Chemical Hygiene Plan
for the MRL TEMPO Facility

March 22, 2013 Update and Revision

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Welcome to the MRL TEMPO Facility

March 22, 2013

Welcome to the MRL Thermal, Electronic/Elemental, Magnetic, Porosity, and Optical (TEMPO) Facility! This is a great place to work. Many many people, from summer interns to five year Graduate Students, have been able to do outstanding research while still enjoying their time here. We are a very well equipped lab with a rich suite of instruments and an excellent array of equipment for synthesis. We may also have a lot of people working in a relatively small facility. We all have to be respectful of each other's needs. Everyone working here must act in a professional, safe, and environmentally responsible fashion.

This Chemical Hygiene Plan (CHP) is intended as a resource for people working in the TEMPO Facility. It contains both rules for laboratory conduct and information about understanding the hazards inherent in the lab work we do. It has a section with general lab safety information that everyone working in the lab should understand before starting to work in the lab; as well as more specific information that will be helpful for people doing specific operations. The most basic information and lab conduct rules are in the Executive Summary.

Even though this is called a Chemical Hygiene Plan, its scope is not limited to chemical hazards. We have tried to include every topic of lab safety that is likely to come up in normal lab use. It also includes a number of housekeeping issues.

While we are officially a Materials Lab this facility really is a chemistry lab. Many of the people working here are new to chemistry with backgrounds in Physics, Engineering, or Materials. People with a chemistry background need to help those without one to work productively and safely. People new to chemistry need to make sure they are working safely and efficiently in their own work, and they need to be aware of any hazards from their neighbor's work.

Everyone here needs to be sure they work in the safest possible manner, and we all need to make sure we all follow all the many laws and regulations about safe work practices. Nearly everyone working in this lab qualifies in some way as an employee of the University of California, as such we all have obligations to work safely and the State has obligations to insure that we are properly trained and that such training is documented.

Safety training begins with the EH&S Laboratory Safety class. Everyone working in the lab is required to take this course BEFORE beginning lab work. Most people will need to take the class in person. Additional training builds on the EH&S class. See the section on Required Training.

The second part of the required safety training is this Chemical Hygiene Plan (CHP). Everyone who wishes to work in the TEMPO Facility needs to read this Chemical Hygiene Plan. After reading the CHP, people need to document that they have read it. This should be documented by signing a log sheet in my office, MRL room 2066F.

The regulations about creating a CHP assume a production lab with unchanging procedures. Our lab is just the opposite; we constantly change our procedures, so this document can never fully cover the safety issues in our lab. Rather it is a foundation upon which each lab researcher can begin to understand how to do their work safely and responsibly.

It is the responsibility of each and every person working in this lab to do the thought, the inquiry, and the literature research required to understand the hazards of their experimental work before they begin. The actual preparation will vary depending on what the project will be, but will certainly include studying the chemical hazards of the materials, the physical hazards of the process, any appropriate Standard Operating Procedures (SOPs), and speaking with people who have done similar work.

One of the key SOPs in this CHP is Preparing For A New Project. It includes the perspective of two experienced lab researchers about how they prepare for a new experiment. It is complimented by an SOP describing where to get more detailed safety information.

Lab Access

The lab has electronic door access through your UCSB ID card. For people only using the instruments, your card access will be restricted to 8 AM to 5 PM. Until the electronic access is fully operational, the doors will automatically open from 8 AM to 5 PM, Mon-Fri. People working in the lab when the doors lock do not have to leave. The lab doors are not to be propped open after hours. Theft has been an issue at the MRL and we want the lab to be a safe and secure place to work.

People who use the lab's analytical instruments but do not do other work in the lab, should plan their work for business hours. Lab access will not be granted to anyone until they have completed the required safety training.

Other Issues

We try as hard as we can to insure that the lab is fully functional, that it is as user friendly as possible, and that it is as safe as possible. To accomplish this we need your help.

If you see any kind of safety problem, that we are low or out of some necessary supply, or that some equipment is not working right please drop me an e-mail describing the problem. E-mail is the best way to keep me up to date with housekeeping issues.

Tell me right away if there is any imminent hazard or any kind of safety problem.

Safety glasses must be worn at all times by all people in the lab. This does specifically apply to people who are only walking through or are only using a computer.

All samples and repackaged reagents must have a label showing the owner's name in a unique and recognizable form and must show the essential composition of the contents.

Individuals may be assigned limited individual drawer space, but bench top space is much too limited for anyone to have exclusive bench top space. We all share bench space and must make every effort to keep our stuff out of the other people's way except when we are actively using it.

Nobody wants to have to clean up someone else's mess before they can begin to work, so everyone needs to put their samples and supplies away every day after working in the lab so that there is a clear work area for the next person. Never leave samples, lab supply, personal effects, glassware, books or papers out in the lab except when you are actually using them. Wash and put away your glassware every day. Put your reagents back in the proper cabinet at the end of every workday.

Chemical storage space is very limited. Before purchasing any new chemical be sure to check if any of the required reagent is already available in the lab. We already have a huge inventory of reagents waiting to be used. Each chemical storage cabinet should have an inventory taped to the door of the cabinet. Besides conserving room, this will save you a lot of time and money. If you have a reagent that someone else needs please share it with them.

From time to time we have Lab Clean up sessions. These may occur when the lab has become particularly messy, before an inspection or a tour, or at the end of the summer intern season. Everyone working in the lab needs to participate.

UCSB is in a drought prone area and water is always in short supply. Always use the minimum amount of water required for a job. Never leave water running when you are not using it.

Energy is limited and expensive. Always try to save energy in the lab. This means turning off lights in the fume hoods when not working there, turning off room lights when you leave, and most important close fume hood sashes when not working at the hood. The air flow required to make a fume hood safe takes an enormous amount of energy, and even more energy is used to heat or cool the room air that gets sucked through the hood. Closing the fume hood sash reduces the air flow and saves a LOT of energy.

Anyone with questions about anything should see me. My office is MRL 2066F and my phone is x7925, my e-mail is jdoyle@mrl.ucsb.edu.

Sincerely,

Joe Doyle

Executive Summary for the TEMPO Facility Chemical Hygiene Plan

Joe Doyle
March 22, 2013

These safety and housekeeping rules are intended for real use. This is not intended to be a showpiece document that no one actually follows. Some of these descriptions are short, while others have an SOP dedicated to the topic later in the CHP. There is useful information about all topics at the campus EH&S web page.

Anyone who does not understand these issues must seek out clarification before doing any relevant lab work.

Anyone who observes a situation that they consider to be unsafe, or possibly unsafe, must to contact MRL staff and report the issue. This may be done anonymously.

1) Training Requirements

It is the responsibility of each and every person working in the lab to work in a safe and environmentally sound way. No one is allowed to work in the lab until they have they have been trained in safe laboratory practice, have demonstrated an understanding of this training, and have been approved by the lab's Development Engineer, Joe Doyle.

Safety training begins with the EH&S Laboratory Safety class. Everyone working in the lab is required to take this course BEFORE beginning lab work.

The course is available in several formats. The class is given in person at the start of each quarter. In summer there is a special in person class just for interns. In the fall the class is provided in person for incoming graduate students at several science departments and at the College of Engineering. Finally there is an online version of the course available for undergraduates and Post Docs.

People whose work in the lab is limited to the use of analytical instruments, for which specific training is offered, may complete any version of the EH&S lab safety class and document the instrument training. The online undergraduate EH&S Lab Safety class is fully acceptable.

People who work At Large in the lab need much more extensive training. Working At Large in the lab includes anyone who:

has a lab door key or 24/7 card access;
uses a furnace or oven;
uses a fume hood; or
does any lab work beyond the use of analytical instruments.

To get the Facility's Development Engineer's approval, At Large lab researchers must complete the following before doing any work in the lab:

Take the EH&S Lab Safety Class in person,

Read the TEMPO Chemical Hygiene Plan,
Pass the TEMPO Lab Safety Test,
Complete the Hazard Assessment with your PI,
Complete the MRL Participant Form and
Turn in all documentation to Sylvia.

Since the In Person EH&S Lab Safety Class is only offered quarterly, lab users may get temporary lab access until the next in-person class by taking the Undergraduate version of the Online Lab Safety class from EH&S. Such people are still required to attend the next available in-person Lab Safety class.

The in-person quarterly training schedule is announced by e-mail one to two weeks before the class and is posted online. The Online training is anytime available at: http://www.lifesci.ucsb.edu/support/safety/lab_safety/undergrad/undergrad.php Everyone should do the Undergraduate version even if they have an advanced degree.

Concurrent with all the above, new arrivals at MRL should contact Sylvia Vogel for a copy of the MRL Participant form. This form directs participants to the required safety training beyond laboratory issues including fire, earthquake, ergonomics and more.

2) Know The Hazards Of Your Work

All of the above is just the beginning of laboratory safety training. Everyone working in the lab must do the appropriate inquiry, literature research, and thought to insure that the lab work they do is safe.

Your supervisor, the MRL, and the campus will do everything possible to insure your safety and each can provide resources to help you understand and mitigate any hazards; but ultimately each individual has a responsibility to do all the effort required to be sure their work is safe for them and for everyone nearby. This starts with studying and considering the hazards of the research.

This may be as simple as reading an SOP, reading a few papers, and talking to other people in the lab. But usually more work is required. Studying the hazards of the chemicals to be used will probably be required as will reviewing any physical or electrical hazards and considering what personal protective equipment may be appropriate.

Vast amounts of information is available, we suggest starting with TEMPO CHP and the campus EH&S web page http://www.ehs.ucsb.edu/units/labsfty/labsafety.html.

You may be undertaking a project your lab has never tried before. Or you may even be trying a procedure no one has ever done before. There may not be a set procedure for your research.

There is no set procedure to research the possible hazards of a new process. The investigation depends on the nature of the project. Researchers starting new projects must be imaginative and diligent.

3) Safety Glasses

Everyone in the TEMPO Facility must wear safety glasses at all times, even if just using an analytical instrument or working at a computer.

Every lab worker must own a pair of safety glasses that fit well and are comfortable to wear. Loaner safety glasses are available for visitors.

Safety glasses for people working in a chemistry lab have large front lenses and side guards. Uncomfortable goggles that fog up should be thrown away with prejudice.

Reading glasses are not safety glasses and are not a suitable substitute.

A full face shield is available for use by anyone who feels it is appropriate.

4) Lab Coats and Clothing

Lab researchers must be aware of their clothing as protection from laboratory chemicals, heat, sharps, and other hazards but at the same time be aware that some clothing can exasperate certain hazards.

Closed toe shoes are always required for anyone in the lab at any time.

In general, people doing chemical synthesis or working with furnaces should avoid poly clothing in favor or cotton or wool. Poly clothing can catch fire and severely burn the person wearing it. This was a significant cause of a lab fatality in 2008.

In general, when doing chemical synthesis or working with furnaces wearing, a lab coat is desirable but not required while working in the TEMPO lab.

For the routine work of the TEMPO Facility, a 100% cotton lab coat will usually be the best choice. Researchers working with strong acids or bases should prefer a poly/cotton blend lab coat. The Physics Stockroom now carries flame retardant treated 100% cotton lab coats.

100% Cotton lab coats may be ordered from Fisher Scientific in most sizes. Search their web page http://www.fishersci.com with item 01-369-2C then choose the right size. The UC price is about $17.

Researchers working with extremely flammable material or pyrophoric liquids should consider Nomex lab coats because they offer the most fire protection.

A resin apron is available for use by anyone who feels it is appropriate.

5) Gloves

Some people wear gloves to protect their hands from hazardous chemicals. These people must make sure that the material their gloves are made from is resistant to the hazardous chemicals they are using. There are a number of choices for glove material and their resistance to various chemicals is compiled on a table available at http://www.ehs.ucsb.edu/units/labsfty/labrsc/lsglove.htm

Nitrile gloves are suitable for most common chemicals and are available in all sizes in a drawer on the North side of room 1033. Users should please let Joe Doyle know when any size of glove is running low.

People whose work demands gloves made of other material should plan on purchasing the optimum gloves for their work.

For most applications thin gloves are superior to heavy gloves because they offer better dexterity. They offer less protection from chemical attack but the improved dexterity usually makes them safer. Should a thin glove be exposed to a hazardous chemical the user should assume that the chemical will eventually penetrate the glove and replace the gloves they are wearing.

Some materials are so toxic that only thick gloves (maybe with a thin glove liner!) are acceptable. There was a case about 10 years ago where a woman researcher died when one drop of an organo-mercury compound fell on her gloved hand! Thick gloves are available in room 1033 under the sink.

People handling hazardous materials must discard their gloves before touching anything in the lab but their apparatus. This includes door handles, computer keyboards, and the telephone. People wandering around the lab and the building wearing lab gloves make me very nervous because they look like they are spreading hazardous materials onto every door handle.

Some people wear gloves to protect their samples from skin oils and salts. These people should not touch door handles keyboards etcetera with gloves on or they will get other people's skin oils onto their gloves and indirectly onto their samples. Regardless of why you are wearing the gloves, please do not touch anything but the instrument and samples with your gloves on.

Gloves for protection from heat and cold are available in a drawer in room 1033.

6) Chemical Hazards

It is every lab worker's responsibility to learn and understand the hazards of the chemicals they use before they start using those chemicals. Some few are harmless, some are only harmful if ingested, and some are deadly. Some chemicals are mostly harmless by themselves but very dangerous in combination with certain other chemicals.

Two good sources of chemical hazard information are the Merck Index and Sax's Dangerous Properties of Industrial Materials. Both are available in MRL room 2066F.

The Material Safety Data Sheet (MSDS) was created to be a one stop source of chemical hazard information. They are very useful for mixtures, like WD40, and have value for reagents. They are widely available on line. Alas they can be full of bureau speak, are not as succinct as we would like, and make everything sound extremely hazardous. Laboratory Chemical Safety Summaries (LCSS) are much better, but not available for as many materials.

The MRL Safety page,
http://www.mrl.ucsb.edu/general-safety-guidelines has links to MSDS and LCSS sources.

7) Carcinogenic, Pyrophoric, and Extremely Hazardous Materials

Most of the chemicals we use in the TEMPO facility are hazardous if used incorrectly, but not extremely hazardous. Extremely hazardous materials require extremely careful handling, extra training, and special approval.

There are numerous federal and state regulations regarding the use of select carcinogens, reproductive toxins, and highly acute toxins, plus there is heightened scrutiny of all work with pyrophoric materials after a fatal accident in a UC lab in December of 2008.

Pyrophoric materials are not to be used in the lab until the researcher has studied the safe handling of these materials and obtained both the specific permission of their faculty advisor and the permission of the Lab Supervisor (Joe Doyle).

Additional information is available at:

http://www.ehs.ucsb.edu/units/labsfty/labrsc/chemistry/lschem.htm
http://www.ehs.ucsb.edu/units/labsfty/labrsc/factsheets/lsfacsheets.htm

8) Chemical Storage

Because of the mixed user base, many people in the TEMPO Lab arrive without understanding the basics of chemical storage. We have a surprising history of lab workers storing chemicals next to each other that would react violently if mixed.

An inspection in April of 2005 found a chemical storage cabinet in 1033 with a 4 liter bottle of concentrated nitric acid, next to ammonium hydroxide, next to oxalic acid, next to hydrofluoric acid, next to sodium hydroxide pellets. In a lab like ours people are expected to understand why chemicals are separated and what might happen if they accidentally mixed.

Incompatible chemicals must never be stored in the same cabinet. Oxidizers may not be stored with fuel. Acids may not be stored with bases. Each TEMPO lab user must study the attached handout on chemical storage. All users must pay attention to what they as individuals place in any cabinet. We cannot have materials in the same cabinet that would react energetically with each other.

Fire fighters like to use the term "Corrosive" for chemicals. It is a meaningless term for us because it applies to both acids and bases.

We have a huge inventory of chemicals in the lab. There may well be the ideal reagent for your project available right now free of charge. Lab users must check existing supply before ordering new chemicals. Chemical storage space is scarce, don't fill it up needlessly.

Each group has designated reagent storage under the group fume hood. There is more chemical storage, available to everyone in the lab, under the middle bench in 1033 and in the chemical storage room. There is a cabinet for Oxidizers on room 1023 below the east bench. Each group needs to make sure that their chemical storage cabinets are accurately labeled and that there is an up to date inventory of the chemicals posted.

Everyone working here must label all of their reagents with their name and the date opened. Some chemicals have a limited safe shelf life and must be disposed of after some period of time. For example, most ethers must be disposed within 12 months from when first opened.

Individuals may keep inert powder reagents in their personal drawers, but it’s better for research groups to combine such storage in a cabinet. Aggressive, highly toxic, carcinogenic, or liquid materials must be kept in approved chemical storage cabinets.

Chemicals need to be put away each day. This means that users put the reagent bottles away before going home every day.

9) Labeling

Every single container containing samples or repackaged reagents must be labeled with a unique and recognizable version of the owner's name. Initials are not acceptable. Sample bottles are often too small to show everything but, at a minimum, containers must also show key information about the contents so that any hazards are revealed should the owner be unavailable.

Gas cylinders must also be labeled with the name of the person who ordered them.

10) Chemical Disposal

With very few exceptions, waste chemicals may not be put down the drain or into the lab trash. Waste chemicals must be disposed of per UCSB policy and state and federal law. We have a waste chemical area in the walk in fume hood in room 1033. All waste chemicals must be labeled with the owner's name as well as a complete and accurate description of what is inside the bottle using the labels from EH&S.

There should be peel and stick EH&S waste labels in a plastic sleeve on the South side of the walk in fume hood in room 1033.

Chemical waste must be segregated in the same way that new chemicals are stored. The most common chemical waste accident at UCSB is mixing nitric acid (an oxidizer) with organic waste (even acetic acid) in an "Acid Waste" bottle and generating CO_2 until the pressure builds and explodes the bottle.

Whenever the waste chemical area is about 75% full, one person from each of the three research groups in the TEMPO Lab must get together and process the waste for EH&S pickup. I have the forms and can provide more detail. The waste must be picked up no longer than 9 months after the date on the sticker.

Campus UCSB EHS has a web page about Hazardous Waste that is worth reading. It is at: http://www.ehs.ucsb.edu/units/hw/hw.html

11) Sharps Disposal

Sharps material like broken glass, razor blades, or hypodermic needles; may not be placed in the regular lab trash as this could injure the custodian. Any uncontaminated sharps waste, other than glass, such as razor blades or syringe needles, needs to go into a sealed container before going into the red-lidded brown-bodied bins outside. If there is a biohazard sticker on the container, it must be defaced to show that the contents are not infectious or contaminated with hazardous materials.

For glass waste, there are two galvanized cans in the labs specifically for nonrecyclable lab glass like Pyrex or flint glass, broken or not. Glass does NOT need to go into secondary containment before being disposed of in the red-lidded, brown-bodied bins outside. When full, lab researchers need to take the broken glass and the sharp waste out to the new red-lidded brown-bodied bins in the trash area. The bins must remained locked so you may get a key from XXX or from Joe Doyle. No sharps or broken glass can go in the regular waste anymore.

12) Furnaces and Ovens

Furnaces can be very hot. The amount of energy in an 1100 degree furnace is enormous. Besides the direct burn hazard, a system failure or sudden sample vaporization might send super-HOT embers flying fast in all directions! Anyone working around hot furnaces must protect their eyes by wearing safety glasses. Even people just walking past a hot furnace should be cautious. Certainly someone standing near someone else manipulating samples in a hot furnace should wear safety glasses and be wary.

Furnace insulation materials have changed over the years so that it is not possible to say what they are without digging up the specs on a particular furnace. While I do not think we have any asbestos in any of our furnaces, I am sure that what we do have should be kept out of our lungs. Even harmless insulating materials may cause one's skin to itch on contact. Nearly all furnace insulation becomes friable over time and sheds dust with little provocation. Every furnace user needs to minimize the dust created when they use a furnace and needs to clean up any dust generated each time they use a furnace. The best way is to use a wet paper towel to wipe up any dust. The wet paper towel may be put in the regular lab trash.

Anyone opening an oven anytime must wear safety glasses. Ovens may contain pressure vessels, affectionately known as bombs and these may vent dramatically spewing hot liquid.

Insulated gloves and tongs are available for handling hot materials.

13) Hydrothermal and Other Pressure/Digestion Bombs

Many people in this lab use pressure vessels for hydrothermal synthesis. The ones we use are made by the Parr Instrument Company. These pressure vessels are also called bombs. Everyone using bombs must study how to use them safely before use. Parr's Operating Instructions for Parr Acid Digestion Bombs and their Safety in the Operation of Laboratory Reactors and Pressure Vessels are a great place to start. In particular they should not be over filled and the chemistry inside must not make explosives, as would might happen with nitric acid and an organic.

These bombs must be visually inspected before each use. Any pressure vessel with a crack or other flaw in the metal must be discarded and never used again.

Everyone in the lab must be aware that these bombs have over-pressure vents that will squirt the hot contents vigorously if the vent activates. A borderline vent may go with just a little mechanical agitation such as removing them from a hot oven. Consequently every time anyone removes any kind of bomb from any kind of hot oven they must wear safety glasses and anyone else in the area should wear safety glasses too.

Of course hot work gloves are also recommended.

15) Earthquakes

There will be a big earthquake here at UCSB. The only question is when.

During an earthquake people should try to stand in doorframes until the earthquake is done and only then evacuate the building.

All storage, especially that of chemicals and glass, must to be done so that the materials will not fall and break in a large earthquake. All gas cylinders need to be secured with welded ling metal chain so they do not fall in a quake.

16) Compressed Gas

When gas is carried inside tubing, the tubing must be compatible with the particular gas and the planned pressure. Air, nitrogen, and inert gasses should be in polyethylene tubing. 1/4" polyethylene can easily carry over 100 PSI. All flammable gases and oxygen must be in metal tubing. Many people are surprised to learn that pure oxygen makes most plastics highly flammable. Teflon tubing is often a great choice for exotic gasses, but it is more expensive than other materials. Except for very low pressures and as a short transition piece, Tygon (soft PVC) is never a good choice for gas.

The Uniform Fire Code has stringent regulations for any gas as or more toxic than carbon monoxide. Compliance is expensive and time consuming. Experiments should be designed to avoid triggering this requirement. If toxic gas is unavoidable, see Joe Doyle.

Gas cylinders must be secured in place with welded link chain so that they cannot fall over in an earthquake.

Each person who brings a gas cylinder into the lab must put their name on it like they would for any other reagent.

When a gas cylinder is no longer needed or is empty, the person who ordered it must get it out of the lab promptly.

17) Drawer Space

The drawer space allocation in the MRL TEMPO Lab is a constant problem. Vast amounts of prime real estate are wasted with old samples that no one gives a damn about. Existing lab users may try to acquire much more drawer space than is fair to the other lab users. To make drawer and cabinet space allocation fair for all lab users, these standards apply:

- Cabinets may be assigned to research groups for group owned equipment, supplies, and powder reagents but not individual lab users.

•Active Post Docs and Graduate Students may have up to 3 drawers.
•Visitors who are here from 3 to 9 months may have up to two drawers.
•Interns and visitors of up to 3 months may have one drawer.
•Drawer allocation is per person not per research group.
•Each drawer occupant must show their name on the drawer face.
•Drawer space is a precious and limited resource. It is to help active researchers do their work, it is not for long term storage or archiving.

The above are upper limits not entitlements.

18) Completing Work at the Lab

On completion of your work in the TEMPO Lab at UCSB you will need to make way for the next person and put your gear back into circulation. Be sure to do the following:

-Let me know when you are leaving a few weeks before you are gone.

-Dispose of most of your samples. A select few may be archived. Consider whether anybody will really ever want to look at them again. Almost none of the current old sample archive has ever been accessed by anybody.

-For those few samples to be archived, place them in the smallest cardboard box possible and write your name prominently on the outside along with basic info about what the samples are. In that same box, place a spreadsheet showing specifically what the samples are. The box should then go on a shelf in the chemical storage room.

-Also have a big lettered note on top of the box identifying whether there are any especially hazardous materials in the box or not.

-Empty out any lab drawers you have been using. Any equipment or glassware that has been assigned to you should be put back into circulation.

-Any hazardous/chemical waste you have needs to be bottled and labeled and then placed into the waste chemical area. If you have more than a few waste items prepare the waste disposal forms, fax them to EH&S, and place your waste in secondary containment where EH&S will find it

-Any reagents in your possession should go back to the Chemical Storage Room or to someone in your group.

-Please take your lab coat with you.

-Let us know how to reach you.

Emergency Assistance

Fire, Police, Medical, Hazardous Materials, etc.

For Immediate Police, Fire and Medical Assistance 9-

911	From campus phone
911	From pay phones or residence hall phones
893-3446	From cell phones on campus

When in doubt, don't hesitate to call the paramedics at these numbers. Their evaluation is FREE and can help determine if an individual needs to go to the emergency room.

Note: Calling 911 from a cellphone will not contact UCSB Dispatch, but rather Ventura – so use of a campus phone to 9-911 is recommended, or dial 893-3446.

Non-Immediate Medical Assistance Students

http://studenthealth.sa.ucsb.edu/

Faculty and Staff (please click on "Notice to Employees")

http://www.busserv.ucsb.edu/workerscompensation/index.htm

Faculty and Staff Mandatory Injury Reporting Procedures

http://www.busserv.ucsb.edu/workerscompensation/reportingprocedure.htm

UCSB Environmental Health & Safety Emergency Assistance

Chemical spills, odors, leaks, accident investigation, other safety matters.

x-3194 From campus phones (24-hour technical assistance line). This number should be staffed during business hours and have a recording other times telling people to either leave a message or call 448-4089. During non-business hours there may a be significant wait until EH&S responds to a message from off-campus.

For immediate life or health-threatening emergencies, do not call EH&S, but dial 9-911 from campus phones.

Emergency Response Guidelines

http://ehs.ucsb.edu/units/labsfty/labrsc/firstrsp/EmergencyResponseGuidelines.pdf

Utilities Problems

x2661 x-4	7:45 am — 4:45 pm, weekdays
x3446	All other times

MRL TEMPO Facility

Required Training for Lab Users

March 22, 2013

Anyone who does any experimental work in the MRL TEMPO Facility is required to do and to document certain lab safety training, to demonstrate an understanding of that training, and to get the Facility's Development Engineer's approval before beginning any lab work. There are two levels of training that are required depending on the level of work to be done.

Safety training begins with the EH&S Laboratory Safety class. Everyone working in the lab is required to take this course BEFORE beginning lab work. Nearly everyone is required to take the course in person.

The course is available in several formats. The class is given in person at the start of each quarter. In summer there is a special in person class just for interns. In the fall the class is provided in person for incoming graduate students at several science departments and at the College of Engineering. Finally there is an online version of the course available for undergraduates and Post Docs.

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To get the Facility's Development Engineer's approval, at large lab researchers must complete the following before doing any work in the lab:

Take the EH&S Lab Safety Class in person,
Read the TEMPO Facility Chemical Hygiene Plan,
Pass the TEMPO Lab Safety Test,
Complete the Hazard Assessment with your PI and
Complete the MRL Participant Form.
Turn in all documentation.

Since the In Person EH&S Lab Safety Class is only offered quarterly, lab users may get temporary lab access until the next in-person class by taking the Undergraduate version of the Online Lab Safety class from EH&S. Such people are still required to attend the next available in-person Lab Safety class. Undergraduate lab researchers who work in the lab one quarter or less are only required to take the online lab safety class.

The in-person quarterly training schedule is announced by e-mail one to two weeks before the class and is posted online. The Online training is available at:

http://ehs.ucsb.edu/4DAction/WebCourseSessionList

Contact the lab Development Engineer, Joe Doyle (jdoyle@mrl.ucsb.edu), for the Welcome package and the TEMPO Lab Safety Test.

Concurrent with all the above, new arrivals at MRL should contact Sylvia Vogel for a copy of the MRL Participant form. This form directs participants to the required safety training beyond laboratory issues including fire, earthquake, ergonomics and more.

All of the above is just the beginning of laboratory safety training. Everyone working in the lab must do the appropriate inquiry, literature research, and thought to insure that the lab work they do is safe.

TEMPO Facility Specific Information

1. General Laboratory Information

Laboratory Name TEMPO Facility

Date(s) written or updated 7/20/98, 6/30/05, 3/13/2006, 1/13/07,9/23/09, 3/26/13

Facility Director Dr. Ram Seshadri

Department: Materials Research Laboratory

Office Phone Number: 893-6129

Office Location (Building & Room Number): MRL, Rm 3008

E-Mail address seshadri@mrl.ucsb.edu

Faculty Supervising Work in Lab

Name	Home Department	Campus Phone Number
Dr. Ram Seshadri	Materials Science	893-6129

Development Engineer Working in Lab Joe Doyle & Amanda Strom

Laboratory Location(s):

Building: Materials Research laboratory, Bldg. 615

Room Numbers: 1013, 1023, 1033, 1103, 1137, 1137A

Department Information:

Dept. Safety Representative (DSR): Joe Doyle, 893-7925, Rm 2066F_

Location of Department "Safety Corner": MRL Room 2042

Chemical Hygiene Officer: Dave Vandenberg, EH&S

2. Emergency Information

A. Emergency Notifications

In the event of an emergency call 9-11 then contact Joe Doyle at 893-7925.

B. Evacuation Procedures

Leave the room and the building as quickly as possible. Proceed to the Emergency Assembly area which is north of the MRL Building at the south west corner of Eng II. If time take valuable personal property.

C. First-aid kit: First aid kits are kept on the West wall in room 1023 and on the South wall of room 1137. It is the responsibility of the Lab DE to maintain the first aid kits.

D. Chemical, biological, or radiological spill cleanup materials:
Chemical spill cleanup kits are kept on a shelf on the west wall of room 1023. Procedures for use are in each kit.

E. Laboratory monitors or alarms:
There are no lab monitors except for low air flow monitors on the fume hoods. These are to be maintained by campus Physical Facilities.

3. Health & Safety References
The MRL Safety page,
http://www.mrl.ucsb.edu/mrl/info/administration/mrlsafety.html
has links to MSDS and LCSS sources. and other useful information.

The UCSB Environmental Health and Safety Web page has a lot of valuable and useful safety information

Lab Safety Info, look below the phone #s on this web page for specific information
http://www.ehs.ucsb.edu/units/labsfty/labsafety.html

Programs	http://ehs.ucsb.edu/program/program.html
Training	http://ehs.ucsb.edu/training/training.html
Lab Safety Fact Sheets	http://ehs.ucsb.edu/units/labsfty/labrsc/factsheets/lsfacsheets
Emergency Assistance	http://ehs.ucsb.edu/units/emplan/eprsc/emergphone.htm

Title and location of any health and safety reference materials associated with the laboratory which employees may use to aid them in their work.

1. Laboratory Safety Program/Chemical Hygiene Plan
(aka the Black Binder) Rm 2066F and in Rm 1023

2. Health And Safety Binder (the Green Binder)
In Rm 2066F and online at
http://ehs.ucsb.edu/units/iipp/iipprsc/greenbook.htm

3. Dangerous Properties of Industrial Materials, 8th ed. Rm 2066F

4. Merek Index Rm 2066F

After the SOPs there is an appendix with more sources of laboratory safety information.

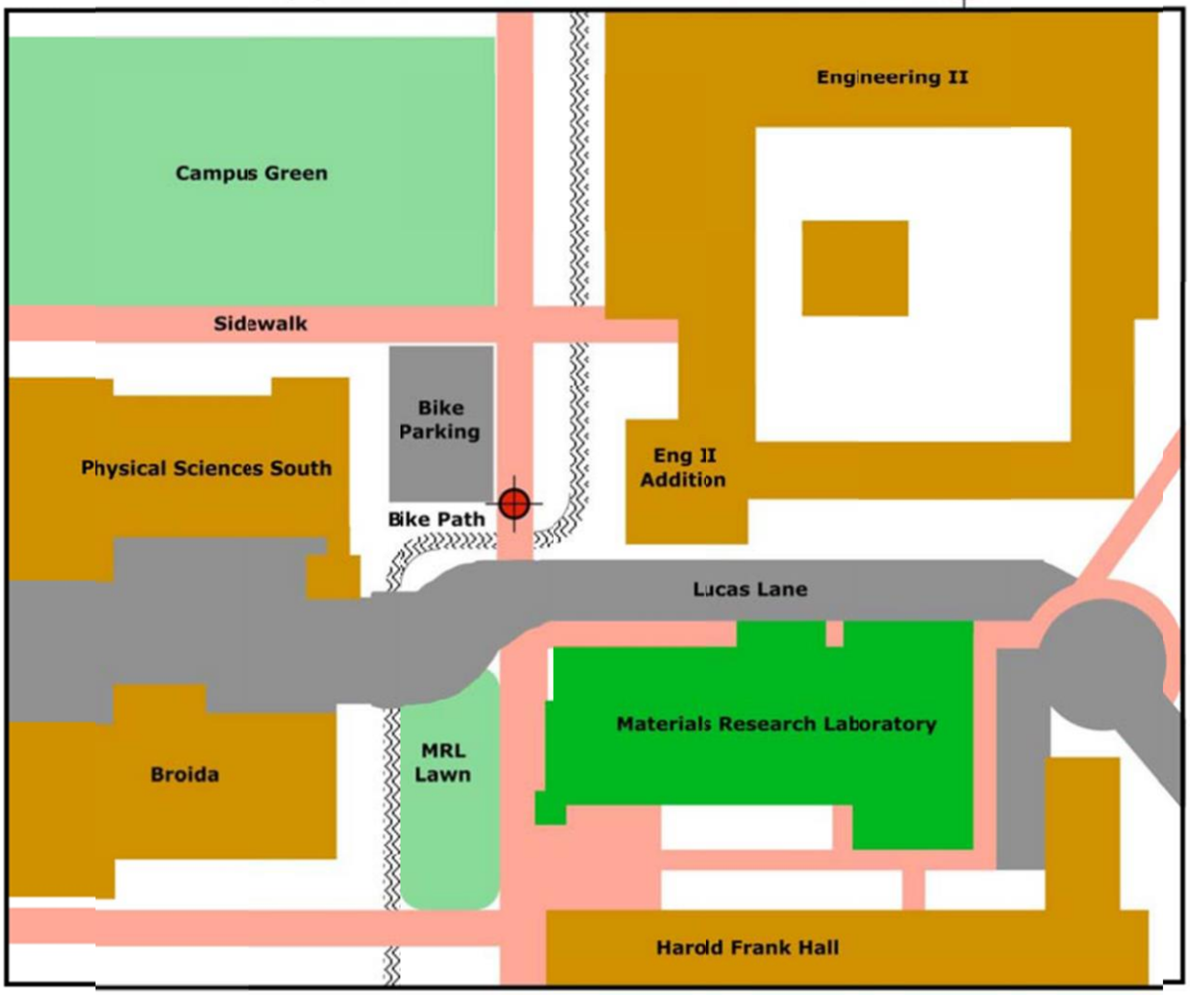
Some recommended general lab safety references include the following:

- *Prudent Practices in the Laboratory, Handling and Disposal of Chemicals,* National Research Council, National Academy Press, 1995.
- *CRC Handbook of Laboratory Safety,* 4th ed., 1995.
- *Safety in Academic Chemistry Laboratories*, American Chemical

Society, 6th ed., 1995.

Emergency Assembly Point for the MRL

The Emergency Assembly Point for the MRL is marked with this



During any real drill or malfunction, evacuation or fire alarm, go to the Emergency Assembly Point (EAP) immediately. Wait there until it can be seen that you and everyone else has evacuated the building safely. Do not re-enter the building until the alarm has been turned off.

Ten Commandments of Safety

1) Thou shalt wear thy safety glasses, as with all other personal protective equipment that shall be required. Thou shalt not mistake reading glasses for safety glasses.

2) Thou shalt label all of thy chemicals and samples with thy name and the essential chemical information. Thou shalt never leave unknown and unmarked chemicals or samples for thy neighbor.

3) Thou shalt never dump thy waste chemicals into the drain. Neither shall thou place any sharp waste, including broken glass, razor blades, nor needles, in the regular trash cans inside the laboratory. Thou shalt place all sharp waste into the dumpster or a special container.

4) If thou shalt wear gloves to protect thy hands from sinful chemicals. Thou shalt remove thy gloves before touching phone, keyboard, doorknob, or other place where thy gloves might spread thy sinful chemicals to thy brothers and sisters.

5) Thou shalt chain all of thy gas cylinders securely with chain of welded link. When the earth shall shake, thy chemicals, bookshelves, and heavy goods must not fall down to the earth or upon thy head.

6) Thou shalt not smoke within the laboratory. Neither shall thou confuse the laboratory with a place of nourishment.

7) Thou shalt purchase only the minimum chemicals for thy needs. Thou shalt share thy chemicals with thy neighbor and covet thy neighbor's chemicals.

8) Thou shalt not covet or contaminate thy neighbor's labware, especially furnace tubes.

9) Thou shalt not deliver oxygen in plastic tubing, lest the fires of Hell visit upon thy experiment.

10) Thou shalt know what thy are doing and about the hazards thy face. Thou shalt never toil in the laboratory until thou hast studied and trained about safe work practices for thy procedure.

MRL TEMPO Facility
Preparing For A New Project SOP

3/26/13 Joe Doyle

If you are undertaking a new project it is your responsibility to do whatever work is required to make sure you understand the hazards associated with the work required for the project. This may be as simple as reading an SOP, reading a few papers, and talking to other people in the lab. But usually more work is required.

If you are supervising or mentoring someone, then it is your responsibility to understand the hazards of the work yourself and to make sure the person you supervise understands those hazards too.

You may be undertaking a project your lab has never tried before. Or you may even be trying a procedure no one has ever done before. There is not set procedure to research the possible hazards of a new process. The investigation depends on the nature of the project.

Two experienced MRL lab experimentalists kindly offered to write down their perspective on this open ended problem. Their approaches complement each other nicely. These provide some specific information but are focused on what questions should be considered when beginning a new project.

Eric Toberer, June 16, 2006

All new projects should begin by considering the health and safety hazards of the materials involved. MSDS sheets is a good place to start.
As well, an educated guess of what the evolved gases, intermediate phases, and final products will be is necessary. If you're not capable of making an educated guess, get educated. Either find someone who is well-acquainted with the chemistry or do a lit search.

Be particularly aware of flash points, inhalation hazards, explosive materials, or air/water sensitive materials. Toxic/hazardous materials are no more dangerous than a loaded gun; if used responsibly, you probably won't kill anyone you didn't mean to kill. If used irresponsibly, you'll end up shooting yourself in the foot.

Determine the appropriate level of personal protective equipment, and then exceed that level.

While we are more aware of short-term acute dangers, long term exposure issues are equally troubling but often ignored.

For all new projects, talk with someone who is doing similar work and see what level of safety they operate within. This is not a green light! Don't emulate bad behavior. Nor should you assume people are correct about the hazard level of chemicals. This lack of knowledge is particularly prevalent for long-term hazards (most people can tell if something is exploding, burning, scarring them).

New projects in the MRL come in two flavors:

wet synthesis-	hydrothermal, refluxing solutions, spin coating, etc
dry synthesis-	heating solid powders

Powder vs. liquid
-exposure- powder exposure is difficult, provided you're not working with dusty materials or dry nanoparticles. In contrast, the vapor pressure of many solvents is high and exposure is easy. Fume hoods are great.

-explosive- be aware of the amount of pressure your container can take, be it a hydrothermal bomb or a round bottom flask. Make sure that your reaction doesn't result in large pressure changes comparable to the strength of the container. Similarly, in solid state synthesis, be aware of decompositions in sealed containers (i.e. quartz ampules).

-flammable- pyrophoric nanopowders can be formed through heating in a reducing environment. These materials are flammable, but not explosive by themselves. Still, molten iron will burn a nice hole in your leg. For liquid reactions, be very aware of flash points and flammability levels. Our lab doesn't have much in the way of open flames, but sparks are still easy to create from electrical outlets, etc.

-ruining equipment- part of designing a new project is not destroying things. Don't do stuff that's going to ruin lab equipment. Ruin your own equipment. Particularly, don't heat stuff that is volatile in box furnaces and don't let acid fume in the hoods. Plus the other 8000 ways to ruin things.

-think about how you're going to dispose of the material you create. Do it efficiently and in a green manner and don't procrastinate.

-when coming up with a new project, try to design your experiments in a concise and efficient manner, so as to minimize waste, time, and use of equipment.

Russell Feller, July 11, 2006

Inorganics--certain elements (lead, cadmium, mercury, thallium, beryllium, arsenic, and selenium) have very specific, known health risks associated with them. Look up suggested precautions when using them. (plus or minus any others you think should be in that list)

Oxidizers--Can the metal that you're using be accidentally oxidized to a stable but toxic high valence, such as chromium III --> chromium VI (chromate/dichromate) or Mn IV --> Mn VII (permanganate)? If so, avoid using harsh oxidizers on this metal. (do you know other examples of this? I've heard of (FeO4)-2 and (IO4)-. Maybe these are so unlikely to happen by accident that it's not worth mentioning.)

(Am I remembering correctly that perchlorate fumes react with metal fume hood vents to produce shock-sensitive explosive residues?)

(I guess HF/F- should be mentioned somewhere, but I don't know the details of what it does. Once I bought BF4- salts, but scared myself into not using them, fearing that they would decompose to BF3 and either F- or F2 hydrothermally. Same problem with PF6- salts.) But if you are working with HF or a reaction that generates F^-, know where the calcium gluconate neutralizing gel is (Rm 1023 by the ICP) or keep it nearby. Also be aware that HF etches glass and should be used in plastic containers.

Is radioactivity an issue?

Organics--does your molecule contain any surprising functional groups such as azides, azas, nitros, or peroxides? Such functional groups may be unstable and decompose explosively. Look up what other groups or reagents or conditions can trigger this decomposition. (do nitrosamines do this? what other groups?) Also consider what reactive or toxic materials that could form as by-products or waste that has to be disposed of.

Ethers and Peroxides--Will you need to use an ether as a solvent? If so, write the date on the bottle when you buy it. Ethers will transform into peroxides over time. These are explosive, and sensitive to the shock of screwing open the bottle.

Oxidation of organics by nitric acid/nitrates leads to heat and CO_2. These can cause pressure buildup in closed containers.

Hydrothermal reactions-see the SOP for Use of Parr Bombs or speak with lab bombers.

Look up decomposition temperatures of any new chemical you put into a bomb. Many small organics will decompose semi-safely (such as decarboxylation of beta-keto carboxylates or meta/para-hydroxybenzoates) but the "surprising" groups listed above may either burst the bomb liner, or release noxious fumes upon opening the liner. (I used thiols extensively for a while, but with full anticipation of fumes.)

Glassware

Pressure--Will your glass vessels and tubes be under significant pressure from the inside? See if a glass manufacturer or the glass shop in chemistry can estimate the sustainable pressure of each component. If there's risk of explosion, or even implosion, insert the questionable component into a plastic netting to contain all the fragments. (what are those plastic things actually called?)

Connections--What type of joints and connections will you need? Again this is based on pressure, but also temperature. Would a plastic clip melt? Verify that the type of joint grease you're using, if any, won't decompose at the temperatures that the glass will reach.

Electronic Components Have some idea if your device will draw an unusually large current. If it does, the wires supplying it should be thick enough to not resistively heat up and melt the insulation.

Will some of your components need electrical grounding? Some older devices don't have the three-pronged grounded plugs even though they really should be grounded.

Do any of your components contain a huge capacitor which is charged up during its operation? If so, be aware of this if you reach in and try to fix anything. As with TVs, a device with a big capacitor, even when unplugged for many hours, can still discharge a massive current into you if you accidentally touch it.

Traditional Solid State Reactions--Experienced lab users may know more about making and using glass tubes, metal tubes, the new arc melter, furnace tubes, glass versus quartz glass, and alumina boats. I don't know if any of their maximum temperatures and pressures lie within a plausibly accessible range. Also if people will be using an odd gas, check which gasses corrode with which tube materials. I bet newer people wouldn't know about things like H_2 eating up Pt or Pd.

We should tell people to think about what their sample's fumes could do to a high-temperature oven. One thing that always surprised me was the relative volatility of certain metals at high temperature, not necessarily consistent with their melting or boiling points. It would be good to have a list of the most volatile metals, to avoid oven contamination.

I was told that cobalt will never come out of an alumina boat because it forms $CoAl_2O_4$ spinel...I assume magnesium would have the same problem with $MgAl_2O_4$. Are there others like this?

Has there ever been a problem with someone touching quartz glass with their bare hands, getting salt on it, and having it devitrify and crack at high temperatures? I've never seen this in person, but I've been told about it, and a new person might not know about it.

Chemical Storage SOP
MRL TEMPO Facility

Jaya Nolt & Joe Doyle August 20, 2009

Proper chemical storage is essential in assuring a safe laboratory environment. Incompatible materials must always be separated in storage. Stored chemicals must always be secured for an earthquake. Stored chemicals should be put away and not allowed to become a trip hazard.

It is the responsibility of each person who uses chemicals to insure that they are put away safely when they are done using the chemicals EACH DAY.

We have chemical storage cabinets in room 1137, under each fume hood in 1033 and 1013, and under benches in 1023. Use these cabinets for your chemical storage! Dry powders may be kept in individual drawers if they are not water reactive, carcinogenic, or especially hazardous in some other way.

Gas storage is a special case and will be covered at the end of this SOP.

Before ordering chemicals, check if someone here already has the reagent. We have a huge inventory of chemicals. Most chemical storage cabinets have an inventory list inside the door. People are encouraged to share our chemicals with other labs at UCSB. People are also encouraged to "borrow" small quantities of chemicals from other labs at UCSB before buying them. Besides saving money and storage space, this sharing can save weeks in executing an experiment.

Only order the minimum amount to meet your needs, even if the large economy size is a bargain.

When new chemicals arrive, write your name and the date opened on the bottle. Add the chemical to the inventory list on the storage cabinet door.

Chemical Segregation

Always Store Chemicals By Their Hazard Class! Never put incompatible chemicals next to each other or even in the same cabinet!

We separate our chemicals into the following categories. New materials may require additional categories.

- Flammable materials
- Acids
- Bases
- Salts and solids
- Water reactive substances
- Oxidizers

- Organic Reagents
- Air/Water Reactive

If you are not sure what category some chemical is, ask. The EH&S Fact Sheet on Chemical Storage in the Appendix has more information of classifying chemicals.

Never Store Chemicals Alphabetically, except within a hazard class.

Prepare for an earthquake. One is guaranteed, we just don't know when! Shelves must have a lip to keep materials from falling in a quake.

Time Sensitive Chemicals

Some chemicals decompose into explosive materials when they are stored for a year or more. Ethers and peroxides are especially prone to this. It is each researcher's responsibility to know if their chemicals decompose and if so to make sure they are disposed well in advance of becoming a hazard.

Anyone seeing a time sensitive chemical that is past due must take action to get it out of the lab and to campus EH&S for proper disposal. Contact Joe Doyle if you find such a chemical.

EH&S has written a Fact Sheet on Time Sensitive Chemicals and it is in the Appendix. Check it out.

Compressed Gas

Should a gas cylinder fall over in an earthquake and damage the valve on top, the release of energy would be huge. A relatively small SCUBA tank was known to have flown over a quarter mile when the valve was broken off!

All gas cylinders must be secured to a wall, stout table, cylinder rack, or gas cabinet at all times, the only exception is when they are being actively moved. Cylinders must be secured with welded link chain and not with a strap that would melt in a fire.

Cylinders being moved must have the regulator taken off and the metal cap threaded in place.

Gas cylinders have all the hazards of the chemical within and the hazards of a highly compressed gas. Like other chemicals, gas cylinders need the name of the person responsible for them clearly marked.

Flammable gas cylinders are to be stored at least 20 feet from oxygen cylinders.

Users of oxygen cylinders must be constantly aware that pure oxygen makes many things burn vigorously!

The most common gas cylinders we use are argon and nitrogen. These are not chemically hazardous, except that they could displace the oxygen in the air in a confined space and asphyxiate someone.

Hydrogen gas is very flammable and should be kept in a gas cabinet if one is available.

Ammonia gas is more flammable that most people think, and is it very toxic. It must be kept in a gas cabinet.

Per Article 80 of the Uniform Fire Code, any gas more toxic than carbon monoxide must be kept in a gas cabinet. There are other requirements. Fortunately we don't use these kinds of toxic gases very often.

SOP

Laboratory Coats & Clothing

August 26, 2009 Joe Doyle

Lab researchers must be aware of their clothing as protection from laboratory chemicals, heat, sharps, and other hazards but at the same time be aware that some clothing can exasperate certain hazards. The worst case is that polyester clothing or lab coats can catch fire and severely burn the person wearing it. This was a significant cause of a lab fatality in 2008.

In general, when doing chemical synthesis or working with furnaces wearing, a lab coat is desirable but not required while working in the TEMPO lab.

Closed toe shoes are always required for anyone in the lab at any time. Appropriate safety glasses are required for all persons working with any chemical more hazardous than Goleta water. Gloves are often a very good idea. Safety glasses and gloves are addressed on other parts of this CHP.

For the routine work of the TEMPO Facility, a 100% cotton lab coat will usually be the best choice. Researchers working with strong acids or bases should prefer a poly/cotton blend lab coat.

Researchers working with extremely flammable material or pyrophoric liquids should consider Nomex lab coats because they offer the most fire protection.

100% Cotton lab coats may be ordered from Fisher Scientific in most sizes. Search their web page http://www.fishersci.com with item 01-369-2C then choose the right size. The UC price is about $17.

Prudent Practices in the Laboratory offers this advice for lab wear.

Sandals and open-toed shoes should never be worn in a laboratory in which hazardous chemicals are in use.

It is advisable to wear a laboratory coat when working with hazardous chemicals. This is particularly important if personal clothing leaves skin exposed. Apparel giving additional protection (e.g., non-permeable laboratory aprons) is required for work with certain hazardous substances. Because many synthetic fabrics are flammable and can adhere to the skin, they can increase the severity of a burn. Therefore, cotton is the preferred fabric. (Chapter 5.C.2.6)

Appropriate laboratory coats should be worn, buttoned, with the sleeves rolled down. Laboratory coats should be fire-resistant. Those fabricated of polyester are not appropriate for glassblowing or work with flammable materials. Cotton coats are inexpensive and do not burn readily. Laboratory coats or laboratory aprons made of special materials are available for high-risk activities. Laboratory coats that have been used in the laboratory should be left there to minimize the possibility of spreading chemicals to public assembly, eating, or office areas, and they should be cleaned regularly.

Protective apparel should always be worn if there is a possibility that personal clothing could become contaminated with chemically hazardous material. Among the factors to be considered in choosing protective apparel, in addition to the specific application, are resistance to physical hazards, flexibility and ease of movement, chemical and thermal resistance, and ease of cleaning or disposal. Although cotton is a good material for laboratory coats, it reacts rapidly with acids. Plastic or rubber aprons can provide good protection from corrosive liquids but can be inappropriate in the event of a fire. Plastic aprons can also accumulate static electricity, and so they should not be used around flammable solvents, explosives sensitive to electrostatic discharge, or materials that can be ignited by static discharge. (Chapter 6.F.1) http://www.nap.edu/catalog.php?record_id=4911

SOP Accidents, Eyewashes, First Aid Kits, and Chemical Spill Clean Up Kits

March 26, 2013

Joe Doyle

We put a lot of effort into preventing accidents, serious chemical spills, fires, and personal injury in the TEMPO Facility. Even so, we do need to be prepared for the worst.

In any serious accident, call 9-911 immediately from any campus phone (or 911 from a cell phone).

For chemical spills, odors, leaks, accident investigation, other safety matters; call extension 3194 from any campus phone, or 893-3194 from a cell phone. This is the 24-hour EH&S assistance line. This number should be staffed during business hours and have a recording other times telling people to either leave a message or call 448-4089. During non-business hours there may be a significant wait until EH&S responds to a message from off-campus.

If appropriate, evacuate the area.
Then contact Joe Doyle.

There are three first aid kits and an HF neutralization kit in the lab. They are wall mounted at these locations:

First Aid Kit	Vestibule, rm 1137	South wall by safety shower
First Aid Kit	Wet Lab, rm 1033	West wall by sink
HF Exposure Kit	Instrument Lab, rm 1023	South wall
First Aid Kit	Rm 1013	West wall by sink

There are Spill Clean UP Kits in these locations:

Vestibule, rm 1137	On top of glass door cabinet
Instrument Lab, rm 1023	Shelf on West wall
Instrument Lab, rm 1023	Shelf on East wall

People working in the lab should make a point of knowing where all of these kits are located.

Each of our three labs has a safety shower on the south wall. Most of the time they are in the way, but in an emergency we will be glad to have them. These showers do have built in eyewashes. Most lab sinks also have hand held eyewashes. People working in the lab should make a point of knowing where each of these is located and be able to find them without thinking or even seeing. In the event of an emergency, people in the lab will need to find a safety shower or an eyewash without having to waste time searching.

If a chemical splashes in someone's eye, rinse with water for a minimum of 10 minutes. 15 minutes may be better. It is a good idea to let the eyewash water run a minute before putting in anyone's eye, they don't get much use and may have a little rust in the first few gallons of water to come out. There are hand-held eyewashes at every sink in the lab and there are eyewashes at each safety shower.

If someone's skin or clothing is splashed with a hazardous chemical, use a safety shower to rinse the chemical off. A small exposure may just need to be rinsed with a hand-held eyewash. Then the chemical may need to be neutralized. Calcium gluconate is well suited for HF or any acid spilled on skin.

Calcium gluconate can neutralize acid burns on the skin. It is the first remedy for an HF exposure because it provides a calcium source. There should be calcium gluconate paste by the ICP in rm 1023 and in the Acid cabinet in the Chemical Store Room.

If a flammable or pyrophoric material is splashed on someone and is on fire, they must be taken to the closest safety shower and doused with water as soon as possible.

If there is a Fire Alarm: evacuate lab and go to the Emergency Assembly Point at the SW corner of Engineering II.

If there is a lab fire, it must be reported to 9-911 even if it gets put out.

If there is a large, seriously toxic, or highly flammable chemical spill, warn people in the area, evacuate the area, notify 9-911, and notify Joe Doyle.

Users should clean up small chemical spills as soon as possible using all appropriate personal protective equipment.

Emergency Response &
Chemical Spill Cleanup SOP

July 12, 2006
Joe Doyle

Call 9-911 if there is a fire, personal injury, or danger to life or property.

Chemical Spill Cleanup Procedures

Do not try to clean up any spill that presents an immediate fire hazard or where exposure to fumes would result in physical injury. Evacuate the area, alert neighbors, and call 9-911 immediately.

You should NOT clean up a spill if:

- You don't know what the spilled material is
- You lack the necessary protection or equipment to do the job safely
- The spill is too large to contain
- The spilled material is highly toxic
- You feel any symptoms of exposure

Instead contact one of the following:

Call extension 3194 from any campus phone, or 893-3194 from a cell phone. This is the 24-hour EH&S assistance line. This number should be staffed during business hours and have a recording other times telling people to either leave a message or call 448-4089. During non-business hours there may a be significant wait until EH&S responds to a message from off-campus.

Call 9-911 if spill is immediately health-threatening

Spill Response Scheme:

If you can safely clean up a spill then:

Evaluate and Notify

- Assess the toxicity, flammability, or other properties of material (see label and MSDS)

http://www.ehs.ucsb.edu/units/labsfty/labrsc/chemistry/lschemmsds.htm

- For flammables, remove or turn off all ignition sources such as motors, pumps, fridges.
- Determine if there is an immediate health threat to you or your neighbors If so, alert neighbors, isolate the area, and call for help using the numbers above.
- If the spill is minor, begin cleanup following steps below

Containment/Cleanup

- Wear gloves, eye protection, lab coat, etc.

- Contain and absorb spill using absorbents appropriate for the material
- Protect floor drains from contamination, by putting absorbents or barriers around them
- Package and label waste. Include contaminated clothes, rags, equipment, etc.
- Store temporarily in a fume hood if material is volatile

Sources For More Information

The Campus EH&S page on Laboratory Safety is at
http://ehs.ucsb.edu/units/labsfty/labsafety.html

UCSB EH&S has posted vast amounts of useful safety information on their web page but it may not be easy to find it. Check
http://www.ehs.ucsb.edu/units/labsfty/labrsc/chemistry/lschemContentsCHP.htm
for information on the following:

Introduction to Campus Procedures and Resources
Website Map for Laboratory Safety (PDF)
Emergency Phone Numbers
Chemical Spill Cleanup Procedures
Fire Fighting and Extinguishers
Personal Protective Equipment in UCSB Storerooms
Eyewear Policy and Selection
Selecting the Proper Gloves
EH&S Lab Safety Class Descriptions
Laboratory Self-Inspection Checklist
Material Safety Data Sheets (MSDS)
Safe Storage of Chemicals (PDF)
Refrigerators in Labs(PDF)
Hazardous Waste Disposal Procedures
Disposal of Sharps (PDF)
Safety Guidelines for Field Research (PDF - UC Berkeley)

At http://www.ehs.ucsb.edu/units/labsfty/labrsc/factsheets/lsfacsheets.html
the following laboratory Safety Fact Sheets are available:

CHEMICALS:
Acrylamide
Arsenic
Azides, Handling Organic
Benzene NEW
Cadmium
Chlorinated Solvents
Corrosives
Dichloromethane (also known as methylene chloride) NEW
Ethidium Bromide Safety
Formaldehyde
Hydrofluoric Acid
Perchloric Acid
Peroxides and Distillations
Phenol
Picric Acid
Pyrophoric Organolithium Reagents NEW
Water Reactive and Pyrophoric Materials NEW

CHEMICAL SAFETY:

Carcinogen Control Chemical Storage Cryogens
Housekeeping Guide for labs
Lab Coats NEW
Nanomaterials Safe Handling NEW
Power Failures Guide
Quenching Solvent Drying-Still Bottoms NEW
Seismic Hazard Reduction
Sharps Disposal
TA Guide
Time-Sensitive Chemicals

LAB EQUIPMENT:
Autoclaves
Centrifuge
Compressed Gas Cylinders
Electrophoresis Equipment
Environmental Rooms
Fume Hood Usage Guidelines
Refrigerator & Freezers in Lab

HAZARDOUS WASTE:
Biological Waste Disposal
Chemical Waste Disposal

Materials Research Laboratory TEMPO Facility

SOP For Use of Parr Pressure Vessels

Facility Director: Dr. Ram Seshadri

Faculty in charge of this process: Dr. Ram Seshadri

Lab Development Engineer: Joe Doyle

Date of last revision to SOP: 7/20/98, 11/9/98, 6/16/06 (Crystal Merrill), 8/20/09

Lab Location(s) covered by this SOP: MRL Building, Room 1013, 1023, 1033, 1103, 1137, 1137a

Process "Name" : Synthesis or digestion in a "Parr" bomb.

1. Laboratory Process or Equipment Description (brief)

The "Parr Bomb" is a closed stainless steel vessel with an internal cup and lid made of teflon. It comes in several forms, most of which are made by Parr Inc. The bomb can be charged with reagents, and closed. It may then be heated and the contents will come to higher temperatures and pressures then could an unsealed container. The bomb is specifically designed for harsh chemicals, high temperatures, and high pressures.

Most people use Parr Model 4749 bombs

2. Approval Required

Specific approval is not required.

3. Describe the training, operation, and maintenance aspects of this process.

People should read both Parr's Operating Instructions for Parr Acid Digestion Bombs and their Safety in the Operation of Laboratory Reactors and Pressure Vessels before using the bombs.

Reagents used in the vessel must not react to release gas. This will lead to excessive pressure buildup.

Before every use both the stainless steel shell and the Teflon liner should be visually inspected for cracks or excessive wear. Steel shells that are cracked or flawed in any way must be discarded. Worn or distorted Teflon liners should be replaced.

New liners and bombs can be purchased from Fisher Scientific. For Model 4749, Teflon liners are part # 01-023-21A and cost about $85; the complete unit is part number 04-731-54 and costs about $533.

Some bombs were made by the Chemistry Machine Shop. These have a history of cracking and failure. They must NEVER be used. Any such bombs should be given to Joe Doyle.

When charging the bomb with reagents, it should never be filled more than half way.

Nitric acid should not be used with organic materials to prevent the formation of nitro explosives.

Reactions which are highly exothermic or which release large quantities of gas (such as an oxidant and an organic compound) should not be done in the bombs.

Percloric acid should not be used in the bombs.

Do not overheat the bombs. All Parr bombs have a maximum temperature of 250 degrees C except model 4745 which has an upper limit of 150 degrees.

Maximum pressure specifications are in the Parr Bomb manual. Tables are given to predict pressure from the volume of material in a bomb of known size for a series of temperatures. For each run, it should be verified if the pressure expected is within the limits of the device.

Most models have a rupture disk to release energy should the temperature or pressure become too high. Nothing must ever be done to inhibit the function of this rupture disk.

If the rupture disk does operate, chances are that a loud noise will accompany the event. Be aware that the contents of the bomb may have been spread to the inside of the oven and may be dangerous to personnel and to the materials of the oven. The contents must be cleaned up by the user using appropriate personnel protective equipment dependent on the nature of the materials released from the bomb.

After use, the bombs must not be opened until they are fully cool. Even then, they should be opened with care because the contents may still be under pressure.

4. Hazardous Chemicals/Class of Hazardous Chemicals

Nearly any chemical may be used in the Parr Bomb, for exceptions see above.

The chemicals used will often be corrosives, solvents, inorganic materials, or amines.

5. Personal Protective Equipment

Safety glasses must be worn when charging or opening the bomb.

Safety glasses must be worn when opening an oven that might contain bombs

Heat insulating gloves may be worn when removing the bomb from the oven.

6. Engineering/Ventilation Controls

Each Parr bomb should have a built in pressure relief valve.

Depending on the reagents used, it may be appropriate to fill and unload the Parr bomb inside a fume hood.

7. Special Handling Procedures and Storage Requirements
Describe any special storage or transportation requirements for hazardous chemicals used in this process.

None.

8. Spill and Accident Procedures
Indicate how spills or accidental releases should be handled and by whom.

See section 3 above.

9. Waste Disposal

There are no special waste disposal procedures for the use of the Parr Bomb. Chemical waste should be disposed of in the usual EH&S approved way.

10. Decontamination
Discuss decontamination procedures for equipment and glassware.

There are no special decontamination procedures.

Ammonia Gas SOP

Facility Director: Dr. Ram Seshadri

Faculty in charge of this process: Dr. Ram Seshadri

Lab Development Engineer: Joe Doyle & Amanda Strom

Date of last revision to SOP: 08/24/98 6/21/06 (Birgit Schwenzer)

Lab Location(s) covered by this SOP: MRL Building, Rooms 1013, 1023, 1033

1. Laboratory Process or Equipment Description (brief)

Ammonia gas is a poisonous gas that is used for the nitridation of compounds at elevated temperatures. The ammonia cylinder should be secured against earthquakes and attached to a regulator and a flowmeter. The flowmeter runs into a stainless steel line that feeds into a quartz tube that is heated in a clamshell furnace up to 1100 ˚C. The exiting gas is bubbled through mineral oil (not water, so that water is not pulled back into the furnace).

All processes using ammonia gas must be inside a fume hood.

More often the ammonia is stored in a large pressurized cylinder in a gas cabinet in room 1013. Ammonia gas is plumbed to a furnace in an adjoining fume hood.

The ammonia may be stored in a lecture bottle in a gas cabinet when not in use.

2. Approval Required

See Joe Doyle for approval of experimental setup prior to use. Caution must be taken if high temperature use is necessary (Ammonia decomposes above 600 ˚C to form nitrogen and hydrogen).

3. Describe the training, operation, and maintenance aspects of this process.

When not in use, lecture bottles of ammonia should be stored in a gas cabinet with their respective "plugs" screwed into the outlet port. No larger size container than a lecture bottle of "pure" ammonia may be used unless it is in a gas cabinet.

Larger containers of diluted ammonia (i.e., <2.5% in nitrogen or helium or other inert gases) may be used with approval from Joe Doyle.

While in use, ammonia lecture bottle must be in fume hood and secured.

If passing through a bubbler, use a mineral oil not water. All operational changes to experimental setup must be reviewed and approved by Joe Doyle. Proper regulator must be used with specific size cylinder (consult Matheson catalogue). Fume hood windows should be near fully closed during operation. If there is a leak in part of the line, detection is readily apparent. In this case, ammonia lecture bottle must be closed and time should be allowed for the concentration to decrease.

To operate, open main cylinder valve, regulator needle valve and flowmeter, respectively, for use. After use, close flowmeter, regulator needle valve, and main cylinder valve. For extended storage, remove regulator and "cap" or "plug" outlet port of cylinder. Store in gas cabinet in Room #1033.

4. Hazardous Chemicals/Class of Hazardous Chemicals
Ammonia is poisonous and surprisingly flammable.

5. Personal Protective Equipment

Safety glasses should be worn at all times. Chemical resistant (nitrile) gloves should also be worn.

6. Engineering/Ventilation Controls

All work should be done in the fume hood. The proper regulator should be attached to the cylinder. Leaks will easily be detectable by the user if gas escapes the hood. Mineral oil bubblers should be used instead of water.

7. Special Handling Procedures and Storage Requirements

Extended storage of lecture bottle or any cylinder of ammonia should be in proper gas cabinet with "plug" inserted in outlet port.

8. Spill and Accident Procedures

See section 3. Person(s) should not be inhaling ammonia; move to fresh air and if high concentrations are inhaled, seek medical attention.
In case of a leak out of the line, close lecture bottle main valve. If leak cannot be contained, contact Joe Doyle immediately. Large leaks may require contacting campus EH&S at 893-3914 for assistance.

9. Waste Disposal

Empty ammonia lecture bottles should be labeled as such. Disposal should be done through campus EH&S.

10. Other Information

Ammonia is poison, do not breathe. Always work in fume hood.

Materials Research Laboratory
TEMPO Facility
SOP For Use OF HF

Facility Director: Dr. Ram Seshadri

Faculty in charge of this process: Dr. Ram Seshardri

Lab Development Engineer: Joe Doyle & Amanda Strom

Date of last revision to SOP: June 24, 2006 (Scott Fillery)

Lab Location(s) covered by this SOP: MRL Building, Room 1013, 1023, &1033

Process "Name": Use of Hydrofluoric Acid (HF)

1. Laboratory Process or Equipment Description (brief)

Liquid hydrofluoric acid (HF) is used for digestion of solid samples for ICP analysis and to get solid reagents into solution for reactions and synthesis. Hydrofluoric acid (HF) is also used extensively to etch or remove native oxides from a number of materials including Silicon, glass and inorganic meta-glasses.

HF is extremely corrosive to all tissues of the body and great care must be exercised in its use.

2. Approval Required

Prior approval is not required.

If at all possible, other materials should be substituted for HF and its use avoided.

3. Describe the training, operation, and maintenance aspects of this process.

No one should use HF until they have studied the laboratory Chemical Safety Summary (LCSS) AND discussed its use with more experienced lab personnel. The LCSS is available at http://www.hhmi.org/science/labsafe/lcss/tlisting.htm or from Joe Doyle. The MSDS is an acceptable substitute, but the LCSS is preferred.

4. Hazardous Chemicals/Class of Hazardous Chemicals

a. For the process, list the hazardous chemicals used and expected byproducts
b. List the hazard class of the chemicals, e.g., "carcinogenic," "highly toxic, " "teratogen," "corrosive, " etc. See Section I: B(6) of this manual regarding "Particularly Hazardous Substances. "

Hydrofluoric acid is extremely corrosive to all tissues of the body. Skin contact results in painful deep-seated burns that are slow to heal. Burns from dilute (<50%) HF solutions do not usually become apparent until several hours after exposure; more concentrated solutions and anhydrous HF cause immediate painful burns and tissue destruction. HF acid burns become visually apparent as a blistered, red/purple burn that will eventually lead to a white spot on the skin surface. HF burns pose unique dangers distinct from other acids such as HCl and H_2SO_4: undissociated HF readily penetrates the skin, damaging underlying tissue; fluoride ion can then cause destruction of soft tissues and decalcification of the bones. Hydrofluoric acid and HF vapor can cause severe burns to the eyes, which may lead to permanent damage and blindness. At 10 to 15 PPM, HF vapor is irritating to the eyes, skin, and respiratory tract. Exposure to higher concentrations can result in serious damage to the lungs, and fatal pulmonary edema may develop after a delay of several hours. Brief exposure (5 min) to 50 to 250 PPM may be fatal to humans. Ingestion of HF can produce severe injury to the mouth, throat, and gastrointestinal tract and may be fatal.

Information about the properties of liquid HF in 5% or lower concentration has not been found. The use of very dilute HF is encouraged over the more concentrated forms, however users should treat it with great respect.

5. Personal Protective Equipment

All users of liquid HF must wear safety glasses (with side splash guards) and gloves. It is recommended that users wear 2 or more sets of gloves as a protection barrier to possible HF acid contamination. The first set of gloves, adjacent to the skin surface should consist of either vinyl or latex gloves. The second layer should consist of purpose built HF acid handling gloves, it is generally recommended that Trionic (a 19mm thick, purpose blended glove of latex, neoprene and carboxylated ntrile) gloves are designated specifically as HF acid handling gloves. The first set of gloves provides limited protection but prevents possible skin contact with HF during application and removal of the second set of gloves.

The use of full face shields and fore arm protection is strongly encouraged. A lab coat would be appropriate for arm protection. For any procedures requiring agitation of HF acid, a rubber apron is recommended as a substitute for the lab coat.

HF should be purchased and used with small (500 ml or less) bottles that allow easy and controlled pouring.

Calcium gluconate paste should be applied to any skin areas contacted with HF within seconds of contact. It must be readily available before any use of HF.

6. Engineering/Ventilation Controls

Liquid HF handling should be confined to the inside of a fume hood. This fume hood should designated as a HF acid handling area and should be located in a low traffic area of the laboratory to prevent spills or accidents from contact.

If HF has to be measured by mass, it should be approximately measured in a plastic graduated cylinder inside a fume hood, then transferred to an appropriate container inside the fume hood, and then carefully moved to a lab balance.

7. Special Handling Procedures and Storage Requirements

Liquid HF should be stored in appropriate "Acid" storage cabinets. In these cabinets, it should be within a plastic tray for additional secondary containment. HF should be stored in separate secondary containment trays from other acids with an "Acid" storage cabinet. Other chemicals, especially organics, solvents, and bases should never be stored with HF.

8. Spill and Accident Procedures

Laboratory personnel should be familiar with first aid procedures before beginning work with HF; calcium gluconate gel should be readily accessible in areas where HF exposure potential exists.

First aid must be started within seconds in the event of contact of any form. In the event of skin contact, immediately wash with water for 15 min and remove contaminated clothing. If available, apply calcium gluconate gel. Obtain medical attention at once, and inform attending physician that injury involves HF rather than other acid. In case of eye contact, promptly wash with copious amounts of water for 5 min while holding the eyelids apart and seek medical attention at once. If HF is ingested, obtain medical attention immediately. If HF vapor is inhaled, move the person to fresh air and seek medical attention at once.

In the event of a spill of dilute hydrofluoric acid; soak up the acid with an HF-compatible spill pillow or neutralize with lime, calcium gluconate, or another mild base; transfer material to a polyethylene container; and dispose of properly, in a labeled storage medium used only for HF acid. Use personnel protective equipment as described above. Respiratory protection may be necessary in the event of a large spill or release in a confined area.

In any spill, alert any other people working in the lab to the hazard.

For a large spill, contact Joe Doyle immediately. Very large spills may require contacting campus EH&S at 893-3914 for assistance. This phone number is available 24 hours a day. After hours, emergency personnel can be paged through this number.

9. Waste Disposal

Describe any special waste disposal procedures for these chemicals.

Waste HF should be collected for disposal with campus EH&S. HF acid should under no circumstances be disposed down the drain. All solutions containing HF must be collected and disposed of using correctly label storage vessels. It should not be mixed with other acids or solvents. HF should be collected for disposal in well labeled plastic containers. Dilute salts mixed in with the HF are not a problem. Any material used to soak up suspected HF acid during spills or normal use must be placed in a plastic bag labeled as HF waste and disposed of correctly. Never throw spill pillows or absorbent material in the normal trash.

10. Decontamination

Materials contaminated with HF may be washed with copious amounts of water in a lab sink. It is generally accepted that equipment and plastic-ware must be washed for at least 6 minutes under flowing water before drying. Proper use of HF acid requires specifically labeled equipment and plastic-ware for use with HF that are labeled as HF only, kept in and around the HF acid handling area and used with HF only.

11. Other Information

Be very careful when handling HF!

Simple Procedure for the use of HF acid

Scott Fillery
June 24, 2006

1. Use HF acid in a specific designated area, inside a fume hood that lies outside of the heavily trafficked areas of the laboratory.
2. Never use glass equipment to measure, manipulate or store HF acid, as HF acid is an excellent etch agent of native oxides, including soda-lime-silicate glass and pyrex.
3. Designate equipment to be used in conjunction with HF acid as HF acid use only and label accordingly. That way you are always sure of where that piece of equipment has been and what is has been used for. A large label also acts to warn others of possible danger.

4. Always put on the first pair of gloves, the first pair consisting of vinyl or latex, before fitting a face shield or any other job within the HF acid handling area. That way your hands are protected against sloppy handling by previous users. Fit the face mask to your comfort before putting on the second pair of gloves, specific HF handling trionic gloves. DO NOT adjust the face mask AT ANY TIME once the second layer of gloves is on your hands.
5. Never handle cleaned HF designated equipment without a pair of gloves. Cleaned equipment may still contain traces of HF acid. Question: Do you trust previous users with your life?
6. Examine the HF handling area, any liquid on the surface should be assumed to be HF liquid and disposed of using absorbent material and placed into the HF disposal bags, NOT thrown away. This way the HF handling area should be free of liquids before you start.
7. Close the fume hood to the recommended level while handing HF acid. Fumes emanating from HF acid can cause permanent damage to your eyes and is irritating to your eyes, skin and respiratory tract. Never place your head inside the fume while using HF acid, no matter how delicate the procedure is.
8. In the event of a spill of dilute hydrofluoric acid; soak up the acid with an HF-compatible spill pillow or neutralize with lime, calcium gluconate, or another mild base; transfer material to a polyethylene container; and dispose of properly, in a labeled storage medium used only for HF acid.
9. First aid must be started within seconds in the event of contact of any form. In the event of skin contact, immediately wash with water for 15 min and remove contaminated clothing. If available, apply calcium gluconate gel. Obtain medical attention at once, and inform attending physician that injury involves HF rather than other acid. In case of eye contact, promptly wash with copious amounts of water for 5 min while holding the eyelids apart and seek medical attention at once. If HF is ingested, obtain medical attention immediately. If HF vapor is inhaled, move the person to fresh air and seek medical attention at once.
10. All samples and equipment must be washed under flowing water for a minimum of 6 minutes before being assumed clean. Equipment must be dried and kept in the HF handling area.
11. Check the HF handling area for any liquid on the bench surface and clean up accordingly.
12. Wash the trionic HF handling gloves in flowing water for 2 minutes before removing and drying.

Standard Operating Procedure
MRL TEMPO Facility

Evacuated Glass

Prepared by Joe Doyle
December 19, 2006

Evacuated Glass

This SOP applies to Rotavaps, Schlenk Lines, vacuum distillation, BET Porosimeters, moisture/contamination traps, glass diffusion pumps, the process of making evacuated glass sample capsules, and any evacuated glass. Permanently sealed dewars are also evacuated glass and must always be handled with care.

Evacuated glass can implode if the surface is flawed, if it is physically hit or shocked, or it can explode if condensed materials inside suddenly vaporize. In every case, sharp shards of glass will go flying in all directions.

It is urgent that anyone evacuating glass wear eye protection.

There is a significant amount of force on the outside of an evacuated container. A small 10" long x 2" OD cylinder will have 923 pounds of force pushing in. For the relatively thin walls of most glass vessels, the only thing preventing collapse and implosion is the evenness of the outside wall and the freedom of weak spots.

Glassware that is intended for evacuation should always be handled carefully so that it does not get scratched or chipped.

Glassware that is to be evacuated must always be inspected before every use to insure that it does not have any cracks or chips that would be weak spots that would invite implosion. Cracked or chipped glass is to be marked and removed from vacuum service.

Whenever possible the evacuated glass should have an external layer to prevent shards flying in an implosion. This layer might be plastic mesh or tape. Black electrical tape is a classic choice.

Standard Operating Procedure
MRL TEMPO Facility

Enclosed Glass with Cryogenic Cooling

Prepared by Joe Doyle
December 19, 2006

Cryo-cooling of enclosed glass has all the hazards of evacuated glass and all of the precautions of the Evacuated Glass SOP apply.

Cryogenic cooling adds another serious and unexpected hazard: oxygen (or other process gas) can condense at liquid nitrogen temperatures and collect in the apparatus if there is any leak whatsoever. If the apparatus is allowed to warm to room temperature the condensate will rapidly vaporize but if there is no way for it to vent, pressure will build until the glass explodes.

If there are small leaks on an evacuated system, then air will come in. If the system is at liquid nitrogen temperature, oxygen in the air can condense into a blue liquid. This can buildup without the operator being aware of either the leak or the liquid oxygen buildup.

Other process gasses or reaction products may also condense into liquids or solids and may explode if they are allowed to become gas too quickly.

Everyone using glass vessels chilled to cryogenic temperatures must be vigilant checking for leaks and watching for condensate, especially oxygen from air.

Any vessel that has been cryogenically cold and evacuated, must only be warmed slowly and should be vented while warming so that if anything has condensed inside it can evaporate slowly.

Other hazards of cryogens are skin damage due to cold and embrittlement of normally flexible materials.

Start Up Procedure
evacuate system
load cryogens
run process

Shutdown
remove from cryogen while evacuated
allow to warm to room temperature with vacuum pump running or large valve open
disconnect and vent

Never
warm a cold closed flask by running it under running water

MRL TEMPO Facility SOP
Cryogenics
August 27, 2009
Jaya Nolt

Cryogenics: Hazards and Safety

Burns

Direct skin contact causes severe frost bite and prolonged exposure may cause permanent injury. Do not bring your finger-tips into close proximity with cryogenics. Safety glasses and gloves are required whenever handling or transporting cryogenics. Wear clothing that covers your body, close toed shoes, long pants, long sleeves and/or a lab coat. Use tongs for freezing samples in liquid nitrogen. Use a suitable ladle for scooping up liquid nitrogen rather than holding a beaker with your hands for this purpose.

If liquid nitrogen or helium contacts skin or eyes, frozen tissues should be flooded or soaked with tepid water (only about 105-115 F / 41-46 C) -- DO NOT USE HOT WATER. Cryogenic burns that result in blistering or deep tissue freezing should be seen promptly by a physician. Liquid helium is colder then nitrogen and can cause more serious burns. Contact 9-911 if a serious burn occurs.

Asphyxiation

One liter of liquid nitrogen produces ~700 volumes of nitrogen gas. If liquid nitrogen is not stored in a well-ventilated area, the concentration of nitrogen gas may build up to the point where it displaces oxygen in the room air and may cause asphyxiation. Even small spills can be severe in a confined space. In the event of a large spill evacuate the area immediately.

Oxygen Condensation

Cold traps or open-mouth dewars containing liquid nitrogen can condense oxygen in the surrounding air. Such oxygen enrichment may result in increased flammability and explosion hazards. The nitrogen container must be handled as if it contained liquid oxygen. No sparks or flames should be allowed in or near the area.

Handling

The lids of liquid nitrogen containers should be loose fitting to allow nitrogen gas to escape, rather than allowing the build-up of pressure within the storage vessel. NEVER place cryogenics in a sealed or glass container.
Carry liquid nitrogen in a suitable thermally insulated container fitted with a bucket-like handle. Use a container with a small surface area for the liquid nitrogen – this will minimize the likelihood of spillage should the container be bumped whilst being carried. Although small foam eskies are often used as a cheap vessel for carrying liquid nitrogen within buildings, bumps are likely to cause spills, particularly if the volume carried is in the liter range.

If there is a serious problem with a liquid nitrogen tank (i.e., serious leaking, icing up, loud creaking or metal straining noises), contact Joe Doyle (jdoyle@mrl.ucsb.edu, x7925)

Drawing and Handling Liquid Nitrogen

September 3, 2009
Jaya Nolt

This applies to MRL participants and to people using MRL equipment who are transferring liquid nitrogen from a large dewar.

Primary Safety Issues

There is a lot of energy in the pressurized liquid nitrogen and much more energy when the liquid evaporates. This energy can cause splashing and strange unexpected things to happen. The most important thing when handling LN2 is to wear the correct personal protective equipment so that if it splashes on you, you will be protected.

Secondary Safety issues

-Liquid oxygen condensation
-sudden pressure and explosion from condensed air pulled into a vacuum from a small leak.
-asphyxiation

Procedure to Transfer into un-pressurized (open) dewar

Only dispense liquid N_2 into approved containers, NO GLASS!
Place the transfer line into your dewar and then open (not all the way) the *Liquid Valve* on the tank.
Fill slowly to minimize loss and sudden splashing.

Procedure to transfer to pressurized dewar

1.) Release pressure in your tank by opening the *Vent* port, again making sure a glove is attached to muffle the noise.
2.) Connect vacuum transfer line from the *liquid* port (tighten with wrench) to your tank *liquid* port (keep loose).
3.) Slightly open *liquid* port on the full tank to purge vacuum line. Once it has been purged then tighten the *liquid* port (with wrench) on your tank.
4.) Completely open both ports. Keep the *Vent* port open on your tank.
5.) Keep an eye on the tank, the level gauges are notoriously inaccurate.

Once transfer is complete, close all the ports, disconnect the transfer line.

Links to Cryogenic Related Websites

http://www.vet.ed.ac.uk/pvs/COPhanLiqNitVBS.htm

http://www.darwinawards.com/personal/personal2000-25.html
guy swallows liquid nitrogen and ends up in hospital

The material safety data sheet for liquid nitrogen is available from the Chem Alert II database, on-line from http://www.usyd.edu.au/risk/course-notes/chemalert.shtml

The University has Guidelines for Transporting Liquefied Gases at http://www.usyd.edu.au/risk/policies/ohs/liqgas.shtml

MRL TEMPO Facility SOP
Use of "Particularly Hazardous Substances"

Date of last revision to SOP: September 2009,
Based on an EH&S Document

PARTICULARLY HAZARDOUS SUBSTANCES (PHS) Include
Select Carcinogens
http://ehs.ucsb.edu/units/labsfty/labrsc/chemistry/lschphazsubstance.htm#Carcinogens
Reproductive Toxins
http://ehs.ucsb.edu/units/labsfty/labrsc/chemistry/lschphazsubstance.htm#Reprotoxins
Highly Acute Toxins
http://ehs.ucsb.edu/units/labsfty/labrsc/chemistry/lschphazsubstance.htm#Acutetoxins

1. Approval Required
- Joe Doyle must approve materials, procedure, and protective measures before work.

2. Personal Protective Equipment
- Protective eyewear such as safety glasses, goggles or face shields. The latter should be used when handling corrosive materials in large quantities (e.g. > 1 gallon).

Gloves: Be sure to use a glove that protests against the materials being used.
Glove Reference Chart
http://ehs.ucsb.edu/units/labsfty/labrsc/lsglove.htm

- Lab coats, particularly when using liquid PHS and/or PHS that are readily absorbed through the skin.

3. Engineering/Ventilation Controls
- Volatile, or dust/aerosol-producing PHS must be used in a fume hood or glove box,

4. Any Special Chemical Handling, Storage, Cleanup or Disposal Requirements
- PHS must be stored in completely-sealed containers. Although hood storage of chemicals is generally discouraged, volatile PHS can be stored in a fume hood if deemed necessary.

- Spills of PHS must be completely cleaned up. Spills that can not be safely and completely handled by lab personnel must be reported to EH&S for assistance.

- Like all chemical wastes, disposal of PHS must be done through EH&S. No PHS, or other chemical wastes can go into the sewer system, trash or be allowed to freely evaporate.

SOP: Pyrophoric Materials

Joe Doyle August 26, 2009

Pyrophoric materials are not to be used in the lab until the researcher has studied the safe handling of these materials and obtained both the specific permission of their faculty advisor and the permission of the Lab Supervisor (Joe Doyle).

Campus EH&S has created an excellent reference about the use, storage, and disposal of pyrophoric materials called UCSB EH&S LABORATORY SAFETY FACT SHEET #34, Pyrophoric Organolithium Reagents. It is excerpted below. The complete original is available at the EH&S web page.

Procedures for Safe Use of Pyrophoric Reagents

Scope

Storage, transfer and use of pyrophoric reagents, especially organolithium compounds.

Hazards

In general these materials are pyrophoric; they ignite spontaneously when exposed to air. This is the primary hazard and reagents must be handled so as to rigorously exclude air/moisture. They all tend to be toxic and come dissolved in a flammable solvent. Other common hazards include corrosivity, teratogenicity, water reactivity, peroxide formation, along with damage to the liver, kidneys, and central nervous system.

On 12/29/2008 a UCLA lab employee, wearing nitrile gloves, safety glasses but no a lab coat, with three months of work experience in this lab was transferring an aliquot of t-butyllithium in pentane when the syringe plunger popped out or was pulled out of the syringe barrel. The employee was splashed with the pyrophoric and flammable solution; upon contact with air the mixture immediately caught fire. The fire ignited the gloves and a sweater she wore. She suffered 3rd degree burns to 40% of her body and died about three weeks later.

Controlling the Hazards

Pyrophorics users must be thoroughly-trained in proper lab technique and working alone with pyrophorics is strongly discouraged. BEFORE working with pyrophoric reagents, read the relevant Material Safety Data Sheets (MSDS) and understand the hazards. The MSDS must be reviewed before using an unfamiliar chemical and periodically as a reminder.

Set up your work in a laboratory fume hood or glove box and ALWAYS wear the appropriate personal protective equipment. Minimize the quantity of pyrophoric reagents used and stored. The use of smaller syringes is encouraged. If handling more than 20 ml of sample - one should use a cannula for transfer or use a 20 ml syringe repeatedly.

Personal Protective Equipment (PPE)

Eye Protection

• Chemical Splash goggles or safety glasses that meet the ANSI Z.87.1 1989 standard must be worn whenever handling pyrophoric chemicals. Ordinary prescription glasses will NOT provide adequate protection unless they also meet this standard. When there is the potential for splashes, goggles must be worn, and when appropriate, a face shield added.

• A face shield is required any time there is a risk of explosion, large splash hazard or a highly exothermic reaction. All manipulations of pyrophoric chemicals which pose this risk should occur in a fume hood with the sash in the lowest feasible position. Portable shields, which provide protection to all laboratory occupants, are acceptable.

Skin Protection

• Gloves must be worn when handling pyrophoric chemicals. Nitrile gloves should be adequate for handling most of these in general laboratory settings but they are combustible. Be sure to use adequate protection to prevent skin exposures. Sigma-Aldrich recommends the use of nitrile gloves underneath neoprene gloves.

• *A lab coat or apron must be worn* but nylon and polyester are easily ignited materials and should not be used. Special fire-resistant lab coats made from Nomex are recommended for labs using these reagents routinely.

Equipment and Notification

• Have the proper equipment and the phone number for the Police (9-911) readily available for any emergencies.

Designated Area

Eyewash

• Suitable facilities for quick drenching or flushing of the eyes should be within 10 seconds travel time for immediate emergency use. Bottle type eyewash stations are not acceptable.

Safety Shower

• Pyrophoric work areas must be setup within 10 seconds travel time from a safety or drench shower where pyrophoric chemicals are used.

Fume Hood

• Many pyrophoric chemicals release noxious or flammable gases and should be handled in a laboratory hood. In addition, some pyrophoric materials are stored under flammable solvent, therefore the use of a fume hood (or glove box) is required to prevent the release of flammable vapors into the laboratory.

Glove (dry) box

• Glove boxes are an excellent device to control pyrophoric chemicals when inert or dry atmospheres are required.

Important Steps to Follow

Handling pyrophoric Reagents

• By using proper syringe techniques, these reagents can be handled easily in the laboratory.

Transferring Pyrophoric Reagents with Syringe

• In a fume hood or glove box, clamp the reagent bottle to prevent it from moving.
• Clamp/secure the receiving vessel too.
• After flushing the syringe with inert gas, depress the plunger and insert the syringe into the Sure/Seal bottle with the tip of the needle below the level of the liquid.
• Secure the syringe so if the plunger blows out of the body it, and the contents will not impact anyone (aim it toward the back of the containment).
• Set the inert gas source to the lowest pressure that will work, preferably less than 3 PSI. Then insert a needle from an inert gas source carefully keeping the tip of the needle above the level of the liquid.
• Gently open the inert gas flow control valve to slowly add nitrogen gas into the Sure/Seal bottle.
• This will allow the liquid to slowly fill the syringe (up to 100mL) as shown in Fig. 2A. Pulling the plunger causes gas bubbles.
• Let nitrogen pressure push the plunger to reduce bubbles. Excess reagent and entrained bubbles are then forced back into the reagent bottle as shown in Fig. 2B.
• The desired volume of reagent in the syringe is quickly transferred to the reaction apparatus by puncturing a rubber septum as illustrated in Fig. 2C.

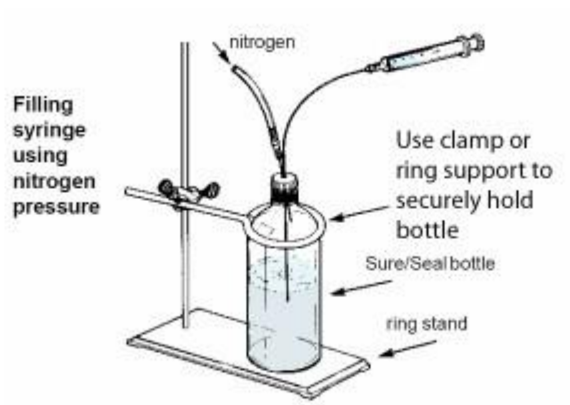


Fig.2A Filling syringe using nitrogen pressure

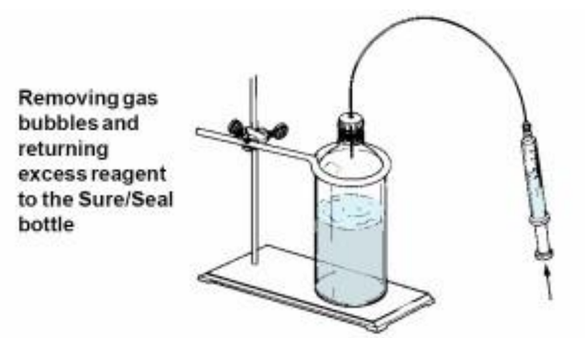


Fig. 2B Removing gas bubbles and returning excess reagent to the Sure/ Seal bottle

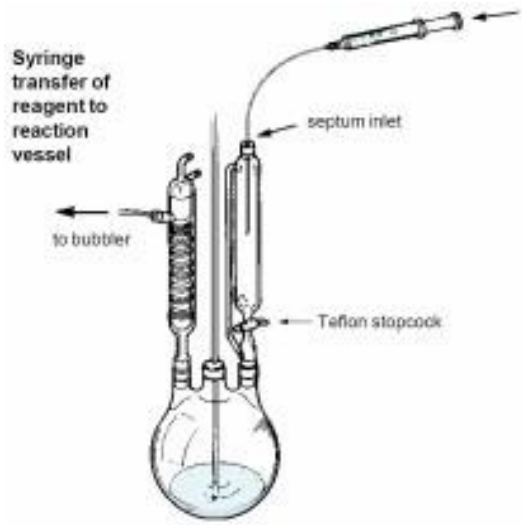


Fig. 2C Syringe transfer of reagent to reaction vessel

Transferring Pyrophoric Reagents with a Double-Tipped Needle

- The double-tipped needle technique is recommended when transferring 50 mL or more.
- Pressurize the Sure/Seal bottle with nitrogen and then insert the double-tipped needle through the septum into the headspace above the reagent. Nitrogen will pass through the needle. Insert the other end through the septum at the calibrated addition funnel on the reaction apparatus. Push the needle into the liquid in the Sure/ Seal reagent bottle and transfer the desired volume. Then withdraw the needle to above the liquid level. Allow nitrogen to flush the needle. Remove the needle first from the reaction apparatus and then from the reagent bottle. (Fig. 3A)
- For an exact measured transfer, convey from the Sure/Seal bottle to a dry nitrogen flushed graduated cylinder fitted with a double-inlet adapter (Fig. 3B). Transfer the desired quantity and then remove the needle from the Sure/Seal bottle and insert it through the septum on the reaction apparatus. Apply nitrogen pressure as before and the measured quantity of reagent is added to the reaction flask.
- To control flow rate, fit a Luer lock syringe valve between two long needles as shown in (Fig. 3C).

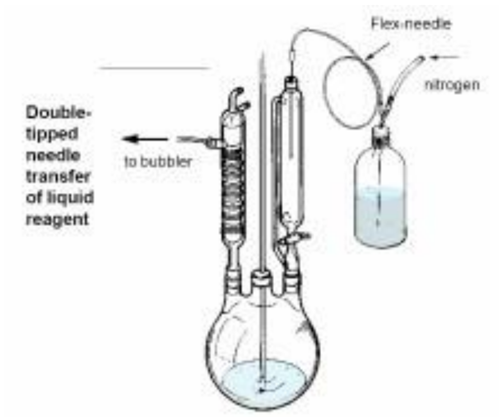


Fig. 3A Double-tipped needle transfer of liquid reagent

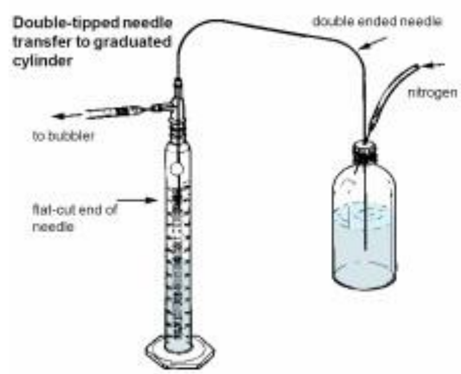


Fig. 3B Double-tipped needle transfer to graduated cylinder

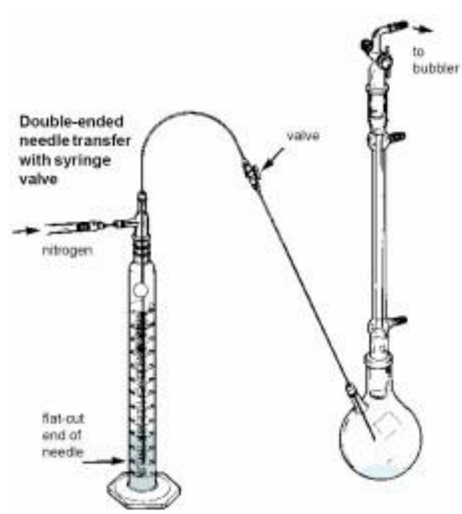


Fig. 3C Double-ended needle transfer with syringe valve

Storage

• Pyrophoric chemicals should be stored under an atmosphere of inert gas or under kerosene as appropriate.
• Avoid areas with heat/flames, oxidizers, and water sources.
• Containers carrying pyrophoric materials must be clearly labeled with the correct chemical name and hazard warning.
• For storage prepare a storage vessel with a septum filled with an inert gas
• Select a septum that fits snugly into the neck of the vessel
• Dry any new empty containers thoroughly
• Insert septum into neck in a way that prevents atmosphere from entering the clean dry (or reagent filled) flask.
• Insert a needle to vent the flask and quickly inject inert gas through a second needle to maintain a blanket of dry inert gas above the reactive reagent.

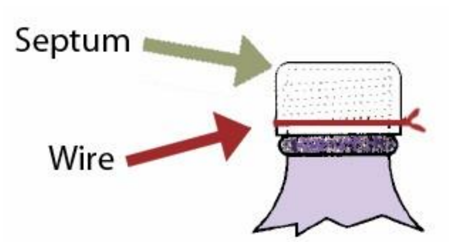


Fig. 4A Septa wired to vessel

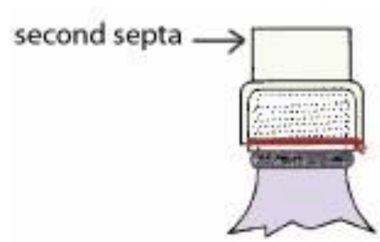


Fig. 4B For long-term storage, use a second septa

• For long-term storage, the septum should be secured with a copper wire (figure 4A).

• For extra protection a second same-sized septa (sans holes) can be placed over the first (figure 4b).

• Use parafilm around the outer septa and (obviously) remove the parafilm and outer septa before accessing the reagent through the primary septa

Disposal of Pyrophoric Reagents

• A container with any residue of pyrophoric materials should never be left open to the atmosphere.
• Any unused or unwanted pyrophoric materials must be destroyed by transferring the materials to an appropriate reaction flask for hydrolysis and/or neutralization with adequate cooling.
• The essentially empty container should be rinsed three times with an inert dry solvent; this rinse solvent must also be neutralized or hydrolyzed.
• After the container is triple-rinsed, it should be left open in back of a hood or atmosphere at a safe location for at least a week. After the week, the container should then be rinsed 3 times again.

Disposal of Pyrophoric Contaminated Materials

• All materials that are contaminated with pyrophoric chemicals should be disposed of as

hazardous waste.
• Alert EH&S for any wastes contaminated by pyrophoric chemicals.
• The contaminated waste should not be left overnight in the open laboratory but must be properly contained to prevent fires.

Emergency Procedures

Spill

· Powdered lime should be used to completely smother and cover any spill that occurs.
· A container of powdered lime should be kept within arm's length when working with a pyrophoric material.
· If anyone is exposed, or on fire, wash with copious amounts of water.
· The recommended fire extinguisher is a standard dry powder (ABC) type. Class D extinguishers are recommended for combustible solid metal fires (e.g., sodium, LAH), but not for organolithium reagents.

• Call 9-911 for emergency assistance

More Information

UCSB EH&S LABORATORY SAFETY FACT SHEET #34 on Pyrophoric Reagents was created principally the Sigma-Aldrich Technical Bulletins and these websites:

www.brandeis.edu/ehs/labs/pyrophoric.html

Advanced Practical Organic Chemistry by J. Leonard, B. Lygo, and G. Procter esp. pages 76-98

Images and advice from Sigma-Aldrich Technical Bulletins AL-134 and AL-164 at: http://www.sigmaaldrich.com/chemistry/aldrich-chemistry/tech-bulletins/tech-bulletin-numbers.html

Use of Hydrogen Gas For Pore Volume Measurement SOP

July 12, 2006
Joe Doyle

Cylinder will be kept upright with welded link chain in a gas cabinet so that it cannot fall over in an earthquake.

Only metal tubing and swagelock fittings are to be used to deliver hydrogen to the Porosimeter.

The pressure regulator used shall normally be set at 20 PSIG and shall be engineered never to exceed 100 PSIG.

All waste hydrogen gas is to be vented to the laboratory fume hood exhaust air stream.

Nano Materials SOP

December 19, 2006
Joe Doyle

Little is known about the safety or environmental consequence of nano materials. It is known that they can have properties quite different from the same compounds on the macro scale. Some nano materials have been shown to be problematic.

Possible problems are compounded by the exceedingly large surface areas of nano particles. This makes them much more bio available. Their small size makes them more likely to get airborne and accidentally pulled into the lungs.

Until more is known, people working with nano materials, especially if in particulate form, should treat them with caution and respect. In particular, they should not be allowed to be suspended in air where they could be inhaled.

Campus EH&S has a new (9/09) Fact Sheet on Engineered Nano Materials. It is part of the CHP as Appendix C.

SOP
Shipping Chemical Samples

Include a letter that states (only if true!)

•Samples are mineral samples and not medical samples
•Samples are for a collaboration and are not part of a sale. There is no invoice.
•Samples are not flammable, explosive, or volatile. They are not harmful unless ingested.

From PDF at UCSB EHS Hazardous Waste Web Page
http://www.ehs.ucsb.edu/units/hw/hw.html

Shipping of hazardous materials (chemical, biological, or radiological) within and outside the U.S. is highly regulated. Violations may subject the shipper to fines and or prosecution by appropriate federal authorities. You need to receive special training in hazardous materials shipping regulations if you handle (receive, package, offer, or ship) these materials for transportation purposes.

•All UCSB employees that receive, package, offer, or ship hazardous materials must be trained to be in compliance with the Hazardous Materials Regulations in 49 CFR, Part172, Subpart H.

• All radiological packages must be shipped by EH&S. Contact the Radiation Safety Program (x-7255, x-7256) for assistance.

• EH&S should be notified of all off-site shipments of hazardous materials.

• A 24-hour emergency response number must be included with all hazardous material shipments.

• EH&S will not ship chemical or biological packages for you, but will provide guidance and assist you with the packaging requirements. Contact the Hazardous Materials Program for chemical packages (x-3293, x-7705) or Biological Safety Program for biological packages (x-8894) for assistance.

• Your Department's receiving/shipping may also be a good resource.

• If you are personally transporting hazardous materials off campus you must comply with the Material of Trade regulations found in 49 CFR 173.6.

SECTION II (2): UCSB POLCIES, PROCEDURES AND RESOURCES (revised March 2013)

Directions to Laboratory Personnel

This section of the document is now provided to laboratories in an electronic version only, rather than a hard copy – Web address provided below. Please remove older versions of Sec. II from your binder and replace with this single page. In contrast, Section I of the document (lab-specific portion) will temporarily remain as hard copies within the binder.

Web address for Section II:

http://ehs.ucsb.edu/units/labsfty/labrsc/chemistry/CHP%20Pages/Sec.II.2013.pdf

Per Cal-OSHA requirements, this document needs to be reviewed and updated ***annually****. Therefore, we ask that this section NOT be printed out as a hard copy, as it becomes very difficult to locate hundreds of hard copies across the campus when the next update needs to occur. Questions can be directed to David.Vandenberg@ehs.ucsb.edu, or for the following departments to Moretto@chem.ucsb.edu : Chemistry & Biochemistry, Materials, Electrical and Computer Engineering, Chemical Engineering*

SECTION III (3):
REGULATORY FRAMEWORK
(revised March 2013)

Directions to the Laboratory

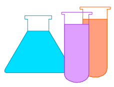
This section of the document is now provided to laboratories only in an electronic version, rather than a hard copy – Web address provided below. Please remove older versions of Sec. III from your binder and replace with this single page. In contrast, Section I of the document (lab-specific portion) will temporarily remain as hard copies within the binder.

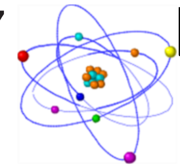
Web address for Section III:

http://www.ehs.ucsb.edu/units/labsfty/labrsc/manuals/section_III.pdf

Per Cal-OSHA requirements, this document needs to be reviewed and updated ***annually****. Therefore, we ask that this section NOT be printed out as a hard copy, as it becomes very difficult to locate hundreds of hard copies across the campus when the next update needs to occur. Questions can be directed to David.Vandenberg@ehs.ucsb.edu, or for the following departments to Moretto@chem.ucsb.edu : Chemistry & Biochemistry, Materials, Electrical and Computer Engineering, Chemical Engineering*

LABORATORY SAFETY FACT SHEET #17





Peroxides and Distillations

Extractions

Before pouring a liquid into a separatory funnel, make sure the stopcock is closed and has been lubricated. Use a stirring rod to direct the flow of the liquid being poured. Keep a beaker under the funnel in the event the stopcock comes open unexpectedly. Do not attempt to extract a solution until it is cooler than the boiling point of the extractant. When a volatile solvent is used, the unstoppered separatory funnel should first be swirled to allow some mixing. Shake with a swirl holding the stopper in place and immediately open the stopcock. Repeat until it is evident that there is no excessive pressure. Swirl again as the funnel is racked, immediately remove the stopper, and separate when appropriate.

Distillations

Ethers must never be distilled unless known to be free of peroxides. Most ethers, including cyclic ethers, form dangerously explosive peroxides on exposure to air and light.

What are organic peroxides?

Organic peroxides are a class of compounds that have unusual stability problems that make them among the most hazardous substances found in the laboratory. The lack of stability is due to the presence of an oxidation and reduction center within the same molecule.

R-O-O-R

where, R = organic side chains and O-O = Peroxo bridge

As a class, organic peroxides are considered to be powerful explosives and are sensitive to heat, friction, impact, light, as well as to strong oxidizing and reducing agents. Peroxide- formers react with oxygen even at low concentrations to form peroxy compounds. The instability of the molecule (R-O-O-R) can cause auto-decomposition simply by bumping or jarring the container, addition of heat, light, or opening the cap. The risk associated with the peroxide increases if the peroxide crystallizes or becomes concentrated by evaporation or distillation. Peroxide crystals may form on the container plug or the threads of the cap and detonate as a result of twisting the lid.

Classes of Peroxide Formers

Aldehydes

Ethers - especially cyclic ethers and those containing primary and secondary alcohol groups

Compounds containing benzylic hydrogen atoms (particularly if the hydrogens are on tertiary carbon atoms)

Compounds containing the allylic structure, including most alkenes.

Vinyl and vinylidene compounds.

Preventing Formation of Organic Peroxides

No single method of inhibition of peroxide formation is suitable for all peroxide formers. Use of different inhibitors is discussed in the literature (0.001 to 0.01% hydroquinone, 4-tert-butylcatechol (TBC) or 2,6-di-tert-butyl-p-methylphenol (BHT)); however, limiting size of container and regular testing (every 3 months) and disposal is probably more effective (and certainly easier) for managing peroxide formation.

Ethers and other organic peroxide formers should be stored in cans, amber bottles, or other opaque containers, and ideally under a blanket of inert gas. It is preferable to use small containers that can be completely emptied rather than take small amounts from a large container over time. Containers of ether and other peroxide-forming chemicals should be marked with the date they are opened, and marked with the date of required disposal.

Common laboratory chemicals that form peroxides during storage include:

Acetal	Decalin	Dimethyl ether	Methylcyclopentane	Tetralin
Butadiene	Diacetylene	Divinyl acetylene	Potassium metal	Vinyl acetate
Cumene	Dicyclopentadiene	Ethyl ether	Sodium amide	Vinyl actylene
Cyclohexene	Diethylene glycol	Ethylene glycol dimethyl ether	Styrene	Vinyl choride
Cyclooctene	Diisopropyl ether	Isopropyl ether	Tetrahydrofuran	Vinyl ethers
Decahydronaphthalene	Dioxane	Methyl acetylene	Tetrahydronaphthalene	Vinylidene chloride

Storing Peroxide Formers

Mark on containers of time-sensitive materials both the date of receipt and the date the container is first opened. Time-sensitive materials should be marked with a tag to make them easily identified. No materials should be used or tested after the manufacturers' expiration date unless evidence of current stability has been obtained via direct testing prior to the expiration date.

NOTE: If material is old (> 1 year past label expiration date) then minimize handling and DO NOT OPEN OR ATTEMPT TO TEST! Call EHS (x-3293) to request special disposal for this item. Isolate the container from possible inadvertent use until picked up. If the material is very old or shows evidence of conversion to a hazardous status (i.e.,
crystalline materials in/under cap of ethers), do not move the container!

Peroxide Detection Tests

From *Prudent Practices in the Laboratory: Handling and Disposal of Chemicals*, 1995: The following tests will detect most (but not all) peroxy compounds and all hyperperoxides. Results of peroxide detection tests must be indicated on the container/tag with test date, test results/method, and initials of the authorized person conducting the test. NOTE: These tests should not be used for testing materials potentially contaminated with inorganic peroxides (i.e., potassium).

Option 1. Add 1-3 ml of the liquid to be tested to an equal volume of acetic acid, add a few drops of 5% potassium iodide (KI) solution and shake. The appearance of a yellow to brown color indicates the presence of peroxides.

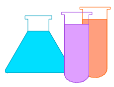
Option 2. Addition of 1 ml of a freshly prepared 10% KI and 10 ml of an organic solution in a 25 ml glass cylinder should produce a yellow color if peroxides are present.

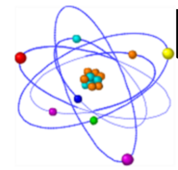
Option 3. Add 0.5 ml of the liquid to be tested to a mixture of 1 ml of 10% KI solution and 0.5 ml of dilute hydrochloric acid to which a few drops of starch solution have been added just before the test. The presence of a blue-black color within a minute indicates the presence of peroxides.

Option 4. Peroxide test strips that turn an indicative color in the presence of peroxides. Take care to follow manufacturer instructions for effective detection. In general, the strips must be air dried until the solvent evaporates and then exposed to moisture for proper operation.

For further information contact the EH&S Laboratory Safety Specialist at x-4899

LABORATORY SAFETY FACT SHEET #36





Engineered Nanomaterials: Guidelines for Safe Research Practices

Introduction

This document provides environmental, health and safety information to researchers working with engineered nanoparticles, and should be formally incorporated as a Standard Operating Procedure into each laboratory's Chemical Hygiene Plan binder (Cal-OSHA requirement).   Researchers are encouraged to customize this document as appropriate to their operation. Given the evolving knowledge base regarding health effects of nanoparticles, this fact sheet may be updated.

Nanoparticles are particles that have at least one dimension between 1-100 nanometers. Particles in this size range have always been present in Earth's air. Nanoparticles may be naturally occurring (such as in volcanic ash), produced as unintentional byproducts (such as in auto emissions) or intentionally created or "engineered." These very small particles often possess radically different properties than larger particles of the same composition, making them of interest to researchers and of potential benefit to society. This fact sheet focuses on lab practices researchers should follow to protect themselves from the hazards of engineered nanoparticles.

Nanoparticles can be spheres, rods, tubes, and other geometric shapes. The small particles may be bound to surfaces or substrates, put into solution or suspension, attached to a polymer, or in a few cases handled as a dry powder. Various nanoparticles can be created in the laboratory under experimental procedures, and some can be purchased from commercial vendors. In most research, the amount of material used is small, generally less than a gram.

Only limited information is currently available on the toxicity of a few types of nanoparticles. It is believed that some engineered nanoparticles may present health effects following exposure, based in part on air pollution studies that show smaller particles get deep into the lungs and can cause human illness. However, laboratory research most commonly involves handling nanoparticles in liquid solutions or other forms that do not become easily airborne, and even free-formed nanoparticles tend to agglomerate to a larger size.

When research involves work with engineered nanoparticles for which no toxicity data is yet available, it is prudent to assume the nanoparticles may be toxic, and to handle the nanoparticles using the laboratory safety techniques outlined below.

Potential Routes of Occupational Exposure to Researchers

There are four possible routes of workplace exposure to nanoparticles: inhalation, ingestion, skin absorption, and injection.

Inhalation. Respiratory absorption of airborne nanoparticles may occur through the mucosal lining of the trachea or bronchioles, or the alveolus of the lungs. Because of their tiny size, certain nanoparticles appear to penetrate deep into the lungs and may translocate to other organs following pathways not demonstrated in studies with larger particles. Thus, whenever possible, nanoparticles are to be handled in a form that is not easily made airborne, such as in solution or on a substrate.

Skin absorption. In some cases nanoparticles have been shown to migrate through skin and be circulated in the body. If the particle is carcinogenic or allergenic, even tiny quantities may be biologically significant. Skin contact can occur during the handling of liquid suspensions of nanoparticles or dry powders. Skin absorption is much less likely for solid bound, or matrixed, nanomaterials.

Ingestion. As with any material, ingestion can occur if good hygiene practices are not followed. Once ingested, some types of nanoparticles might be absorbed and transported within the body by the circulatory system.

Injection. Exposure by accidental injection (skin puncture) is also a potential route of exposure, especially when working with animals or needles.

Laboratory Safety Guidelines for Handling Engineered Nanoparticles

The current practices for working with engineered nanoparticles safely are essentially the same as one would use when working with any research chemical of unknown toxicity.

1. Wear double gloves (preferably nitrile gloves), safety glasses or goggles, and appropriate protective clothing. The gloves will help prevent skin exposure and reduce the chances of accidental injection by needle, or animal bite. Outer gloves should always be removed inside the hood or under the influence of local exhaust ventilation and placed into a sealed bag. This will prevent the particles from becoming airborne. Place Tacki-Mat at the exit to reduce the likelihood of spreading nanoparticles.

2. All personnel participating in research involving nanoscale materials need to be briefed on the potential hazards of the research activity, as well as on proper techniques for handling nanoparticles. The contents of this Fact Sheet can serve as a useful component of this training. As with all safety training, written records need to be maintained to indicate who has been trained on this topic.

3. To prevent ingestion, eating and drinking and chewing gum are not allowed in laboratories, except perhaps in designated areas.

4. When purchasing commercially available nanoscale materials, be sure to obtain the Material Safety Data Sheet (MSDS) and to review the information in the MSDS with all persons who will be working with the material. Note, however, that given the lack of extensive data on nanoparticles, the information on an MSDS may be more descriptive of the properties of the bulk material.

5. In some cases, the manufacture of nanomaterials involves the use of chemicals that are known to be hazardous. Be sure to consider the hazards of the precursor materials when evaluating the process hazard or final product. Users of any chemicals should make themselves familiar with the known chemical hazards by reading the MSDS or other hazard literature.

6. To minimize airborne release of engineered nanoparticles to the environment, nanoparticles are to be handled in solutions, or attached to substrates so that dry material is not released. Where this is not possible, nanoscale materials should be handled with engineering controls such as a HEPA-filtered local capture hood or glove box. If neither is available, work should be performed inside a laboratory fume hood. HEPA-filtered local capture systems should be located as close to the possible source of nanoparticles as possible, and the installation must be properly engineered to maintain adequate ventilation capture.

7. Use fume exhaust hoods to expel any nanoparticles from tube furnaces or chemical reaction vessels. Do not exhaust aerosols containing engineered nanoparticles inside buildings.

8. If you must work outside of a ventilated area with nanomaterials that could become airborne, wear a respirator with NIOSH-approved filters that are rated as N-, R- or P-100 (HEPA). EH&S will work with researchers to provide the most appropriate type of respirator.

9. Lab equipment and exhaust systems used with nanoscale materials should be wet wiped and HEPA vacuumed prior to repair, disposal, or reuse. Construction/maintenance crews should contact EH&S for assistance.

10. Spills of engineered nanoparticles are to be cleaned up right away.
    a. The person cleaning up should wear double nitrile gloves and either vacuum up the area with a HEPA-filtered vacuum or wet wipe the area with towels, or combination of the two.
    b. For spills that might result in airborne nanoparticles, proper respiratory protection should be worn (see item 8 above). For assistance with cleaning up any chemical spill contact EH&S.
    c. Do not brush or sweep spilled/dried nanoparticles.
    d. Place Tacki-Mat at the exit to reduce the likelihood of spreading nanoparticles.

11. Work surfaces should be wet-wiped regularly – daily is recommended. Because many engineered nanoparticles are not visible to the naked eye, surface contamination may not be obvious. Alternatively, disposable bench paper can be used.

12. All waste nanoparticles should be treated as unwanted hazardous "toxic" materials unless they are known to be non-hazardous. Dispose of and transport waste nanoparticles in solution according to hazardous waste procedures for the solvent. If you have questions on how to dispose a specific nanoparticle waste, call EH&S for more information.

For more information on Health and Safety of Nanotechnology visit the following web sites:

University of Wisconsin –Madison
(http://www.nsec.wisc.edu/NanoRisks/NS--NanoRisks.php)

National Institute of Occupational Safety and Health
(http://www.cdc.gov/niosh/topics/nanotech/)

National Nanotechnology Initiative (http://www.nano.gov/)

EPA (http://www.epa.gov/oppt/nano/nano-facts.htm)

Woodrow Wilson International Center for Scholars (http://nanotechproject.org/)

LABORATORY SAFETY FACT SHEET #7

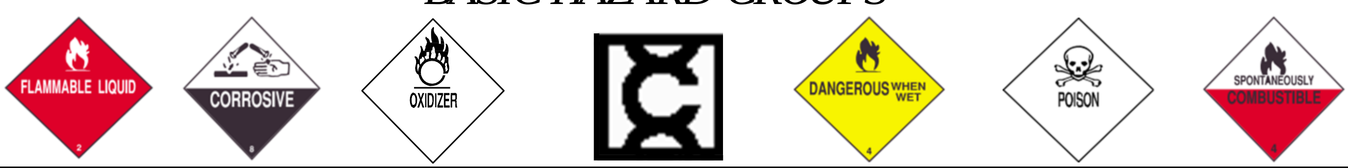
SAFE STORAGE OF CHEMICALS

INTRODUCTION: If incompatible chemicals are inadvertently mixed a fire, explosion, or toxic release can easily occur. In earthquake-prone areas like Santa Barbara, it is particularly vital that chemicals be stored safely. Take steps now to prevent damage to your facility, or harm to lab personnel.

Below are some basic guidelines for chemical storage. Note however, that chemicals can often fall into more than one hazard category and therefore the chemical label and/or Material Data Safety Sheet (MSDS-*see below*) should be reviewed for specific storage requirements. Separate chemicals by adequate distance, or preferably by using physical barriers (e.g. storage cabinets). Avoid using the fume hood for chemical storage - this practice may interfere with the proper air flow of the hood. For especially dangerous materials, use a secondary container (e.g. plastic tub) large enough to contain a spill of the largest container.

Chemicals should be disposed based on - but not limited to - the following criteria: material has exceeded it's shelf life; the cap is deteriorating or the container is leaking; the container has inadequate hazard information; material is waste (by law all chemical wastes must be disposed of within one year).

BASIC HAZARD GROUPS



Flammables | *Corrosives* | *Oxidizers* | *Carcinogens* | *Water Reactives* | *Toxics* | *Pyrophorics*

With the wide variety of chemicals used in laboratories, the list below is prioritized for materials that are *COMMONLY* used in a research laboratory. This chart indicates the most obvious chemical incompatibilities, and provides a segregation plan. For more specific chemical incompatibility information, please consult the manufacturer's MSDS, available at http://www.ucmsds.com, or contact EH&S at 893-8243.

ACIDS

Acetic Acid
eChromic Acid
Hydrochloric Acid
Hydrofluoric Acid
eNitric Acid
Phosphoric Acid
Sulfuric Acid

eIndicates strong oxidizing acids, store per oxidizers section

Storage Precautions:

[Store bottles on low shelf areas, or in acid cabinets.
[Segregate oxidizing acids from organic acids, AND flammable materials.
[Segregate acids from bases, AND from active metals such as sodium, potassium, etc.
[Segregate acids from chemicals which could generate toxic gases such as sodium cyanide, iron sulfide, etc.

BASES

Ammonium Hydroxide
Potassium Hydroxide
Sodium Hydroxide

Storage Precautions:

[Separate bases from acids.
[Store bottles on low shelf areas, or in acid cabinets.

FLAMMABLES-fuels are reducing agents

Acetone	Ethyl Acetate	Isopropyl Alcohol	Toluene
Benzene	Ethyl Ether	Methanol	Xylene
Cyclohexane	Gasoline	Propanol	
Ethanol	Hexane	Tetrahydrofuran	

StoragePrecautions:

[Store in approved flammable storage cabinet(s) (required if there is more than 10 gallons in the lab).
[Separate from oxidizing acids and oxidizers.
[Keep away from any source of ignition(flames, localized heat or sparks).
[Use only "flammable storage" (desparked) refrigerators or freezers.

OXIDIZERS-react violently with organics.

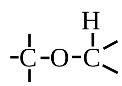
Solids	*Liquids*
Calcium Hypochlorite	Bromine
Ferric Chloride	Hydrogen Peroxide
Iodine	Nitric Acid
Nitrates, Salts of	Perchloric Acid
Peroxides, Salts of	Chromic Acid
Potassium Ferricyanide	
Sodium Nitrite	

Storage Precautions:

[Keep away from flammables, organic solvents, and other combustible materials (i.e. paper, wood, etc.).
[Keep away from reducing agents.
[Store in a cool, dry place.

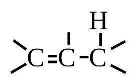
PEROXIDE-FORMING CHEMICALS-peroxides can be explosive and shock-sensitive.

Ethers and acetals with 〈-hydrogen



(e.g. ethyl ether, tetrahydrofuran)

Alkenes with allylic hydrogen



(e.g. cyclohexene)

For a more complete list of these materials visit our website at http://ehs.ucsb.edu/resources/MANUALS/l-s/sect6.html

Storage Precautions:

[Dispose before expected date of initial peroxide formation.
[Label containers with receiving, opening, and disposal dates.
[Store in airtight containers in a dark, cool, and dry place.

PYROPHORIC SUBSTANCES-spontaneously ignite in air.

Some finely divided metals
Some organoaluminum compounds ($LiAlH_4$, $Al(CH_3)_3$)
Silane
ePhosphorus, Yellow

ePhosphorus, yellow should be stored and cut under water

StoragePrecautions:

[Rigorously exclude air and water from container.
[Store away from flammables.
[Store in a cool, dry place.

WATER REACTIVE CHEMICALS-reacts violently with water to yield flammable or toxic gases.

Solids	*Liquids*
Calcium Carbide	Phosphorus Trichloride
eLithium	Thionyl Chloride
Magnesium	
ePotassium	
eSodium	

eLithium, Potassium, and Sodium should be stored under Kerosene or Minerial Oil

StoragePrecautions:

[Rigorously avoid exposure to water and air.
[Store away from flammables
[Store in a cool, dry place.

HIGHLY TOXICS, CARCINOGENS, REPRODUCTIVE TOXINS

These chemicals can be very hazardous by themselves, or in combination with other chemicals. If they are easily inhaled, (gases and volatile liquids) then they are particularly hazardous. Suspected human carcinogens should also be stored as highly toxic. Lists of these materials are provided on our website: http://ehs.ucsb.edu/resources/MANUALS/l-s/sect6.html

Gases - Store in a gas cabinet or other ventilated cabinet

(Ex) Chlorine Fluorine Hydrogen chloride Nitric Oxide

Liquids - Seal tightly and store in a ventilated cabinet apart from incompatibles. Use secondary containment (e.g. plastic tub) to contain any spills.

(Ex) Formaldehyde Carbon disulfide Mercury Nickel carbonyl Cyanide solutions

Solids -Store away from incompatibles (usually acids) that would release toxic gas upon contact.

(Ex) Cyanides, Salts of Sulfides