaminated waste container. They should never be placed in a regular trash container.

Waste containers must be used properly and **NOT** overfilled.

Disposal of chemical waste is expensive. Generally, it costs 5 to 10 times the original cost of the chemical to dispose of the waste. Please minimize your chemical waste by minimizing both your purchases and your usage. With your help we can minimize the number of waste bottles sitting around, minimize the cost, and minimize the damage to the environment all at the same time.

## 7.0 Gas Safety

The gases used within the facility are generally supplied under high pressure from steel compressed gas cylinders. In many cases, these cylinders are housed in special gas cabinets and fitted with a variety of high purity valves, regulators and flow control devices. Gas cylinders must be treated with respect in all cases. An enormous amount of energy is stored in the compressed gas. **Gas bottles are to be securely chained or strapped at all times.** In addition, many of these gases are toxic, or at least severely corrosive. Finally, improper use of gas cylinders and valves can result in contaminated gas and ruined samples and equipment.

**Compressed gas equipment in the facility is not user serviceable. Gas bottles are to be changed only by the appropriate staff members.**

### 7.1 Hazardous Gases Used

#### 7.1.1 Pyrophoric Gases

Pyrophoric gases are defined as gases that will ignite spontaneously in air at temperatures of 130 F or below. **Silane (SiH<sub>4</sub>)**, which is used for the deposition of CVD silicon nitride and silicon dioxide, is an example of a pyrophoric gas. It spontaneously ignites in air at concentrations between 4 % and approximately 90 %.

#### 7.1.2 Corrosive Gases

Corrosive gases used in the SMIF facility include the chlorine-based gases, **Chlorine (Cl<sub>2</sub>)**, **Boron Trichloride (BCl<sub>3</sub>)**, and **Silicon Tetrachloride (SiCl<sub>4</sub>)**. These gases are used in reactive ion etch systems and are confined in exhausted cabinets.

Chlorine is severely corrosive and is choking to breathe. Chlorine forms HCl in the lungs, causing severe tissue damage that can be fatal. As with many other corrosive gases, the effects of exposure may not be noticed for a few days. In all cases, medical attention should be sought immediately following exposure, not at the onset of symptoms.

Because of the small amounts used, the ventilation used, and the low odor threshold for chlorine exposure, accidental chlorine gas exposure is not considered a significant risk.

### 7.1.3 Flammable Gases

Flammable gases used in the SMIF facility include **Hydrogen (H<sub>2</sub>)** and **Methane (CH<sub>4</sub>)**. These gases are used in reactive ion etch and/or annealing systems and are confined in exhausted cabinets.

### 7.1.4 Cryogenic Gases (Liquid Nitrogen)

Most people do not think of **Nitrogen** as a hazardous gas. However, excessive amounts of Nitrogen released into a confined space can quickly produce an oxygen deficient environment. More people die of asphyxiation by nitrogen than by any of the “toxic” gases discussed here.

We use liquid nitrogen for many things in the laboratory. Liquid nitrogen dewars are used to fill cold traps for some of the analytical equipment, and the boil off from a large tank is used for the supply of “house” nitrogen in the clean room.

### 7.1.5 Highly Toxic Gases

SMIF does not currently house any highly toxic gases. Examples of highly toxic gases used in the semiconductor industry include phosphine and arsine.

## 8.0 Fire Safety

You will be shown the locations of all emergency exits as well as the fire pull alarms when you use the clean room or characterization labs for the first time.

In the event of fire, follow the posted Duke fire emergency procedures (**RACE**):

**R** = Remove all persons in immediate danger

**A** = **Activate manual fire pull alarm and dial 911**

**C** = Close doors to prevent spread of fire

**E** = Extinguish the fire (emergency personnel only)