



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

SMIF Safety Policy	1
1.0 Consequences of Violating SMIF Rules and Policies.....	4
2.0 General Procedures	4
2.1 Location	4
2.2 Lab Access and Equipment Use Certification	4
2.2.1 Card Access for After Hours and Cleanroom Usage	6
2.2.2 SMIF Access Renewal.....	6
2.3 Lab Etiquette.....	6
2.4 Hours of Operation	7
2.5 Computer Abuse and Data Security.....	7
2.6 User Communication	8
3.0 Equipment Use and Availability.....	8
3.1 Certified Users	8
3.2 Equipment Use Charges.....	9
3.3 Equipment Scheduling.....	9
4.0 General Laboratory Practices	10
4.1 Visitors.....	10
4.2 User Storage.....	10
4.3 Phones.....	11
4.4 Buddy System	11
5.0 Clean Room Practices.....	11
5.1 Protocol for Contamination Control	11
5.2 Clean Room Attire	12
5.3 Clean Room Services.....	13
5.4 Areas off limits to users	14
5.5 Clean Room Monitoring	14
6.0 Alarms and Evacuations	14
6.1 Toxic Gas Monitoring System	14
6.2 Manual Alarm Pulls/Buttons.....	14
6.2.1 Fire Alarm Pull	14
6.2.3 Gas/Chemical Alarm Button.....	14
6.3 Alarms Types and Evacuation Response.....	15
6.4 Summary.....	15
7.0 Personal Chemical Safety.....	16
7.1 Safety Overview and Philosophy.....	16
7.2 Laboratory Hazards.....	16
7.3 Sources of Chemical Information	17
7.3.1. SDS (Safety Data Sheet).....	17
7.3.2. SOPs (Standard Operating Procedures)	18
7.4 Terminology.....	18
7.4.1 Chemical Properties Terms.....	18
7.4.2 Types of Exposure	18
7.4.3 Types of Effects	19
7.4.4 Exposure Levels.....	19
7.4.5 Health Hazards.....	20
7.5 Chemical Use	20

7.5.1 Chemical Authorization	20
7.5.2 Chemical Practice	20
7.5.3 Lab-ware	21
7.5.4 Chemical Hood Operation and Rules	22
7.5.5 Hot Plates	22
7.5.6 Personal Protective Equipment	23
7.5.7 Chemical Supplies and Storage	24
7.5.8 Pregnancy.....	24
7.5.9 Buddy System for Chemical Usage	24
7.5.10 Contact Lenses	25
7.5.11 Emergency Response Equipment	25
7.5.12 Chemical Accidents	25
7.5.13 Reminders and Final Checklist	26
7.6 Specific Chemical Hazards	26
7.6.1 Acetone and Flammable Solvents.....	26
7.6.2 Hydrofluoric Acid.....	27
7.6.3 Sulfuric Acid.....	28
7.6.4 Piranha	28
7.6.5 Nitric Acid	29
7.6.6 Chlorinated Solvents.....	29
7.6.7 Photoresists (Glycol Ethers)	29
7.6.8 Peroxides.....	29
7.6.9 Highly Toxic Compounds used in EM Sample Preparation.....	30
7.7 Chemical Waste	30
8.0 Gas Safety	31
8.1 Hazardous Gases Used.....	31
8.1.1 Pyrophoric Gases	31
8.1.2 Corrosive Gases	32
8.1.3 Flammable Gases.....	32
8.1.4 Cryogenic Gases (Liquid Nitrogen).....	32
8.1.5 Highly Toxic Gases.....	32
9.0 X-Ray Safety.....	33
10.0 Fire Safety.....	33

1.0 Consequences of Violating SMIF Rules and Policies

The consequences for violating SMIF safety rules and operating policies are outlined below:

- If a user is found to violate SMIF rules or policies:
 - They will be sent one warning e-mail (with their advisor copied) and will be put on “probation” for a period of 3 months
 - During the probation period, the user will only be allowed to use SMIF during normal working hours, Mon-Fri 8am-5pm.
- If SMIF rules are violated a second time while on probation
 - Their SMIF access will be removed for 1 month
 - Probation period extends to 1 year
 - The e-mail notification of the violation to the user and advisor will be followed up with a written letter that requires a signed acknowledgement
- If SMIF rules are violated a third time while on probation
 - The user and their advisor will attend a hearing with the SMIF Advisory Committee and the length of expulsion from SMIF will be determined by the committee (minimum of 6 months)
 - The meeting will be followed up with a written letter that requires a signed acknowledgement

Note: Activity within the SMIF facility is monitored for safety and security reasons using video cameras placed around the labs.

2.0 General Procedures

2.1 Location

The SMIF clean room and characterization lab facilities are housed on the first floor of the 1500 wing of the Fitzpatrick CIEMAS building and in room B606 in the basement of the Fitzpatrick CIEMAS building.

2.2 Lab Access and Equipment Use Certification

Access instructions and forms, equipment operating procedures, equipment certification checklists, and this safety manual can be downloaded from the SMIF web site at <https://smif.pratt.duke.edu/access-training>.

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. Get set-up in the CoreResearch@Duke system, which is used for equipment scheduling and billing.

Duke Users

- Contact your PI or Grant Manager to get set-up in the CoreResearch@Duke system.
- Instructions for doing this can be found at <https://medschool.duke.edu/research/research-support/service-centers/coreresearchduke-training-resources/coreresearchduke-0> Scroll down to find the guide for “How to Add a Facility User and Link to a Project”
- Note: Only the PI or grant manager of the fund code can set you up in CoreResearch@Duke. SMIF does not have the ability to do this.

External Users

- Complete the SMIF External User Access Form found at <https://smif.pratt.duke.edu/external-users>. Return a signed copy to SMIF via email to smif-billing@duke.edu.
 - SMIF will request a Guest Account for you. Duke OIT will send you an email requesting information from you. Follow the instructions in the email to submit your information. Duke OIT will email you and copy the SMIF Director with your Duke NetID and UniqueID once they are assigned. Note: Your Duke guest account will need to be renewed annually. You will automatically receive an email from Duke about a month before your renewal date. When you receive this email, just forward it to smif-billing@duke.edu and SMIF will ensure that your Guest account is active for another year.
 - If you are associated with an institution or company that was not previously set up in CoreResearch@Duke, SMIF will submit the information to get your institution or company added.
 - SMIF will notify you when you are completely set up in CoreResearch@Duke.
2. Review this SMIF Lab Safety and Procedures manual
 3. Complete the required SMIF Safety & Procedure Classes
 - All users must complete the on-line SMIF General Lab Safety and Procedures class and score 100% on the associated test.
 - Cleanroom users must also complete the Cleanroom Safety and Procedures class offered via zoom. The class schedule and sign-up can be found [here](#).
 - Chemical Hood users must also complete the on-line SMIF Chemical Safety class and score 100% on the associated test.
 4. Schedule training for the equipment you are interested in using by one of the following methods:
 - Go to the appropriate Equipment and Procedures page (<https://smif.pratt.duke.edu/capabilities>) and click on the primary training contact name to send an email requesting training.
 - Train with a current certified user (e.g., someone from your lab who has been certified to use the equipment). Note – SMIF labor fees are not charged for this option

5. Contact the appropriate SMIF staff member to request certification on the equipment. Certification by a SMIF staff member is required, even if you have trained with someone else.
6. Following certification you will be authorized to use the equipment on your own, and will be given access to the equipment reservation calendar.

2.2.1 Card Access for After Hours and Cleanroom Usage

Your Duke Card will be activated for the appropriate card readers after completion of the SMIF safety classes:

- SMIF Main door and Soft Materials Lab (B606) door following a 100% score on the SMIF General Safety and Procedures test
- Clean Room entrance following completion of the Cleanroom Safety class

A Duke card is only needed for after hours and weekend access to SMIF, or for access to the cleanroom and the B606 Soft Materials Lab. External Users can request to have a Duke Visitors Card for access.

2.2.2 SMIF Access Renewal

External users must renew their Duke Guest accounts (NetID) and Duke cards annually. Duke OIT will automatically notify you via e-mail one month before renewal is due. The email smif-billing@duke.edu to request renewal of your Guest account. If SMIF does not receive a renewal request from you, your access to SMIF and CoreResearch@Duke will expire.

There is no annual renewal requirement for Duke internal users. Duke users just need to always be assigned to at least one active Duke fund code in CoreResearch in order to reserve equipment time. CoreResearch will not allow you to make a reservation using an expire fund code.

Users are not required to re-take any safety classes or equipment training sessions. However, you are strongly encouraged to contact the appropriate SMIF staff member if you have not used an instrument for a long period of time, and SMIF will be glad to provide refresher training and assistance.

2.3 Lab Etiquette

- **Be considerate of other users**
 - Use respectful, courteous and helpful language when communicating with other users and SMIF staff. Loud, boisterous and intimidating language will be a violation of accepted workplace standards and is not appropriate
 - Disruptive, discourteous and/or insubordinate conduct will not be tolerated

- Acting inappropriately (such as using profane, suggestive or abusive language) is a violation of accepted workplace standards and will not be tolerated
- Conduct that causes or threatens harm to others or that constitutes persistent, unwanted behaviors will not be tolerated and will be grounds for removal of access
- 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
- **Food and Drink are not allowed in any of the Equipment Labs or in the Clean Room.**
- **Clean-up your work areas after you are done**
- 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

2.4 Hours of Operation

- The SMIF facilities are open to certified users during the following times:
 - Clean Room Lab: 7am to 12am 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 7am to 12am.

Per the above schedule, clean room users must exit the clean room prior to 12am, and card access is not activated until 7am.
- **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).

2.5 Computer Abuse and Data Security

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

Data Security

- As long as disk space allows, users may save their data and image files on the SMIF instrument computers. However, the SMIF shared instrument computers have inherent data security risks. If a user's project requires them to keep their data secure, then it is the user's responsibility for minimizing the security risk of their own data by promptly removing data and images from the SMIF shared instrument computers and moving it to secure storage.

2.6 User Communication

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.

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 invoke the consequences outlined in Section 1.0 and 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

- ***Do NOT attempt any equipment repairs on your own without first contacting SMIF.***
 - Even what might appear to be a simple fix or repair can result in longer term equipment damage if not done correctly. This is especially important for those of you working after hours and weekends, when SMIF staff are not around. Always contact SMIF by e-mail or phone if problems occur rather than attempting to fix them on your own.
- 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 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 at <http://smif.pratt.duke.edu/user-fees-and-billing>.

3.3 Equipment Scheduling

All SMIF equipment is reserved through the *CoreResearch@Duke* system. SMIF specific guides for using this system can be found at <http://smif.pratt.duke.edu/reservations>. General help for *CoreResearch@Duke* is available [here](#). Users should understand the scheduling system and use it to reserve time on all 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.

Important Things to Know about CoreResearch@Duke

- CoreResearch works **ONLY** on ***Chrome, and the Microsoft and Apple web browsers***. Mozilla FireFox is not a supported browser at this time.
- You must have a Duke NetID and password to login and use CoreResearch@Duke

- SMIF bills you based on your actual usage of the instrument or lab. However, the system defaults to your reserved time for billing if you do not start/stop your reservation manually. ***This also means that you will be billed for your reservation if you do not show up, unless you cancel the reservation ahead of time.***
- ***If you can't login to CoreResearch@Duke*** (assuming you've entered your NetID and password correctly): this means you probably have not been set up with a fund code or project. For Duke users, only your PI or Grant Manager can set you up – this can't be done by SMIF. For external (Non-Duke) SMIF users, please contact smif-billing@duke.edu to get set up in the system.

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

Users may use their personal cell phones while working in the SMIF facility. A cordless phone is provided in the SMIF cleanroom for the use of staff and users. This phone is not a substitute for office phones for users. Users should not routinely make or receive calls on the SMIF cleanroom phone. SMIF staff will not routinely take messages for SMIF users.

Phone numbers for the SMIF staff are posted at the entrance to the sample prep lab, the entrance to the cleanroom, inside the cleanroom near the cordless cleanroom phone, and the entrance to the basement Soft Materials Lab (B606). Please only use the after hour numbers in case of equipment or facility emergencies.

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

Dial 911 (from a Duke Phone number)

Dial 919-684-2444 (from a cell phone)

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 7.5.9) 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 SMIF hallway across from the large whiteboard. 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** 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.
 - It is recommended that you refrain from wearing make-up in the cleanroom. Make-up can shed, causing particulates, and also tends to stain the inside of the cleanroom head cover.
- **Entrance and Donning Sequence**
 - Enter into the pre-gown area
 - Using the computer in the pre-gown area, log into CoreResearch and start a Cleanroom Tool Group Reservation
 - Note: Each user must independently log into CoreResearch and start a Cleanroom Tool Group reservation. 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 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

You must have your cleanroom identification badge displayed on your garment at all times while working in 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 discard them in the waste container.
- Using the computer in the pre-gown area, log into CoreResearch and stop your Cleanroom Tool Group Reservation. ****You must stop your Cleanroom Tool Group Reservation in CoreResearch or you will be billed using an end time of 11:59PM.***

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 N2 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.

5.4 Areas off limits to users

Users are not allowed to enter the following areas without permission from SMIF:

- Chases (mechanical areas between the equipment bays)
- Equipment move-in (area through the double doors to the left)
- Gas/Chemical storage room

5.5 Clean Room Monitoring

All entries into the cleanroom are monitored via card reader swipe records and the CoreResearch computer logins to insure that users are logging in as appropriate and entering and exiting the cleanroom during the appropriate hours.

Activity within the cleanroom is also monitored for safety and security reasons using video cameras placed around the lab.



6.0 Alarms and Evacuations

6.1 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 Fitzpatrick building and fire alarm system.

6.2 Manual Alarm Pulls/Buttons

6.2.1 Fire Alarm Pull

Manual fire alarm pulls are located at all exit doors of the SMIF facility. These should only be pulled in the event of fire or smoke.



6.2.3 Gas/Chemical Alarm Button

Manual alarm buttons are located at all emergency exit doors in the clean room, at each exit door in the characterization labs, and along the hallways in SMIF. 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.



6.3 Alarms Types and Evacuation Response

There are two types of alarms within the SMIF facilities:

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 Fitzpatrick building and gather outside in the area between Fitzpatrick 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. ***Do not re-enter the building until the Fire Alarm is silenced***



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 locate a SMIF member to relay this information. After hours, please call or text a SMIF staff number using the emergency numbers posted on the SMIF door. ***Do not re-enter the SMIF labs until the TGM alarm is silenced.***



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.

6.4 Summary

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

7.0 Personal Chemical Safety

7.1 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.

Many chemicals and gasses used in the SMIF labs are extremely hazardous. They may cause severe and permanent damage, resulting in serious injury or even death. It is each user's responsibility to be alert and cautious when using these chemicals and gasses 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.**

7.2 Laboratory Hazards

Hazards in the SMIF laboratory include the following:

Wet Chemicals

Wet chemicals, i.e. acids, bases and solvents, are commonly used in etching, cleaning, lithography, and sample preparation. These are “hands on” hazards and, in a multi-use facility like ours, are hard 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. Mixing of incompatible chemicals (such as oxidizers and organics) can cause reactions and/or fires/explosions. 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.**

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.

Cryogenic Liquids/Gasses (e.g. LN₂)

Most people do not think of **Nitrogen** as a hazardous gas. Liquid nitrogen (LN₂) vapors can rapidly freeze skin tissue and eye fluid, potentially resulting in cold burns, frostbite, and permanent eye damage. Excessive amounts of Nitrogen released into a confined space can quickly produce an oxygen deficient environment, causing unconsciousness or even death. More people die of asphyxiation by nitrogen than by any of the “toxic” gases.

Nanomaterials and Powders

Users wanting to characterize powders or nanomaterials in the SMIF facility must review the appropriate safety information documents supplied by Duke University’s Occupational and Environmental Safety office

[Working Safely with Toxic Powders](#)

[Working Safely with Nanomaterials](#)

These information documents are also posted in the SMIF characterization labs.

Powders of any kind are not allowed inside the SMIF cleanroom

7.3 Sources of Chemical Information

7.3.1. SDS (Safety Data Sheet)

The **Safety Data Sheet (SDS)** is a convenient, condensed source for information on the properties of any chemical. The **SDS** 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 should read the SDS for any chemical you use in SMIF.** Safety data sheets for all chemicals approved for use in SMIF can easily be found by typing the chemical name and “SDS” (or the older term “MSDS”) into an internet search engine.

7.3.2. SOPs (Standard Operating Procedures)

The chemical page on the SMIF web site (<https://smif.pratt.duke.edu/chemicals>) contains links to the appropriate SOP for each chemical that is listed. You should use this page and the SOPs for information on the proper use, storage, and disposal of chemicals used in SMIF.

7.4 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 SDS or other references. This is not intended to be a complete reference on Toxicology or Chemical Safety.

7.4.1 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.
- **Oxidizers** are chemicals that make combustible materials and liquids burn easier and with more intensity. For this reason, oxidizers should never come in contact with solvents or acetic acid. Examples of oxidizers in SMIF include Hydrogen Peroxide, Nitric Acid, and “Piranha” solution.
- **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).

7.4.2 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.

7.4.3 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.

7.4.4 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.)

7.4.5 Health Hazards

- **Carcinogen**- A substance producing or inciting cancerous growth.
- **Mutagen**- Capable of inducing mutations.
- **Teratogen**- A substance causing damage or death to a fetus.
- **Acute Toxicity** – Some chemicals, such as hydrofluoric acid and osmium tetroxide, can be fatal by skin contact or inhalation. Others, such as propylene oxide, are also considered acutely toxic but are described as “toxic” or “harmful”
- **Skin Corrosion and Eye Damage**- Some chemicals, such as nitric, sulfuric, and hydrofluoric acids can cause severe damage to the skin and mucous membranes

7.5 Chemical Use

Users must take the SMIF Chemical Safety class and be trained and certified on chemical hood usage before using the chemical hoods on their own. Chemicals are to be used ONLY in the exhausted chemical hoods.

7.5.1 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 SDS 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 SDS
- 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.

7.5.2 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. The following chemical hazard labels are provided by SMIF:



Corrosive – may cause severe skin burns



Oxidizer – may cause or intensify fire



Causes serious eye or skin irritation



Flammable



Health Hazard – may cause cancer, fertility issues, or organ damage

Fatal if inhaled, swallowed, or in contact with skin

- Users are NOT allowed to use chemicals that have someone else's name on them without the permission of the chemical owner.
 - SMIF supplies para-film and a marker that can be used to seal personal chemicals and protect them from unauthorized usage
- **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.

7.5.3 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.

7.5.4 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.

Keep solvents and oxidizers separated

- Oxidizers are to be used **ONLY** in the white color polypropylene chemical hoods
- Solvents are to be used **ONLY** in the stainless steel chemical hoods.

Acetic Acid is an exception – it should be used in a Solvent Hood and stored in a Solvent Cabinet

- Because Acetic acid is incompatible with oxidizers and many acids, it should be stored in a solvent cabinet and used in a solvent hood

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 7.7).

7.5.5 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.

7.5.6 Personal Protective Equipment

The SOP for a particular chemical will instruct you on the personal protective equipment you need to wear when using that chemical. In general, when using chemicals, users must **minimally** wear the following items:



Clean Room Laboratory

For Acid Use	For Solvent or Spin Coating Use
<p>In addition to the standard clean room attire, the following items are to be worn:</p> <ul style="list-style-type: none"> • Chemical Apron • Safety Goggles • Chemical Face Shield • Chemical Gloves 	<p>In addition to the standard clean room attire (<i>including nitrile gloves</i>), the following items are to be worn:</p> <ul style="list-style-type: none"> • Safety Goggles • Users should “double” glove when using chlorinated solvents.

Offline/Sample Preparation Laboratory

For Acid and High Toxic Chemical Use (see 7.6.7)	For Solvent Use ONLY
<ul style="list-style-type: none"> • Chemical Apron • Safety Goggles • Chemical Face Shield • Chemical Gloves 	<ul style="list-style-type: none"> • Safety Goggles • Lab gloves (nitrile). Users should “double” glove when using chlorinated solvents • Lab coat (optional)

- Always first check chemical gloves for holes or damage
 - If damaged, dispose of the gloves and get a new pair
- Never purposefully touch a chemical even while wearing the chemical gloves. If a glove does come in contact with a chemical
 - Remove the exposed glove and dispose of it.
 - Get a new pair of gloves
- 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.

7.5.7 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. SMIF annually inventories all chemicals being stored in the chemical cabinets, and following notification of users, will properly dispose of any chemicals no longer in use.

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.

7.5.8 Pregnancy

If you are planning a pregnancy, you are strongly encouraged to use the “Reproductive Health” resources through Duke University’s Occupational and Environmental Safety Office. This information can be found at the following web site:

<https://www.safety.duke.edu/occupational-hygiene-safety/reproductive-health>

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 7.6.5.)

7.5.9 Buddy System for Chemical Usage

A buddy system applies to the use of wet chemicals in any of the SMIF facilities. These rules apply for chemical usage when SMIF staff is not present, which includes weekends and holidays, and Mon-Friday from 5pm to 12 midnight. ***Chemical usage of any kind is not allowed between the hours of 12 midnight and 7am.***

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.

Using Chemicals in the Cleanroom

- Another person must be in the cleanroom and aware that you are using chemicals
- Exception: You may use the hoods in the Lithography area (spin coating, develop, and solvents) without a buddy present

Using Chemicals in the Sample Prep Lab

- Another person must be in the sample prep lab and aware that you are using chemicals

7.5.10 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 goggles and full-face shield when using acids and goggles when using solvents and lithography chemicals.

7.5.11 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 at all of the Acid Hoods for application to HF skin exposures. This should be applied promptly, but is not a substitute for prompt medical attention.

7.5.12 Chemical Accidents

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

Spills Contained Inside a Chemical Hood

- Avoid breathing vapors from the spill and leave the immediate area of the chemical hood
- Alert people in the immediate area of the spill

- Notify SMIF immediately by calling emergency numbers posted near the phone
- Wait for instructions from SMIF or for SMIF personnel to arrive to complete the clean-up of the affected area.

Spills Outside of a Chemical Hood

- Attend to injured or contaminated persons and remove them from exposure
- Press the closest manual alarm button (blue box) and evacuate the lab
- 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.

7.5.13 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 N₂ blowguns near open chemicals
- Don't use plastic beakers on hot plates
- Clean up after yourself and be careful

7.6 Specific Chemical Hazards

7.6.1 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.

7.6.2 Hydrofluoric Acid

Hydrofluoric acid, HF, presents a significant hazard for personal injury. It is a high risk category 1 chemical because it can be fatal in contact with skin. Over time, the fluorine ions in HF can seep into your skin and into your bloodstream, causing fluoride poisoning. Fluoride poisoning can cause cardiac events (e.g. heart attacks) which can potentially be fatal.

HF is used in the lab directly out of the bottle (49% concentration), diluted with water, 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 typically used in the laboratory, an HF “burn” may be 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 and bone damage as well as fluoride poisoning, and **at some point, if left untreated, you may die.**

HF Potential Hazards

- Fluoride poisoning leading to cardiac arrest and potentially death.
- Liquid HF is one of the strongest and most corrosive acids. It can be irritating to the skin, eyes, and respiratory tract. Contact with exposed body parts can cause painful burns and even death.
- In high concentrations (more than 50%), HF usually causes immediate burns that are extremely painful and slow to heal.
- In lower concentrations, exposure may not be apparent for several hours, but can still cause burns and further damage if not washed off.
- HF causes such severe burns because it penetrates beneath the skin and dissociates into hydrogen and fluoride ions. When fluoride ions bind with calcium in the body, it can result in tissue destruction, decalcification of bone, cardiac arrhythmia, and liver and kidney damage.
- The OSHA Permissible Exposure Limit for hydrogen fluoride is 3 ppm. The American Conference of Governmental Industrial Hygienists recommends a ceiling (instantaneous) limit of 2 ppm and an 8-hour limit of 0.5 ppm.

HF Exposure Prevention

- HF and HF containing solutions must only be used in the Acid Hoods while wearing the full acid chemical protection gear as described in Section 6.5.6.

- 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. ***Always carefully inspect chemical gloves before usage!***
- 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 First Aid and Medical Treatment

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. Immediately 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. **Make sure that a copy of the HF Medical Treatment and First Aid Guidelines (see bold note below) are available to medical personnel.**

Complete First Aid Guidelines for Treating HF exposures can be found as an Appendix to this manual, and printed copies are available at all SMIF Acid Hoods and the SMIF Emergency Response Station.

7.6.3 Sulfuric Acid

Sulfuric acid is particularly hazardous because it is a carcinogen. It breaks through the chemical gloves that SMIF uses more readily than other chemicals. Per SMIF policy (section 7.5.6), chemical gloves that have come into contact with Sulfuric acid should be disposed of and never reused.

7.6.4 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. Piranha solutions are only to be used in the Acid Hoods while wearing the full acid chemical protection gear as described in Section 7.5.6. The Sulfuric Acid in the Piranha solution breaks through the chemical gloves that SMIF uses more readily than other chemicals. Per SMIF policy (section 7.5.6), chemical gloves that have come into contact with Sulfuric acid should be disposed of and never reused. Ensure that the used Piranha solution has stopped "fizzing" before pouring the waste into the acid hood drain. Piranha solution must never be stored, so make only the amount you need.

7.6.5 Nitric Acid

Nitric Acid is considered particularly hazardous because of its oxidizing properties. It must never be mixed with organic materials such as solvents or acetic acid.

7.6.6 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. Chlorinated solvents also typically penetrate laboratory gloves easily. For example, trichloroethylene (TCE) will penetrate a nitrile glove in less than 5 minutes. As such, users of chlorinated solvents should take extra caution when handling these chemicals, and should double glove (wear two pairs of the nitrile lab gloves).

7.6.7 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 SDS for it. One family of chemicals, the **glycol ethers**, commonly used in older 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.

7.6.8 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.

7.6.9 Highly Toxic Compounds used in EM Sample Preparation

Chemical compounds used commonly in sample preparation for electron microscopy can be highly toxic, and should only be used in the proper chemical hood with the appropriate personal protective gear as described in section 7.5.6.

- **Osmium Tetroxide** is considered a high risk chemical because it is fatal by inhalation and skin contact, category 1. It affects the central nervous system, eyes, male reproductive system, and kidneys. Since it is high-risk, a chemical-specific SOP approved by OESO is attached to this manual as an appendix. This must be reviewed by anyone working with Osmium Tetroxide. Waste containing Osmium Tetroxide cannot be mixed with other wastes and the original manufacturer's container must be submitted as waste.
- **Sodium Cacodylate** is a known carcinogen (contains arsenic) and can also affect the kidneys, GI tract, heart, brain, skin, bone marrow, nerves, and liver
- **Uranyl Acetate** is highly toxic and radioactive
- **Propylene Oxide** is a potential carcinogen, and can affect the reproductive system and damage DNA and chromosomes. It also affects the skin, eyes, and respiration system.
- **Lead Citrate** affects the reproductive system (fertility or an unborn child) and may cause damage to organs

7.7 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.

The SOP for a particular chemical will instruct you on how to properly dispose of the used chemical. 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.

- Acetic acid containing chemicals: Use the designated waste container located in the solvent hood.
- 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. Waste bags containing solvent waste are placed in the designated contaminated solvent waste container. Waste bags containing acid waste should be left in the back corner of the acid hoods. SMIF staff will regularly pick up the chemical waste bags for transport to OESO for proper disposal. Chemical waste bags 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.

8.0 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.

8.1 Hazardous Gases Used

8.1.1 Pyrophoric Gases

Pyrophoric gases are defined as gases that will ignite spontaneously in air at temperatures of 130 F or below. **Silane (SiH₄)**, 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 %.

8.1.2 Corrosive Gases

Corrosive gases used in the SMIF facility include the chlorine-based gases, **Chlorine (Cl₂)**, **Boron Trichloride (BCl₃)**, and **Silicon Tetrachloride (SiCl₄)**. 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.

8.1.3 Flammable Gases

Flammable gases used in the SMIF facility include **Hydrogen (H₂)** and **Methane (CH₄)**. These gases are used in reactive ion etch and/or annealing systems and are confined in exhausted cabinets.

8.1.4 Cryogenic Gases (Liquid Nitrogen)

Most people do not think of **Nitrogen** as a hazardous gas. Liquid nitrogen expands 695 times in volume, and can quickly displace oxygen, potentially resulting in an oxygen deficient environment. More people die of asphyxiation by nitrogen than by any of the “toxic” gases discussed here.

LN₂ vapors can also rapidly freeze skin tissue and eye fluid, potentially resulting in cold burns, frostbite, and permanent eye damage



We use liquid nitrogen for many things in the laboratory. Liquid nitrogen dewars are used to fill cold traps for the Cryo-EM microscopes and 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.

8.1.5 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.

9.0 X-Ray Safety

All X-ray generating SMIF equipment are equipped with safety interlocks that prevent X-Ray exposure during operation. Users should never attempt to defeat these interlocks or perform maintenance or repair of this equipment. X-Ray based instruments use in SMIF are:

- Micro CT (MICROCT1)
- Small Angle X-Ray Scattering (SAXS2)
- X-Ray Diffractometer (XRD2)
- X-Ray Photospectrometer (XPS1)

10.0 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

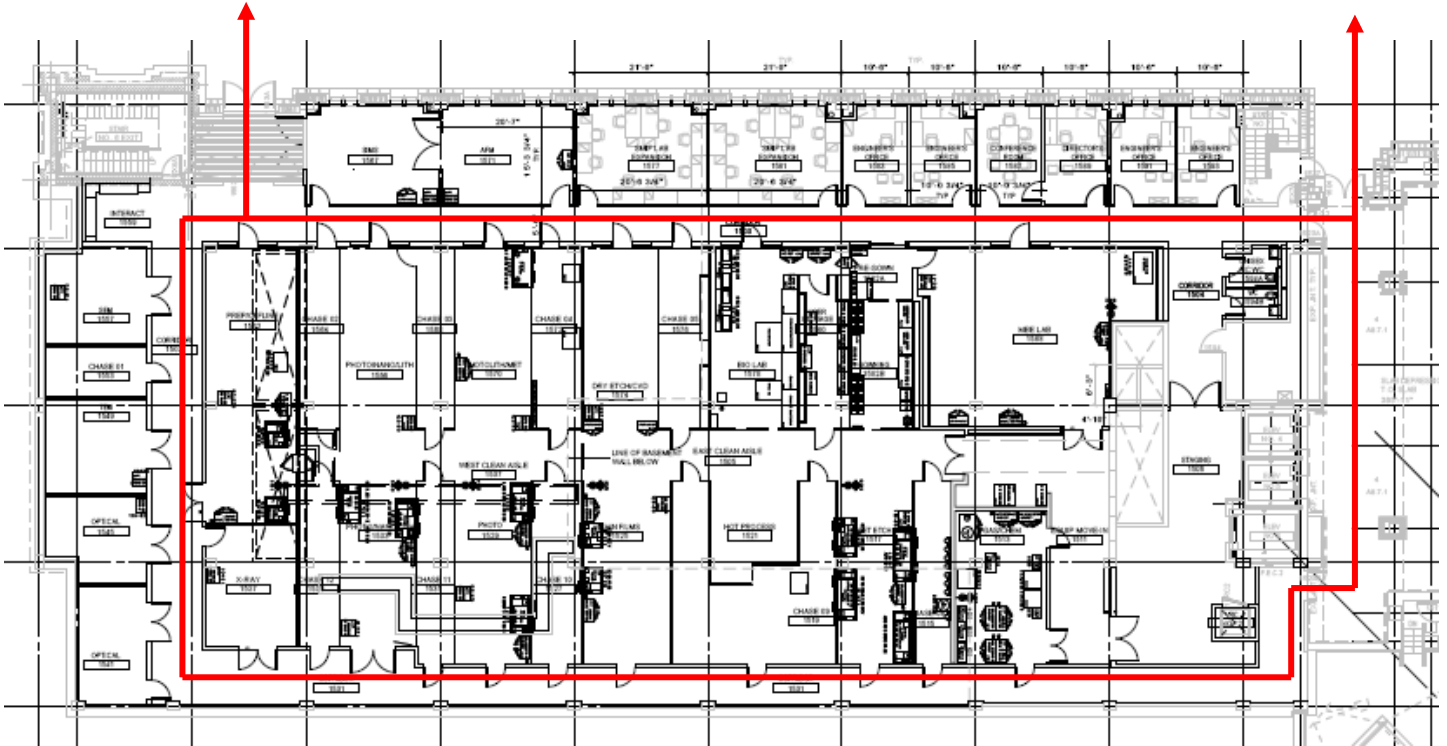
C = Close doors to prevent spread of fire

E = Evacuate (only emergency personnel should attempt to extinguish a fire in SMIF due to the presence of various flammable gasses and chemicals)

When evacuating the building, you should go to the approved emergency assembly point (EAP). The EAP for SMIF is the courtyard area between the Fitzpatrick/CEIMAS and Hudson Hall buildings.

Evacuation Routes out of the SMIF Facility

First Floor Labs (Fitzpatrick 1500 wing)



SMIF Soft Materials Lab (Fitzpatrick B606)

