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Laboratory Safety Manual and Chemical Hygiene Plan

MRL Energy Research/Battery Characterization Facility

Manual Purpose: - this manual serves two basic purposes:

1. It is the basic laboratory safety manual for UCSB

2. Serves as the campus **Chemical Hygiene Plan (CHP***)* as required by the *California Occupational Safety and Health Administration (Cal-OSHA)*. In short, OSHA requires that a written chemical safety plan address the policies and procedures that an employer has in place to minimize the exposure of its lab employees to chemicals. Workers are required to receive documented training on their CHP. A full summary of the OSHA standard is in Sec. III.

Manual Structure:

Section I: Introduction and Lab-specific Chemical Hygiene Plan. Forms and templates for customizing your CHP with SOPs and other local information. Links to other lab safety programs.

Section II: UC & UCSB policies, procedures and resources. Summaries of key/core lab safety issues that apply to most/all laboratories. Primarily based on specific OSHA requirements.

Section III: Appendices. Includes further information on PI responsibilities, laboratory inspections, and the GHS classification system, as well as a list of particularly hazardous substances.

Section I: Laboratory-Specific Chemical Hygiene Plan: Standard Operating Procedures

Introduction:

Welcome to the MRL Battery/Energy Facility. Everyone working here has to act in a professional, safe, and environmentally responsible fashion otherwise it becomes difficult for anyone to get any work done. Nobody wants to have to clean up someone else's mess before they can begin work, so everyone needs to take care for the lab. We all need to make sure we all follow the many laws and regulations about safe work practices.

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 Battery Characterization 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 the Google Form MRL Energy Research Facility Chemical Hygiene Plan User Agreement (UCSB), which I can send to you. The link can also be found in this document.

Please remember that although the work that you may be doing is not particularly hazardous, hazardous procedures may be going on around you. Due to this, we must follow safety guidelines and wear the proper PPE for the environment we are working in.

I try as hard as I can to ensure that the lab is fully functional, as user friendly as possible, and as safe as possible. To accomplish this, I need your help. If you see any kind of safety problem, or if we are low or out of some necessary supply, or that some equipment is not working right, please send me e-mail describing the problem. E-mail is the best way to keep me up to date and to help me remember. Please let me know if there is any imminent hazard and any kind of safety problem. Never leave lab supplies, personal effects, glassware, books, or papers out in the lab except when you are actually using them.

Chemical storage space is very limited, especially inside of the glove box. Before purchasing new chemicals please check the laboratory's inventory. Besides conserving room, this will save you time and money. If you have a reagent that someone else needs, please share it with them.

From time to time we have to clean the lab. These may occur when the lab has become particularly messy, before an inspection or a tour, or at the end of the summer intern session. Everyone working in the lab should participate. With everyone's help, we will continue to perform safe and ground-breaking research at one of the top materials research facilities in the world.

Required Training:

In order to become an authorized user of the Battery Characterization Facility in MRL 1050, the individual must fulfill ALL the requirements listed below

1. Complete EH&S general laboratory safety training
2. Review MRL general safety documents
3. Be trained to operate the instruments/equipment in a safe manner
4. Addition to the Battery Lab Slack Channel (contact Ashlea Patterson- ashleapatterson@ucsb.edu to be added)
5. Review the laboratory specific Chemical Hygiene Plan (this document)
6. Document all above steps and submit records to appropriate personnel

Safety training begins with the UCSB EH&S "UC Lab Safety Fundamentals". Everyone working in the lab is required to take this course before beginning lab work. The EH&S Lab Safety class is offered online and in-Person twice quarterly. The online course and the in-person schedule are posted at: http://learningcenter.ucsb.edu

After the EH&S Lab Safety course, people working in the MRL Energy Research/Battery Characterization Lab need to read:

1. MRL Safety Information: http://www.mrl.ucsb.edu/mrl-safety-information
2. The Energy Research/Battery Characterization Facility Chemical Hygiene Plan (CHP) paper copy or online. If you have any questions, please contact Dr. Rachel Behrens, MRL 2003, phone: x5850 and e-mail: rachel@mrl.ucsb.edu.
3. To document the completion of this training step, please fill out the form below: http://www.mrl.ucsb.edu/sites/default/files/mrl_docs/forms/safety_training_form.pdf

Instrument training can be arranged by requesting training through the Facilities Billing Services (FBS: https://ucsb.fbs.io/Anon/Logon.aspx) or by emailing the technical director. This training will cover hands-on training to operate the instruments/equipment and introduction to the safety information of the laboratory.

As much as it may seem, all of the above is just the foundation of the laboratory safety training. Everyone working in the lab must do the appropriate inquiry, literature research, and thought to ensure that the specific lab work they do is performed safely. The actual preparation will vary depending on what the project will be, but will certainly include studying the chemical hazards of the materials to be used and speaking with people who have done similar work. More work may be necessary, such as reviewing any physical or electrical hazards and considering if specialized personal protective equipment is required. I am available to answer questions and to help, but you ultimately will be the one carrying out the work, so you will need to be familiar with the potential hazards.

General Laboratory Information:

Laboratory Supervisor (PI): Prof. Michael Chabinyc, 3219 Elings Hall, x4042

Laboratory Technical Director (TD): Dr. Rachel Behrens, MRL 2003, x5850

Laboratory Location(s) (Building /Rooms): MRL 1050

Department Information
Department Safety:
Alex Moretto, Research Safety Division Manager, UCSB EH&S, phone: 617.480.6630

Dr. Amanda Strom, MRL 2066F, phone: x7925 (DSR)
Sara Bard, MRL 2066E, phone: x8519 (DSR alternate)
Dr. Rachel Behrens, MRL 2003, x5850 (DSR alternate)

Location of Department Safety Bulletin Board: MRL 2042

Location of Building Emergency Assembly Point: South West corner of Engineering II.

Emergency Information:

As applicable, please provide information regarding emergency procedures and equipment specific to the lab(s) under your control. Where applicable, you may just reference the emergency contact information on your lab door placards.

- **Evacuation procedures** (e.g., close fire doors, secure certain equipment, etc.)

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.

Earthquake
During an earthquake, you should try to stand in a doorframe until all shaking has stopped and only then evacuate the building. Another option is to seek shelter under a desk.

Fire
If a fire alarm goes off you must leave the building and proceed to the Emergency assembly location (SW corner of Eng II). **Do not use the elevators.**

For reporting a fire, fire alarm pull stations are located on the walls of the main hallways. Per SB County Fire and UCSB campus policy, all fires must be reported to 9-911 immediately even if the fire is out. If a fire extinguisher is used it must be reported as it will need to be replaced.

- **First-aid kit** (e.g., location, contents, maintenance responsibility, etc.)
  Two first-aid kits are located near the hallway exit in MRL 1050.

It is responsibility of the Lab TD to maintain the first aid kits.

In the Event of an Injury
Per campus policy, all **significant injuries must be documented** through Employee Injury & Illness Reporting (IIR). This is necessary for potential reimbursement for personal medical costs, or Worker's Compensation Claims. For directions on how to create a claim, visit: https://www.ehs.ucsb.edu/sites/default/files/docs/wc/RSS-IIR-Injury-Illness-Reporting-User-Guide.pdf

Serious Injuries
If the situation is **immediately threatening to life or limb**, get emergency care, e.g. by calling 9-911 from any campus phone. This is preferred to taking an injured person directly to the Goleta Valley Cottage Hospital Emergency Room or Sansum Occupation Medical Clinic, where they may not be seen or treated for a long time if they don't arrive in an ambulance.

Other Injuries
Students – For serious injuries not threatening to life or limb, undergraduates and graduate students who are not "employed" by UCSB, may be treated at Student Health Services.

UCSB Employees – Staff, Faculty, Graduate Student employees, undergraduate employees, Post Doc, and other UCSB employees with serious work related injuries, which are not threatening to life or limb, should use an Urgent Care Facility (that UC has contracted with, such as Goleta Valley Cottage Hospital Emergency Room or Sansum Occupation Medical Clinic) for walk-in treatment.

- **Spill cleanup materials** (e.g., location, contents, maintenance, procedures, etc.)

Chemical spill cleanup kits are kept in 1050 MRL under the cabinet by the FBS log in computer. Please contact technical director if more supplies are needed.

- **Laboratory monitors or alarms** (e.g., operation, response, maintenance, etc.)

There is a glovebox environment monitor on each of the gloveboxes in 1050. If the alarm is going off on the glovebox monitor, please contact the glovebox emergency contact immediately to remedy.
There are no other lab monitors except for low air flow monitors on the fume hoods. These are to be maintained by campus Physical Facilities.

- ***Other Lab-specific emergency information***

The MRL Emergency Operations Plan (see also appendix A): http://www.mrl.ucsb.edu/mrl-emergency-operations-plan

Per campus policy, all significant injuries must be documented via the Employee Injury & Illness Reporting (IIR), as soon as possible. This is necessary for potential reimbursement for personal medical costs, or Worker's Compensation Claims¸ or in serious cases reporting to Cal-OSHA

Per SB County Fire and campus policy, all fires must be reported to 9-911 immediately – even if the fire is out. This is particularly true if there is use of an extinguisher (must be replaced); an injury; or property damage.

Health and Safety References:

Here is a short list of the title and location of health and safety reference materials (reference books, Safety Data Sheets, experimental protocols, etc.) associated with the lab which employees may use to aid them in their work.

	Reference	Location
1.	Laboratory Safety Program/Chemical Hygiene Plan	online only
2.	Paper Copies of (M)SDS	none
3.	Electronic Copies of select (M)SDS	computers in 1050 MRL
4.	Merck Index	2003 MRL
5.	Handbook of Chemistry and Physics	2003 MRL
6.	Fisher Safety- Safety Products Reference Manual	2003 MRL

The book entitled: *Prudent Practices in the Laboratory* by the National Research Council is widely considered to be a definitive reference. It can be purchased, but is also available free on-line in a searchable format. It is recommended that all lab workers have ready access to this important reference.

Safety Data Sheets (formerly known as MSDS). Per OSHA, all lab users must know:
a) what an SDS is,
b) SDS relevance to their health and safety,
c) how to readily access them*
These issues are all covered in the EH&S lab safety orientation program.

*Labs are encouraged to maintain hard copies of their own SDS for the hazardous chemicals they routinely use, or at minimum, have this link bookmarked by all individuals in the lab.

General Laboratory and Chemical Safety:

In addition to the guidelines provided below, it is recommended that you go over the Laboratory Safety Self-Checklist in Appendix E. This document is also available on the web at:
https://www.ehs.ucsb.edu/sites/default/files/docs/ls/Lab_Self_InspectionChecklist_web_July2022.pdf

General

- No storage of food and drinks in the MRL labs, which have an extensive list of chemicals.
- Smoking is prohibited anywhere on campus.
- Do not block lab aisles with chairs, stools, or equipment.
- Observe all posted signs and instructions.

Electrical Safety

- Do not use damaged electrical cords. Do not chain extension cords/ power strips.
- Do not leave extension outlets or power strips on the floor where it may be flooded.

Gas cylinder handling

- All compressed gas cylinders need to be secured with welded link metal chain on the top and bottom of the cylinder, so they do not fall over in an earthquake.
- When moving a gas cylinder, place the safety cap over the valve before undoing the chains securing the cylinder.
- Use the special dolly for gas cylinders that is kept in the MRL gas cage (across the little parking lot on the ocean side of the building).

Chemical Safety

- For transport of larger (> 1 L) glass bottles with chemicals, use designated carriers or plastic buckets.
- ***Keep chemicals stored in the appropriate cabinets or designated storage rooms when not in use (NOT IN FUME HOODS).*** Only obtain an amount to keep your test or research going, like a one day/week supply. This will free up lab bench space and, if you do have a spill, it will minimize the amount of chemical released.
- Put away all reagents, samples, and personal materials when not in use.
- ***Keep the lids on chemical containers.*** This sounds obvious but it will effectively reduce the possibility of a spill and reduce any fumes released into your lab, and it's the law.
- ***Label all containers.*** Make sure there are no unidentified containers; reagents, samples, drying papers with sample, or crucibles/boats with samples. Label all material by chemical name (Not just initials or formulas).

Cleaning the lab

- Properly dispose of old or unwanted chemicals or any unnecessary items.
- Damp wipe all bench-tops until clean and in particular areas near weighing stations.
- Clean up inside fume hoods.
- Look inside all cabinets for leftover waste and any storage hazards.
- Recycle paper and cardboard properly.
- Unused or spare equipment should be stored in a designated storage room/area.
- Equipment or furniture should not block walkways, electrical panels, or emergency eyewash or showers.
- Check emergency egress path is maintained (minimum exit pathway in rooms is 28 inches).
- Verify the lab(s) are clean, organized and anything else required to make lab look professional.
- Check for trip and slip hazards (oil leaks from pumps, electrical cords or hoses across walking path).

Fume Hoods

- Always work with the sash at the level of the arrow sticker and closed when not used. Your hood should be producing a face velocity of 100-120 ft/min. EH&S tests your hood and posts the arrow tickers at the proper sash level.

- Many newer hoods are equipped with the airflow monitor and alarm to warn you if the air velocity is too low. If the alarm engages, lower the sash slightly until the alarm stops. If your alarm sounds consistently this indicates a real problem- call Amanda Strom (ext. 7925) or EH&S (805-893-4899).
- Store the bare minimum of equipment and chemicals in your hood. Excess materials will block the air flow and reduce performance significantly.
- Chemicals should not be stored in the fume hood- most fires and explosions occur in the hood during chemical manipulations.
- Keep the lab windows and doors closed. Draft from open windows and doors can significantly affect your hood's performance.

Disposal of Sharps

- Lab glassware not contaminated by hazardous materials (eq. Pasteur pipettes) can be placed into labeled "Broken Glass" trash box or other sturdy container. When full, dispose of contents into the red sharps bins outside by the trash and recycling dumpsters.
- Sharps contaminated with chemicals should be placed into a sharps container and labeled as "Sharps contaminated with (chemical name)" and sent to EH&S for disposal. (See Appendix C: Laboratory Sharps Disposal)

Chemical Waste Disposal

- Hazardous waste regulations are stringent and penalties for violations can be severe. Santa Barbara County inspects UCSB labs for compliance on a regular basis.
- Store chemical waste in a designated area. Label area as, "Hazardous Waste Storage Area".
- Store chemicals in containers compatible with, and durable enough for, the waste. Liquid must be in screw top containers. Do not overfill container, allow for expansion.
- Labeling- identify waste by proper chemical name.
- Deface existing labels when reusing containers.
- Label and date containers when the first drop of waste is added. Hazardous waste shall be disposed within 9 months. Labels are available in all science storerooms and in the laboratory.
- Chemicals may not be disposed in a regular trash, sink disposal, or allowed to evaporate. (See Appendix C: Chemical Waste Disposal)

Chemical Spills

- For detailed instructions, please refer to Chemical Spills in Section II of this document
- Clean up a spill using the proper equipment (please use spill kit contents in 1050 MRL).
- Cal EH&S 24-hour line 805-893-3194 if necessary.

Safe storage of chemicals

- In earthquake-prone areas like Santa Barbara, it is particularly vital that chemicals be stored safely. Use a secondary container (plastic tub) large enough to contain a spill of the largest container).

- Store or waste using the following criteria: Flammables, Corrosives, Oxidizers, Carcinogens, Water reactive, Toxics, Pyrophorics. (Globally Harmonized System (GHS) Pictograms and their meanings can be found in Section III)

- Acids- store bottles in the acid cabinets, segregate oxidizing acids from organic acids, and flammable materials. (E.g. nitric acid must be stored in a separate bin.)
- Segregate acids from bases, and from active metals such as sodium, etc.
- Segregate acids from chemicals which could generate toxic gases such as sodium cyanide, etc.
- Flammable store in approved storage cabinet. Keep away from any source of ignition (flame, heat or sparks).
- Oxidizers-react violently with organics. Keep away from flammables, from reducing agents, store in a cool, dry place.
- Pyrophoric substances-spontaneously ignite in air. Some organo-aluminum compounds, silane, divided metals, phosphorus yellow. Rigorously exclude air and water from container. Store away from flammables, store in a cool dry place.

Refrigerators and Freezers

- There is one fridge/freezer in the lab (MRL 1050). It is designed for the storage of flammables but is not owned by the Energy Facility, so chemical storage for Energy Facility users should be done so in their own lab space.
- *No food or drink must be stored in any of the fridges in the lab.*
- Minimize the time that this freezer is opened, as moisture from the air rapidly condenses on it.

Personal Protective Equipment (PPE)

Pants and Closed-Toe Footwear

- Pants that come down to your ankle and closed-toe footwear must be worn in the lab at all times!

Lab coats

Laboratory coats are required to be worn while working on, or adjacent to, all hazardous chemicals, biological or unsealed radiological materials. It is imperative to consider the nature of the work performed when choosing a lab coat. In general, you must wear a flame-resistant (blue) lab coat when working in the main MRL Labs, including MRL 1050.

- Note that “standard” lab coats are typically made from a polyester/cotton mix and are not suitable for work with flammables.
- Laboratory coats must not be worn outside of a laboratory unless the individual is traveling directly to an adjacent laboratory work area.
- Each person should have their personal lab coat, which they will receive as part of the PPE provided by UCSB to new lab workers. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.
- Lab coats must not be cleaned at home nor in public laundry facilities. Rather, a professional cleaning

service must be used. See the information at the lab coat laundering drop off station in the MRL (near the restrooms on the first floor). Any clothing that becomes contaminated with hazardous materials must be decontaminated before it leaves the laboratory. If a lab coat is very heavily contaminated, it should be packaged safely and disposed of as hazardous waste.

Gloves

- Protective gloves must be worn while utilizing any hazardous chemical, biological or unsealed radiological material. These gloves must be appropriate for the material being used and conditions under which such use takes place (e.g., extreme cold).
- A glove recycling program has just been initiated to help reduce our environmental impact. There will be specific receptacles in each of the participating labs that are only for *uncontaminated* and powder-free nitrile, latex, and vinyl gloves
    - ***Uncontaminated*** can include light contamination from non-toxic chemicals as long as they are rinsed and mostly dry prior to disposal
    - **DO NOT** include any gloves that should be considered biohazardous or hazardous waste
- Educate yourself as to which chemicals the gloves you are using are resistant and (im)permeable to. You may be unpleasantly surprised. However, there is a tradeoff between chemical resistance of gloves and the dexterity they allow. The increased dexterity offered by thinner gloves may offset their poorer chemical resistance. After all, it is safest not to spill anything in the first place! The latex or nitrile (purple) single- use examination gloves readily available in our lab are a good choice for most powders and for aqueous solutions, as well as simple alcohols (such as methanol, ethanol, and isopropanol) and diethyl ether.
- EH&S has a page with information on gloves, including links to several reference charts with compiled data on chemical resistance of lab gloves at: https://www.ehs.ucsb.edu/programs-services/lab-safety-chemical-hygiene/labsafety-chp/sec2/selecting-proper-gloves

Spills and Exposure to Hazardous Chemicals

For all incidents in which injury has occurred or may be imminent, follow these steps: Emergency procedure

- Administer First Aid as needed
- Warn people in the area
- Evacuate the area if needed
- Notify 9-911
- Notify the Departmental Safety Representative (DSR) or alternate DSR as soon as feasible

Exposure to Chemicals First Aid

If a chemical splashes in someone's eye, rinse with copious amounts of water for a minimum of **10 minutes**. Small burns or splashes with corrosive chemicals on the skin are also flushed with water for five minutes. Use the emergency showers if a person’s hair or clothing has caught fire (rolling the person on the floor is another option for extinguishing flames) or in the event of a larger spill of a hazardous chemical on skin or clothing.

Some Best Lab Practices

These make the lab a better place to work for everybody:

- Put your reagents back in the proper storage location at the end of every workday
- Refill squirt bottles when they are nearly empty
- Get new solvent bottles from the storeroom before running out
- Label all your bottles/flasks with proper chemical names. Preferably use pencil on tags, not a marker
- Label all running reactions, especially reactions running overnight
- Scales/Scale areas: Keep the scale and surrounding area clean. After weighing, take all your stuff with you, and completely clean up any spills you made. Put a note on the scale if you need the tare to remain set; only do this if you will return after a short time (< 15 minutes), else record the tare weight.
- Close the regulator on gas tanks once you are done using them
- Don't 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 everyday
- Before purchasing new chemicals be sure to check if any of the required reagents are available in the lab via the chemical inventory

Leaving Lab

On completion of your work in the Energy Research/Battery Characterization Facility you will need make a way for next person and put your gear back into circulation. Be sure to do following:

- Let the technical director know when you are leaving a few weeks before you leave.
- Dispose any samples that do not need to be archived. Transfer your samples to your supervisor's labs.
- Empty out all drawers and lab space which you have been using. Any equipment and glassware that has been assigned to you should be placed back into circulation.
- Any hazardous/chemical waste you have should be labeled and then placed into the waste chemical area.
- Any reagents in your possession should go back to someone in your group.
- Return keys to Sylvia Vogel (MRL 2066G). Let your PI or lab manager know how to reach you.

Ten Commandments of Safety:

- Thou shalt wear thy safety glasses, as with all other personal protective equipment that shall be required.
- 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.
- Thou shalt not store thy chemicals alphabetically, but only compatibles upon compatibles.
- Thou shalt not smoke within the laboratory. Neither shall thou confuse the laboratory with a place of nourishment.
- 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.

- Thou shalt label all of thy chemicals and samples with thy name and the date. Thou shalt never leave unknown and unmarked bottles of chemicals or samples for thy neighbor.
- Thou shalt purchase only the minimum chemicals for thy needs. Thou shalt covet thy neighbor's chemicals and thou shalt share thy chemicals with thy neighbor.
- Thou shalt not have multiple extension cords in series.
- Thou shalt not deliver oxygen in plastic tubing, lest the fires of Hell visit upon thy experiment.
- Thou shalt know what thou are doing and about the hazards thou faceth. Thou shalt never toil in the laboratory until thou hast studied and trained about safe work practices.

Identifying Chemical Hazards

Every lab worker has the responsibility to learn about and understand the hazards of the chemicals they use before starting to use those chemicals. Do not assume that a material is harmless just because you haven't heard otherwise. Many chemicals are harmful, and some chemicals are mostly harmless by themselves but very dangerous in combination with certain other chemicals.

Besides talking to other people in the lab that use these materials (but don't assume that they have done their homework, even if they are senior to you!!), these are some resources:

- Safety Data Sheets (SDS). Widely available online (see the Resources section of this CHP), they are especially useful for mixtures, but also for reagents. SDS were intended to be a one-stop source of chemical hazard information, but they frequently are not very specific, not as succinct as one would like, and make everything sound extremely hazardous because they err on the side of caution e.g. for personal protective measures.
- Laboratory Chemical Safety Summaries (LCSS) are available for far fewer compounds, but more succinct and useful. Sources for LCSS are on the MRL Safety webpage (see the Resources section of this CHP).
- The Merck Index is a compendium that has relevant information for many common chemicals. A copy of the Merck Index is kept in room 2003.
- See also the Resources section of this CHP

Communicating Safety and other Lab Issues

You should report any procedure, condition or situation that you consider to be unsafe, or potentially unsafe. Except for an actual emergency, the best way to communicate a safety problem is to write an email to the DSR or alternate DSRs, depending on the nature of the problem. Forms for anonymously reporting a hazardous condition or practice (Hazard reporting forms) are available at the MRL Safety Corner bulletin board in room 2042 if you feel that reporting the hazard in the usual manner would jeopardize you in some way. If supplies are missing, a hazardous waste pickup needs to be arranged, or a piece of equipment is not working, contact the technical director.

Background: Standard Operating Procedures

Per Cal/OHA regulations, a Chemical Hygiene Plan must include Standard Operating Procedures (SOPs) that pertain to the storage and use of the hazardous chemicals in your laboratory. The following steps should be followed in order to complete this requirement.

1. **Determine which SOPs you need:** Compare your chemical inventory and lab processes against the UCSB Standard Operating Procedures library. There, SOP templates are available for most hazard classes, a number of specific chemicals, and certain laboratory processes. Additionally, a blank SOP template is available. If you require an SOP template that is not available in the library, feel free to contact EH&S for assistance.
2. **Customize the SOP templates you selected:** Sections in red on the template must be filled out to reflect the details specific to your research group. Specifically, the *Laboratory Specific Information* section must be filled out to generate a Cal/OSHA compliant SOP. This can be very detailed if so desired, but in many cases, this can be satisfied by just a few sentences.
3. **Add completed SOPs to the end of this document.**
4. **PI completes the Certification Page below**
5. **Laboratory workers review the SOPs, as well as the UCSB Chemical Hygiene Plan, and sign off on the Laboratory Worker Training Record page below.**

Standard Operating Procedure Library Certification Page

PI/Laboratory Supervisor Name:

Applicable Laboratory Locations (Building, Room #):

PI/Laboratory Supervisor Signature:

I certify that I have reviewed and approve the attached Laboratory Specific Chemical Hygiene Plan with Standard Operating Procedures for laboratory operations being conducted in the locations noted above.

Signature:____

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Laboratory Safety Manual and Chemical Hygiene Plan

MRL Energy Research/Battery Characterization Facility

Laboratory Worker Training Record:

UCSB Chemical Hygiene Plan and Laboratory Specific Chemical Hygiene Plan with Standard Operating Procedures

PI/Laboratory Supervisor:____________________________________

The following laboratory workers have reviewed and understand the contents of the UCSB Chemical Hygiene Plan and this laboratory's Laboratory Specific Chemical Hygiene Plan with Standard Operating Procedures:

Name (Please print)	**Signature**	**Date**

Standard Operating Procedure General Information

The following apply to all chemicals unless specifically noted in the customized SOP. Any additional requirements will also be noted in the SOP:

Engineering Controls:

Fume Hood: All chemicals should be transferred and used in an annually certified chemical fume hood, in an effort to keep exposures as low as possible. If your specific protocol does not permit the handling of certain chemicals in a fume hood, contact EH&S to determine whether additional *respiratory protection* and/or *specialized local ventilation* is warranted.

Safety Shielding: Shielding is required of there is significant risk of explosion, implosion or splash. This risk can be due to the nature of the chemicals involved, the reaction conditions (temperature, pressure) or scale.

Storage: All chemicals should be stored upright, tightly sealed, and in a cool, dry, and well ventilated space. Segregate incompatible materials from each other based on information from the SDS and as described in the Chemical Hygiene Plan. All containers must be labeled in English with the name of the material (no formulas or acronyms) and all relevant hazard statements (e.g. corrosive, flammable, etc.)

First Aid and Emergencies:

Fire: DO NOT use water to put out a fire. A class ABC fire extinguisher can be used to extinguish most laboratory fires. If pyrophoric or water reactive metals are involved in the fire, use a class D extinguisher.

Spills: Evacuate the location where the spill occurred. Notify others in the areas of the spill, including your supervisor. Notify EH&S in case of personal exposure. If the spill is <1 Liter and of a known material of limited toxicity, flammability and volatility, post someone just outside of the spill area, don proper PPE, and clean the spill following the procedure in the Chemical Hygiene Plan Chapter 4 and the UCSB Emergency Flip Chart. Otherwise, call EH&S at X3194, or 911 if there is immediate danger to life, health or property.

Exposures:

Skin or eye contact: Remove contaminated clothing and accessories. Flush affected area with water for 15 minutes. If symptoms persist, get medical attention.

Inhalation: Move person to fresh air. If symptoms persist, get medical attention.

Ingestion: Rinse mouth with water. If symptoms persist, get medical attention.

Decontamination: Wear proper PPE, decontaminate equipment and benchtops using soap and water. Dispose of contaminated paper towels as hazardous waste, following the UCSB hazardous waste procedures described in the UCSB Chemical Hygiene Plan.

Waste Disposal: Refer to Chapter 3 of the UCSB Chemical Hygiene Plan.

Battery Characterization Facility Specific Standard Operating Procedures (SOPs) Following This Page.

These documents are addendums to the UC Santa Barbara Chemical Hygiene Plan, and covers additional information on the safe handling and storage of the materials described beyond the practices described therein. Users must be familiar with the UC Santa Barbara Chemical Hygiene Plan before utilizing these SOPs. The SOPs included in this CHP may not cover all of the hazardous material used in the Energy Research/Battery Characterization Lab. Please refer to your group's CHP for additional SOPs.

SOPs included in this section:

1. Battery Lab Glovebox
2. Lithium Hexafluorophosphate(LiPF6/NaPF6)
3. Carbonate Solvent for Li Ion Battery Electrolyte
4. Casting Electrodes for Batteries
5. Lithium Metal
6. Pyrophoric and Other Air/Water Reactive Materials Handling
7. Cell Disassembly
8. Battery Material Waste Disposal
9. Carcinogens, Reproductive Toxins and Acute Toxins
10. Dichloromethane
11. Peroxide Forming Chemicals
12. Flammables
13. Corrosives
14. Compressed Gases
15. Vacuum Systems
16. High-Pressure Reaction Vessels

Standard Operating Procedure

Battery Lab Glovebox

Hazard:

Overview	The glovebox is a contained argon (sometimes nitrogen) environment used to work with air (or water) sensitive materials. Bonking your head on the glovebox window when you forget it is there presents a physical hazard during use of the glovebox. Most of the hazards involve damaging the glovebox and samples contained
Personal Protective Equipment (PPE)	Standard PPE (gloves, lab coat, safety glasses, closed toed shoes, and long pants) must be worn during use of the glovebox. Gloves must also be worn under AND over the black glovebox gloves to protect against contaminants and sharp objects.
Engineering Controls	Consider changing the argon tank when it is below 400 psi before the weekend, and below 200 psi on a week night. Usually change every 2 weeks. Use ultra high purity argon. Close all antechambers when changing tank. **Bringing in objects through small antechambers A, C, and D** Check the run log book to ensure that the particular antechamber you want to use is not still in use. Fill chamber with argon until it reaches ambient pressure. Make sure the valve is set to static (closed) when opening antechamber doors. You may then gently open the outside antechamber door. When putting supplies in chamber, make sure they won't get stuck under the drawer! Fill out the run log book so labmates know the chamber is in use. Close chamber, leave evacuating for 10 minutes or more, then refill chamber with argon and evacuate again. Cycle at least 3 times in this way. (Chamber can also be left evacuating overnight, in which case 3 cycles is not necessary.) It is necessary to check the pressure gauge when starting to evacuate the chamber. If the pressure does not drop immediately, then at least one of the antechamber doors (outside or inside) has not been properly sealed. The inside door of the antechamber can be opened once the three cycles are complete. Once you move your items inside of the glovebox, fill out the remaining columns in the logbook so indicate to other labmates that you are done with the antechamber. Do not overtighten chamber doors – the vacuum is what seals the chamber, so there is no need to be aggressive on the doors. Leave the antechamber under active vacuum

	Putting in new supplies, taking out trash Use the big antechamber B. Flip sign on chamber B to "in use". Before you can open antechamber B, close the vacuum line (below chamber). Flood chamber with argon to increase pressure to ambient so that you can open the outside door. Open the seal and lever on outside door to open. Leave supplies evacuating overnight in chamber because glass, paper, and plastics especially, off-gas water (could also put them in the vacuum oven beforehand). Glass off-gasses less if you put in drying oven first (the oven behind glovebox is always set to drying temperature of 100 C). Similarly to the smaller antechambers, cycle at least 3 times, this time leaving the chamber evacuating for at least 30 minutes each cycle to guarantee no air gets into the glove box. After new supplies have been taken in, trash inside the chamber can be removed through chamber B. Supplies in – trash out is often done right before the glovebox catalyst is regenerated. More recently we have implemented a policy of not storing any waste in the glovebox long term. Remove trash through small antechambers when transferring out samples. **Regenerating the catalyst** Usually this is done every 6 weeks. This is a custom gas order (95% argon 5% H_2) and can take up to 2 weeks to order. One tank should last a number of regens. One regen will use approximately 300 PSI of the tank. Check the flow meter on the bottom right of box containing the catalyst (under antechamber B). (We don't actually usually go by the flow rate at the box, but instead from the regulator on the tank) Evacuate then close all antechambers (static vacuum) (there was once a strange problem where pump oil got into antechambers, though still not sure how). Turn off circulation again, then go to Menu → regeneration purifier. Answer yes to both of the next questions, however not until after double checking that the regeneration gas tank and regulator are open, and that gas is flowing (should also check the exhaust bubbler in the fumehood). Put signs up so people know regen is happening. Check exhaust lines from the box, they should be closed and routing into the fume hood. Change pump oil after regen is complete.

	Changing pump oil Use Edwards 19 ultragrade performance vacuum oil. Turn off glovebox circulation. Vacuum pump vpg → off. Use old empty oil container as waste container. Open valve on top right of pump – even though we drain from the bottom valve, this should be open to let air in to replace oil that's drained. USE A FUNNEL, and place spill pads on the floor before changing the oil. Wrench needed to open valve. Have to move pump out to access the drain, to do that need to detach the clear line (the exhaust line) and drain it. Do not detach the gray line (vacuum line) as that will let O2 into the antechambers (not a disaster if you open, circulation was off, still purge the box just in case). Take off the mist filter that the clear line was attached to by taking off the black clamp. Drain it through the black little spout. Also use an allen wrench to take the gray part of the mist filter box off and clean the inside of the box. Have paper towels and additional spill pads on hand, this is very messy. The pump is on a tray. It is a 2 person job to lift the pump off the tray, offset so that you can drain the pump from the bottom right valve. USE A FUNNEL. Pour more new oil into the top right valve to keep cleaning out the gunk (still draining pump into waste container). Do this until the oil comes out clear looking. When the oil is mostly clear, but the pump back onto the tray bolts. Close the drain valve. Fill the pump back up with oil to approximately 75% full on the sight glass. Oil levels are best measured while the pump is running, so check the oil level a second time once the pump has been restarted. Adjust the amount of oil accordingly. Put the box back on with the black clamp, then reattach the clear exhaust line. Close the top valve on the pump. Clean off floor oil with ethanol, don't go too hard or the wax will come off the floor. Make a new waste bag for all the oily rags and gloves. Function → vacuum pump back on. Let it run a bit before you turn circulation back on. Can also test that pump is working by pulling vacuum on one of the antechambers. Log the box O2 levels etc in logbook.

Emergency Procedures	**Purging the glovebox** Turn off circulation. Increase minimum pressure to 10 and max to 12. Open manual purge valve above glovebox for desired length of time. Close valve. Change pressure to minimum 3 maximum 6. Turn circulation back on. **Patching glove holes** Be extremely careful in the glove box around sharp objects, as these can easily puncture/rip/tear the gloves. By using a pair of latex gloves over the rubber gloves inside the glovebox, you can add an extra layer of protection for sharp objects. In the case of a minor puncture or tear, there should be enough positive pressure to not allow the ambient air into the glovebox, but you must move quickly in this scenario. You must immediately retrieve the tire patch kit from its spot near the glovebox. The first step is to locate the leak, which you can do by carefully listening for it in addition to feeling for a small stream of air. Once the leak is located, use something such as chalk to mark the spot so you can easily locate it. Depending on the bike tire patch kit at hand, follow directions on the package for plugging the hole as this can vary by patch kit. This is only a temporary fix of the glove to stop the immediate leak and so it is important to then follow the protocol for actual replacement of the glove. **Replacing gloves** To replace the glove, the first step is to seal off opening of the of the glove of interest. This will ensure that when the glove is removed, you will not allow any infiltration of external air into the carefully regulated glovebox environment. The o-rings and damaged glove can then be removed. After removal, you can install the new glove by folding the glove cuff inside out by a few inches. Then place the glove into the glove port and stretch the cuff onto the glove port flange. It is important that the glove stays rolled up as much as possible to prevent external air from getting into the box. Also make sure that the thumb of the glove is pointed in the appropriate direction. Stretch both of the o-rings over the glove and fit them into the grooves, ensuring no wrinkles are present. You may then remove the cover and the positive pressure from the box should inflate the glove! The O2 levels will likely spike after a glove change, so purge the box to bring them back down.

Emergency Procedures continued	**Following a power outage** After almost every power outage / surge, the fuse/circuit breaker on the glovebox circulation blower will trip. One sign that this is the case are elevated O2 and H2O readings that are fluctuating wildly. Another clue: If you turn the circulation purifier off you should hear a noise like normal, but when you turn it back on if there is no new sound then likely the circuit on the blower has tripped. There may be an error message saying as much on the glovebox touchscreen as well. Further instructions are in the manual. To reset the circulation blower: there should be a key in the red box sitting with the manual under antechamber B. Use the key to open the electrical panel at the front of the glovebox. Flip the F3 switch off and back on again. You should hear the blower turn on.
Chemical Storage and Disposal	Read the MSDS for any chemical to be used in the glovebox. Dispose of waste following standard disposal procedures for the material. Do not leave waste inside the glovebox, remove waste when you are done working and properly label it.
Lab-specific Information	Approval must be given by Ram Seshadri or Rachel Behrens after receiving training from an experienced user.

Standard Operating Procedure

Lithium Hexafluorophosphate(LiPF6/NaPF6)

Overview

Lithium/Sodium Hexafluorophosphate(LiPF6/NaPF6) is commonly used as a electrolyte salt in the lithium battery. LiPF6/NaPF6 can easily react with moisture and decompose under heating to generate corrosive and toxic products (HF, PF5). Thus handling the LiPF6 and disposing LiPF6 extremely care.

Handling Process

1. LiPF6 should always be used in Glovebox. Once bottle opened, the bottle should not be brought outside the glove box.
2. Avoid heat source and water containing chemicals.
3. When transferring open-bottle *LiPF6/NaPF6*. The bottle has to be sealed tightly before taken out from glovebox. Transfer quickly. Try to minimize the time for *LiPF6/NaPF6* to stay outside the glovebox.
4. The *LiPF6/NaPF6* waste needs to taken care. Most of the *LiPF6/NaPF6* waste will be a mixture of LiPF6 and Ester. Hence, the waste can be both flammable and corrosive. Avoid accumulate more than 10mL (liquid)/ 5g (powder) LiPF6 waste in Glove box. The LiPF6 waste SHOULD NOT be stored in glass containers.

Special Handling and Storage Concerns

Precautions for safe handling
Avoid contact with skin and eyes. Avoid inhalation of vapor or mist.
Keep away from sources of ignition – No smoking.
Take measures to prevent the buildup of electrostatic charge.

Conditions for safe storage, including any incompatibilities
Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.
Do not store in glass. Air and moisture sensitive. Hygroscopic. Handle and store under inert gas.
(TRGS 510): Flammable liquids Engineering Controls

Decontamination

Quarantine contaminated area at 75 feet (25 meters) radius from the center of contamination. Wear protective equipment and clothing. Do not touch Spilled material. Use only non-sparking tools and equipment. **Do not expose spilled material to moisture.** Seal all possible locations where contaminates might migrate into the environment. Clean up solids and place them into a waste container safe for disposing of contaminated trash. Clean up spilled liquids with vermiculite and place them into the same container. Appropriately transport contaminated material to a waste facility capable of handling contaminated materials.

Waste Management

Store separately. Never mix it with other chemical waste. Store in the plastic container. Store it in the fume hood. Avoid accumulation of the waste for large quantity (50 ml). Usually, *LiPF6/NaPF6* come with flammable electrolyte. Hence, the *LiPF6/NaPF6* waste should not be stored for more than a week. Ask for waste collection as frequently as possible.

First Aid and Emergencies

If inhaled.

If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact

Take off contaminated clothing and shoes immediately. Wash off with soap and plenty of water. Take victim immediately to hospital. Consult a physician. First treatment with **calcium gluconate paste**.

In case of eye contact

Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician. Continue rinsing eyes during transport to hospital.

If swallowed

Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

Laboratory Specific Information

Prior Approval Required

- ☒ **YES (describe): clearance with PI and training with an experienced group member should be completed and documented.**

Designated Area

- ☒ **Other (describe): only inside fume hoods or gloveboxes.**

Experimental Conditions of Use

Temperature Range: Ambient temperature
Pressure Range : Ambient pressure
Scale Range: 1 mg to 1 g scale

Standard Operating Procedure

Carbonate solvent for Li ion battery electrolyte

Overview

Carbonate solvents for Li ion battery electrolytes are ethylene carbonate (EC), diethyl carbonate (DEC), dimethyl carbonate (DMC) and ethyl methyl carbonate (EMC). They are used as solvent to dissolve electrolyte LiPF6. They are highly volatile, flammable. In contact with Air/O2 for long period can produce peroxide, which is pyrophoric.

Handling Process

1. *Ethylene carbonate (EC), diethyl carbonate (DEC), dimethyl carbonate (DMC) and ethyl methyl carbonate (EMC)* should always be used in Glovebox. Once bottle opened, the bottle should not be brought outside the glove box. Never store them in the air environment once the seal is opened.
2. **Avoid heat source and other oxidizing chemicals for both chemicals and waste.**
3. When transferring open-bottle Carbonate solvent, the bottle must be sealed tightly before taken out from glovebox. Transfer quickly. Try to minimize the time for *LiPF6/NaPF6* to stay outside the glovebox.
4. **C**arbonate solvent waste needs to be taken extreme care, because they can be **pyrophoric**. A small waste container (<50 ml) should be always in the glove box to collect the carbonate solvent when you are working with those chemicals. **Caution: most of carbonate solvent wastes come with LiPF6/NaPF6 dissolved in the solvent, in that case, plastic container instead of glass container.** Never store the carbonate solvent waste outside the glove box for more than a week. Ask for waste pick-up as soon as the waste start to accumulate outside the glove box. Isolate the waste container from other waste.

Special Handling and Storage Concerns

Precautions for safe handling

Keep away from heat, sparks, open flames or other hot surfaces. – No smoking. Keep container tightly closed. Ground or bond container and receiving equipment. Use explosion-proof electrical, ventilating, lighting, and equipment. Use only non-sparking tools. Take precautionary measures against static discharge. Wear protective gloves, eye protection and face protection.

Conditions for safe storage, including any incompatibilities

Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully
resealed and kept upright to prevent leakage.
Keep cool

Decontamination

ELIMINATE all ignition sources (no smoking, flares, sparks, or flames in immediate area). All equipment used when handling the product must be grounded. Stop leak if without risk. Ventilate the area. Absorb with an inert material and put the spilled material in an appropriate waste disposal container. Use clean non-sparking tools to collect absorbed material.

Waste Management

Store separately. Never mix it with other chemical waste. Store in the plastic container if it has LiPF6/NaPF6 solute. Store it in the fume hood. Avoid accumulation of the waste for large quantity (50 ml). The waste should not be stored for more than a week. Ask for waste collection as frequently as possible.

First Aid and Emergencies

Inhalation: Call a poison center or doctor if you feel unwell. Move victim to fresh air. Give artificial respiration if victim is not breathing. Administer oxygen if breathing is difficult. Keep victim warm and quiet. Treat symptomatically and supportively. Ensure that medical personnel are aware of the material(s) involved and take precautions to protect themselves.

Skin contact: Call a poison center or doctor if you feel unwell. Remove and isolate contaminated clothing and shoes. In case of contact with substance, immediately flush skin with running water for at least 20 minutes. Treat symptomatically and supportively. Ensure that medical personnel are aware of the material(s) involved and take precautions to protect themselves.

Eye contact: If this chemical contacts the eyes, immediately wash (irrigate) the eyes with large amounts of water, occasionally lifting the lower and upper eyelids. If eye irritation persists get medical advice/attention. Move victim to fresh air. Check for and remove any contact lenses. Keep victim warm and quiet. Treat symptomatically and supportively. Effects of exposure to substance may be delayed. Ensure that medical personnel are aware of the material(s) involved and take precautions to protect themselves.

Ingestion: Call a physician or Poison Control Center immediately. Do not use mouth-to-mouth method if victim ingested the substance; give artificial respiration with the aid of a pocket mask equipped with a one-way valve or other proper respiratory medical device. Loosen tight clothing such as a collar, tie, belt or waistband. If a person vomits place them in the recovery position so that vomit will not reenter the mouth and throat. Rinse mouth. Keep victim warm and quiet. Treat symptomatically and supportively. Ensure that medical personnel are aware of the material(s) involved and take precautions to protect themselves.

Laboratory Specific Information

Prior Approval Required

☒ YES (describe): clearance with PI and training with an experienced group member should be completed and documented.

Designated Area

☒ Other (describe): only inside fume hoods or gloveboxes.

Experimental Conditions of Use

Temperature Range: Ambient temperature
Pressure Range : Ambient pressure
Scale Range: 1 mg to 1 g scale

Standard Operating Procedure

Casting Electrodes for Batteries

Overview

Electrodes for batteries are often casted from a solution of active material (typically transition metal oxides, but could include others), carbon additives, polymer binder, and solvent (typically N-Methylpyrrolidone (NMP)) onto a current collector substrate (typically copper or aluminum films). A doctor blade is used to cast the film to the desired thickness and the film is then thoroughly dried of solvent before use in a battery.

Process:

5. Casting can be done inside a fume hood (Fume hood #2) or glovebox. Each case will be dependent on the stability of the active material in ambient air.
6. Add a suitable amount of polymer binder to the desired solvent and mix till homogenous. Mixing can be done with a mortar and pestle or FlackTek speed mixer.
7. Add the active material and carbon additive into the mixture and additional solvent if necessary. Mix until homogenous.
8. Add solvent until the mixture reaches the desired consistency.
9. Tape current collector substrate onto a glass or aluminum plate.
10. Add electrode slurry onto the current collector. The pool of slurry should be smaller in width than the doctor blade used to cast the film.
11. Set the doctor blade to the desired thickness and pull towards you at a slow and steady speed.
12. Allow the solvent to evaporate during a waiting period after casting. After most of the solvent has evaporated, place the glass or aluminum plate onto a hot plate or vacuum oven to dry.
13. Electrodes can then be punched out to the desired size and used.

All researchers handling these materials must receive thorough hands-on training and receive approval from the PI.

Special Handling and Storage Concerns

Personal Protective Equipment

- Lab coat
- Safety goggles
- Nitrile gloves

Special Storage Requirements

Store chemicals in the glovebox and in an air tight container to prevent moisture contamination.

Engineering Controls

Glovebox: as much as possible, handle all components inside a glovebox

Fume Hood: when not handling inside a glovebox, handle all components in a fume hood (Fume hood #2), preferably with an active fire suppression system in place, or with a sand bucket on hand.

Special Handling Considerations

N-Methylpyrrolidone(NMP) is easily absorbed into the skin and is a highly toxic chemical. Always use the *proper PPE when handling this chemical.* Consult material SDS for detailed hazard information.

Decontamination
Consult material SDS for detailed hazard information.

Waste Management

Wipe down all tools used with a solvent soaked kim wipe and place in a properly labelled waste bag. Use a suitable solvent to rinse all tools of electrode slurry into a waste beaker and dispose of in a properly labelled waste container.

First Aid and Emergencies

Personnel Exposure
Consult material SDS for detailed hazard information.

Laboratory Specific Information

Prior Approval Required

☒ **YES (describe): clearance with PI and training with an experienced group member should be completed and documented.**

Designated Area

☒ **Other (describe): only inside fume hoods or gloveboxes.**

Experimental Conditions of Use

Temperature Range: Ambient temperature
Pressure Range : Ambient pressure
Scale Range: 1 mg to 1 g scale

Standard Operating Procedure

Lithium Metal

Safety Contact Person	SOP prepared by Howie, 714-470-1650, howie@ucsb.edu Raphaële Clément, 805-893-4294, office MRL 3009, rclement@ucsb.edu
Hazard	Lithium is an alkali metal and is classified as water-reactive or 'dangerous when wet'. It is highly flammable and reactive with water producing flammable gases that can ignite if exposed to water. It may be harmful if ingested, inhaled, or absorbed through the skin. It can cause skin and eye burns with irreversible damage. Extreme caution is advised. Keep away from heat and sources of ignition. Gaseous by-products are extremely destructive to the tissue of the mucous membranes and upper respiratory tract, potentially leading to pulmonary edema.
Overview	Lithium metal is used as an electrode in battery research. It must always be handled in an air-free environment due to its' high reactivity. Preferably an Ar-filled glovebox since it can react with nitrogen. Lithium metal waste is generated when preparing electrodes and must be neutralized properly.
Personal Protective Equipment (PPE)	Standard personal protective equipment including closed toed shoes, a flame-retardant lab coat, nitrile gloves (underneath AND above the glovebox gloves), and safety glasses should be worn always.
Engineering Controls	All work with lithium metal should be done in an Ar environment with O_2 and H_2O levels maintained below 0.5 ppm, and in a sealed container under an inert environment or vacuum, such as a Swagelok or Coin cell. Li waste neutralization should be conducted in a chemical fume hood designated for neutralization (west wall of battery lab).
Handling, Storage and Disposal	Store in a well-ventilated, inert environment. Keep container tightly closed. Lithium metal must be handled with tools designated specifically for lithium use since it is a sticky metal and can leave residual scraps on the tool. Lithium waste should be disposed of as soon as possible by neutralizing. Before neutralizing, make sure that there is a bucket of sand available to put out Li metal fires. Lithium waste neutralizing should be done in small batches with the following protocol: 1. Lithium scaps should be removed from the glovebox and into the fumehood (Fume hood #2) or cell disassembly should be conducted in the fumehood (Fume hood #2) away from ignition sources due to H_2(g) forming.

	2. Place a small beaker into a larger beaker and fill the small beaker with an ethanol/water mixture. The solution should be in large excess of the lithium waste to be neutralized. 3. Place the lithium waste inside the beaker and loosely cover the container to prevent solution from splashing out. 4. Lithium will react with water to form LiOH(aq) + H_2 (g) and produce a corrosive solution with heat generation. 5. Once the reaction is completed, allow the beaker to cool down before handling. The reaction is completed when the bubbling stops. 6. Dispose of the solution into a properly labeled container for base waste and wash the beakers. 7. Waste is picked up in battery lab every Tuesday, but contact EH&S for a pick up if a significant amount of waste is generated.
Lab-specific Information (Required)	Use the proper safety equipment and safety protocols when working with lithium metal. If you are uncertain how to safely proceed, speak with your PI, or another appropriately trained user.
Spill and Incident Procedures	Immediately evacuate area and ensure others are aware of the spill. For large fires that cannot be managed, evacuate immediately and pull the nearest fire alarm station to evacuate the building. For small lithium fires that can be managed, use a class D fire extinguisher or sand to put out the fire. Inform your PI and or lab managers of the spill/incident.
Authorized Use	Approval must be given by your PI and documented after having been trained and supervised by an experienced user for as many uses as deemed appropriate.

Standard Operating Procedure

Pyrophoric and other Air/Water Reactive Materials Handling

Overview

Pyrophoric and other air/water reactive materials are among the most dangerous materials that lab personnel work with in the laboratory. They are all highly reactive to oxygen and/or water. Pyrophorics ignite spontaneously upon exposure to air. Water reactive chemicals react violently with water, including the moisture in air, often emitting flammable or toxic gases. The information provided below is meant to provide guidance and is not a substitute for actual hands-on training conducted by experienced laboratory personnel.

Representative examples of some pyrophoric and air/water reactive materials:

Metal-Hydrides
Sodium borohydride
Lithium borohydride
Lithium aluminum hydride
Sodium hydride
Diisobutylaluminum hydride

Metals
Potassium
Sodium
Lithium
Cesium
Francium
Rieke metals (magnesium, zinc)

Organometallics
Trimethylaluminum
Diethyl zinc
Dimethyl zinc
Butyl lithiums (all)
Grignards (all)

Non-metals
Trimethylphosphine
Fluorine
White phosphorous
Phosphine

Metal Catalysts
Raney nickel
Palladium on carbon (after solvent contact)
Platinum oxide

Metal-Alkoxides
Potassium t-butoxide
Sodium methoxide
Sodium ethoxide

Metalloid/metalloid halides
Silane
Germane
Tetrachlorosilane
Arsine
Boranes

Please review the full pyrophoric material list to check whether the material you will be working on is pyrophoric.

Special Handling and Storage Concerns

Notify the PI and Safety personnel before working with this type of materials.
Safety evaluation needs to be evaluated by strom@ucsb.edu and rachel@mrl.ucsb.edu

How to Handle Pyrophoric Reagents
An instructional video produced by
Dartmouth: https://www.youtube.com/watch?v=iLMl10X0Naw
Due to the severe hazards associated with these materials, consider using a less hazardous reagent (substitution) or a reagent in a less hazardous form (attenuation) when possible.
Examples:

- Use a hydrogen transfer agent rather than Raney nickel;
- Use *n*-BuLi rather than *t*-BuLi;
- Use NaH 60% dispersion in mineral oil rather than NaH, dry, 95%;
- Use 10% Pd on carbon (wet) rather than 10% Pd on carbon (dry);
- Use 0.1% fluorine gas rather than 99% fluorine gas;

Universal Best Practices While Working with Air and Water Reactive Materials

- Documented training is mandatory before personnel are allowed to work with pyrophoric or other air/water reactive materials.
- Inexperienced users of air/water reactive materials must be supervised while performing experiments.
- Never work with air/water reactive materials while you are alone.
- All reactions using air/water reactive materials must be performed under an inert atmosphere in a properly operating chemical fume hood, glove box, or appropriate engineering control with functional fire extinguishing devices (class ABC and D fire extinguishers) nearby.
- Have the fume hood sash lowered at all times unless actively working in the fume hood.
- Have an appropriate spill kit available and ready before beginning work. Sand, metal-x, or lime works well for smothering air/water reactive materials.
- Wear appropriate PPE (gloves, safety glasses, and lab coat; preferably flame-resistant) and avoid wearing clothes that will melt or are highly flammable.
- Clean up and remove from the work area any flammable material that is not necessary. If there is a spill of flammable material, clean out the work space thoroughly before resuming work.
- Prior to commencing work, flame or oven dry all equipment (e.g., glassware, syringe body, needles, cannula), and then cool it to ambient temperature under an inert atmosphere.
- Manipulation of air/water reactive materials should use locking mechanisms (e.g., Luer-locks) and clamped apparatus connections (e.g., keck, cable).
- Use gas/vacuum manifold systems (with multiple lines) under an inert gas and mineral oil bubblers to prevent over-pressurization. Inert-gas balloons may also be used.
- Have the reaction apparatus staged/setup and fully purged with inert gas, and keep it under low positive pressure before transferring air/water reactive materials to it.
- Use septa to seal reaction vessels as shown in the example below.

- Use ≥16 gauge needles of ~8-12" in length (24" for cannula). Wider gauge needles help prevent the plumbing from being obstructed; however wider gauge needles more readily damage the integrity of Sure/Seals.

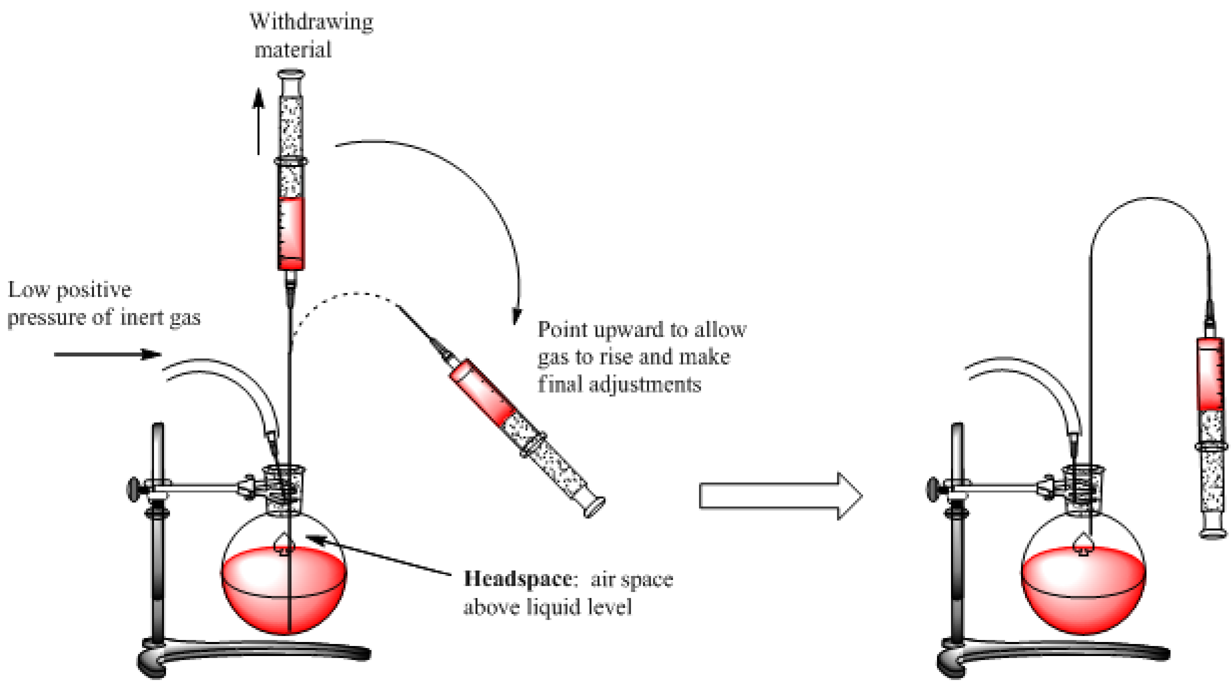
*The needle size depends on the volume of liquid being dispensed. For larger quantities, a wider bore needle (16 gauge) is preferred.

- Never use a syringe at its maximum volume. Maximum withdrawal should be ½ to 2/3rd of syringe capacity. Syringe plungers can quickly pop-out near their maximum volume. Examples;
  - If you need 5 mL of reagent, use a 10 mL syringe.
  - If you need 25 mL of reagent, use a 40 mL syringe.
  - If you need >50 mL of air/water reactive material use a cannula, double-tipped needle, or use multiple smaller withdrawals via syringe.

Examples of septa that may be required for working with air/water reactive materials:
Sealed containers with PTFE-lined septa, but it's best to work in a glove box!

Liquids Syringe Technique

1. Clamp the bottle of reagent to a solid and immovable support to prevent accidental movement. If the bottle is coming from cold storage and is stable at ambient temperature, allow it to come to ambient temperature before working with it.
2. Remove the screw cap from the reagent bottle and quickly insert a seal/septum if one is not already in place on the bottle. Insert an inert gas line needle through the septum into the headspace of the reagent to place the contents under low positive pressure of inert gas. Headspace is the gas-filled portion of the reagent bottle above the liquid.
3. Purge a syringe and locked needle with inert gas a few times.
4. With the plunger fully forward, insert the needle into the reagent bottle through the septum until the needle approaches the bottom of the reagent bottle.
5. Draw the reagent out slowly (to avoid air bubbles) with the syringe pointing downward until a small excess of reagent is withdrawn.
6. Flip the syringe so that the plunger is pointing upward and not toward the researcher and allow the gas in the syringe to rise to the tip. With the syringe still pointing upward, push on the plunger until reaching the exact volume of liquid desired and any gas bubbles are expelled.
7. With the syringe still pointing upward, bring the tip of the needle out of the liquid reagent until it is in the headspace of the reagent bottle; then slowly pull the plunger back to bring in inert gas as a head space inside the syringe. Make sure not to pull the plunger out too far.

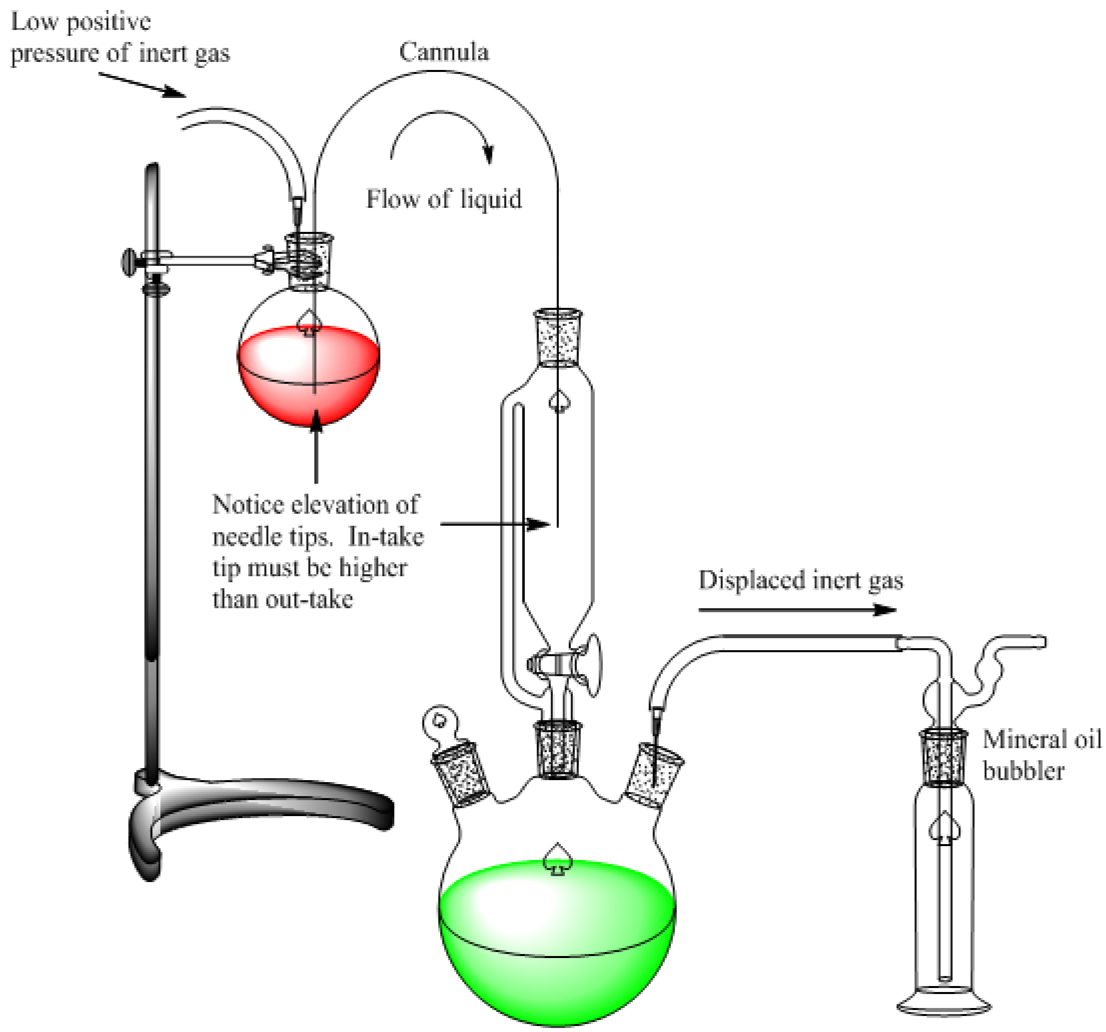


8. With the syringe still pointing upward, pull the needle out of the septum of the reagent bottle, but leave the inert gas needle in place. At this point you may see a flame or spark at the needle tip once it comes into contact with the atmosphere. Don't panic; this is a sign that the reagent is still of good quality, although the lack of a flame or spark does not mean the reagent is not active.
9. With the syringe still pointing upward, insert the needle through the septum into the headspace of the reaction apparatus that has already been set up, dried, purged, and is under inert gas. Slowly dispense the contents of the syringe by pushing the plunger of the syringe inward until it stops. Pull back on the plunger to withdraw a few mL of inert gas and remove the needle from the septum of the apparatus.
10. Rinse the syringe and needle with a solvent that is the same or compatible with the solvent in which the reactive material was dissolved. Do this by inserting the needle into a flask of the solvent (twice the volume of the syringe) and slowly withdrawing and ejecting the solvent a few times. Then rinse with water and clean as normal.
11. Once done with the reagent bottle, withdraw the inert gas needle from the reagent bottle and recap the reagent bottle. Be sure to use parafilm, bakelite caps, or other means to ensure that the air/water reactive liquid remains under inert atmosphere. Place the reagent bottle back into its appropriate storage location.

Cannula/Double-Tipped Needle Technique

All of the above mentioned precautions still apply except that the reaction apparatus should not be placed under low positive pressure of inert gas when the reagent is being transferred. Thoroughly purge the reaction apparatus with inert gas. Then remove the inert gas line inserted via the septum and connect the reaction apparatus to a mineral oil bubbler to allow displaced gas to escape (see illustration below).

1. Clamp the bottle of reagent to a solid and immovable support higher in elevation than the reaction apparatus into which you intend to transfer the reagent.
2. Remove the screw cap from the reagent bottle and insert an inert gas line needle through the septum into the headspace to place reagent contents under low positive pressure.
3. Insert the cannula/double-tipped needle into the reagent bottle through the septum so that the tip is in the headspace of the reagent bottle. Do not insert the tip into the reagent itself!
4. Allow inert gas to purge the cannula/double-tipped needle for 5-10 seconds; then insert the other tip of the cannula through the septum into the headspace of the reaction apparatus.
5. Insert the cannula tip from the headspace of the reagent bottle into the liquid itself. An immediate transfer of liquid into the reaction apparatus should start to take place.
6. Once the desired amount of liquid is transferred, pull the needle tip out of the liquid and into the headspace of the reagent bottle.
7. Remove the needle from the reagent bottle, and then immediately remove the other end from the reaction apparatus. Place the reaction apparatus under a low positive pressure of inert gas by inserting a gas line needle from the gas manifold.
8. Remove the inert gas line needle from the reagent bottle, seal it, and place it back in its appropriate storage location.
9. Clean the cannula by placing a septum over the mouth of a side-arm vacuum flask, inserting the cannula through the septum, applying vacuum to the flask via the house vacuum, and placing the other end of the cannula into a flask of compatible solvent so that the solvent is pulled through the cannula.

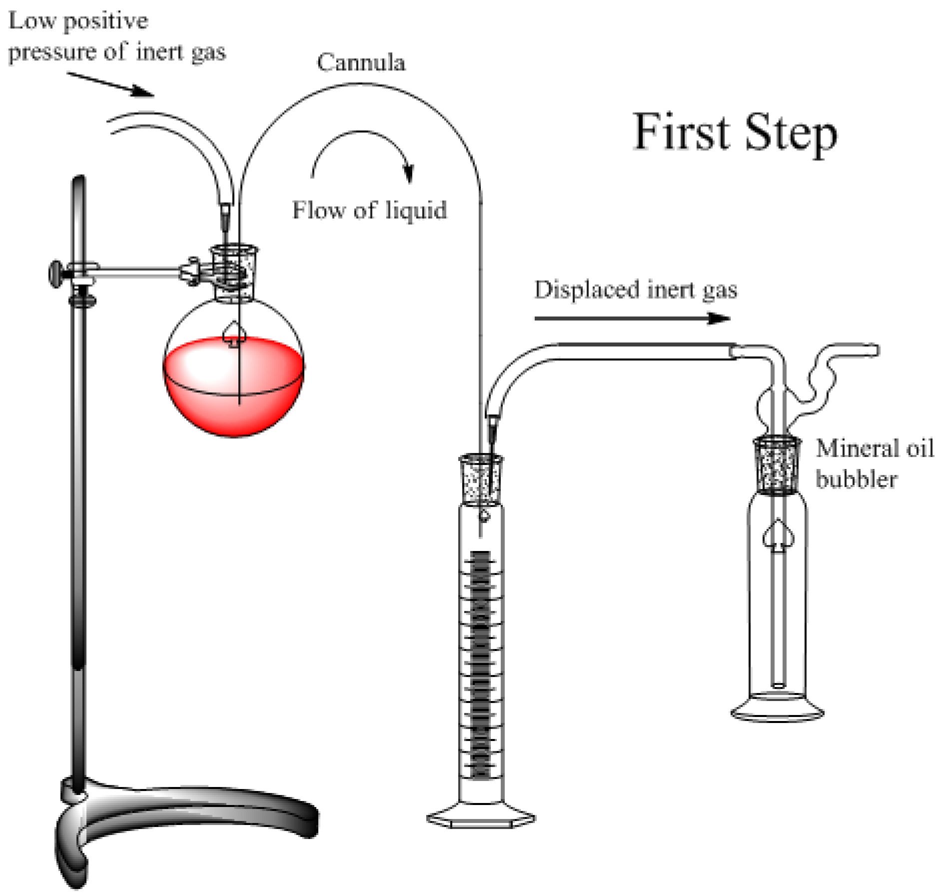


Alternative Cannula Technique for a Non-Graduated Apparatus
If the reaction apparatus is not graduated, this technique may be used to transfer a known volume. Everything for this alternative technique is the same as described in the previous technique except for the following:

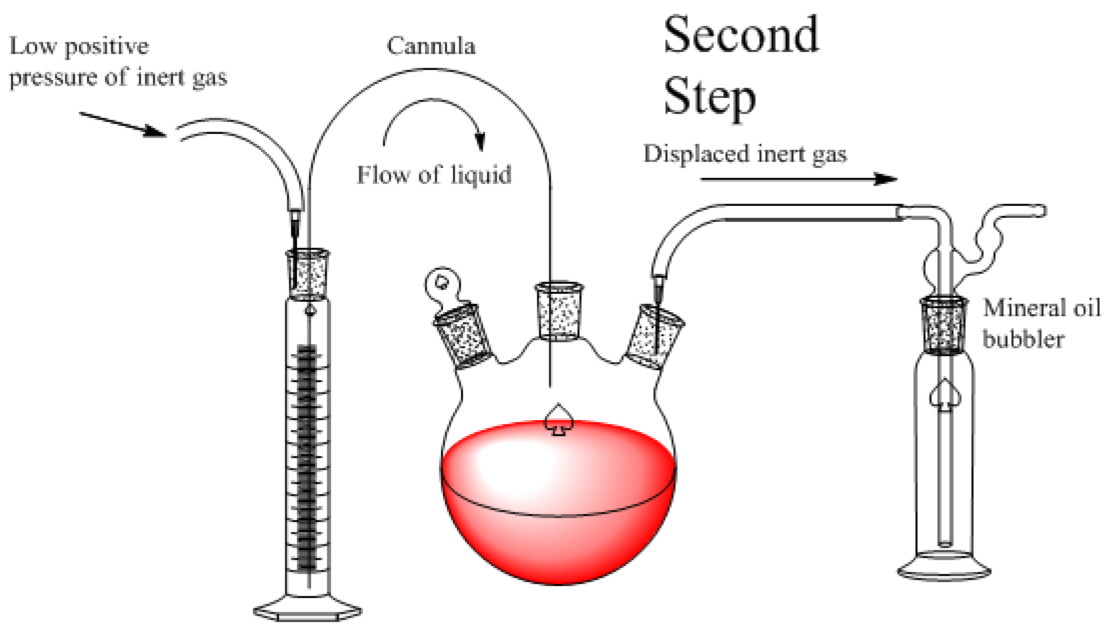
1. Clamp a flame or oven-dried graduated cylinder (at least 10 mL larger than the desired volume of the reactive liquid) to a solid and immovable support. Purge the graduated cylinder with an inert gas by placing a septum over the mouth of the graduated cylinder and inserting an inert gas line needle into the septum as well as a needle connected to a mineral oil bubbler.
2. Once the graduated cylinder is cool, remove the inert gas line needle. Then remove the screw cap from the securely clamped reagent bottle and insert an inert gas line needle into the septum to place reagent contents under low positive pressure.
3. Insert the cannula/double tipped needle into the reagent bottle through the septum so that the tip is in the headspace of the reagent bottle. Do not insert the tip into the reagent

itself.

4. Allow inert gas to purge the cannula/double-tipped needle for 5-10 seconds; then insert the other tip of the cannula into the septum of the graduated cylinder.



5. Insert the cannula tip from the headspace of the reagent bottle into the liquid itself. An immediate transfer of liquid should start to take place into the graduated cylinder.
6. Once the desired amount of liquid is transferred, pull the needle tip out of the liquid and into the headspace of the reagent bottle.
7. Remove the needle from the reagent bottle, and then immediately insert the same needle tip through the septum into the headspace of the reaction apparatus.
8. Cap the reagent bottle and store it appropriately.



9. Transfer the mineral oil bubbler from the graduated cylinder to the reaction apparatus and insert an inert gas line needle into the septum on the graduated cylinder.
10. Transfer the reagent from the graduated cylinder to the reaction apparatus in a manner identical to that used to transfer the reagent directly from the reagent bottle to the reaction apparatus. Then remove the cannula in the same manner as was done for the direct transfer.
11. Remove the septum from the graduated cylinder. If the reagent is exceptionally reactive, allow the cylinder to sit in the back of the chemical fume hood for a day before cleaning as normal. Otherwise, you may rinse the graduated cylinder with a compatible solvent into a beaker and then quench both the rinse and the residue in the graduated cylinder with an isopropyl alcohol/water/aqueous HCl sequence and dispose of the quenched material as normal.

Solids

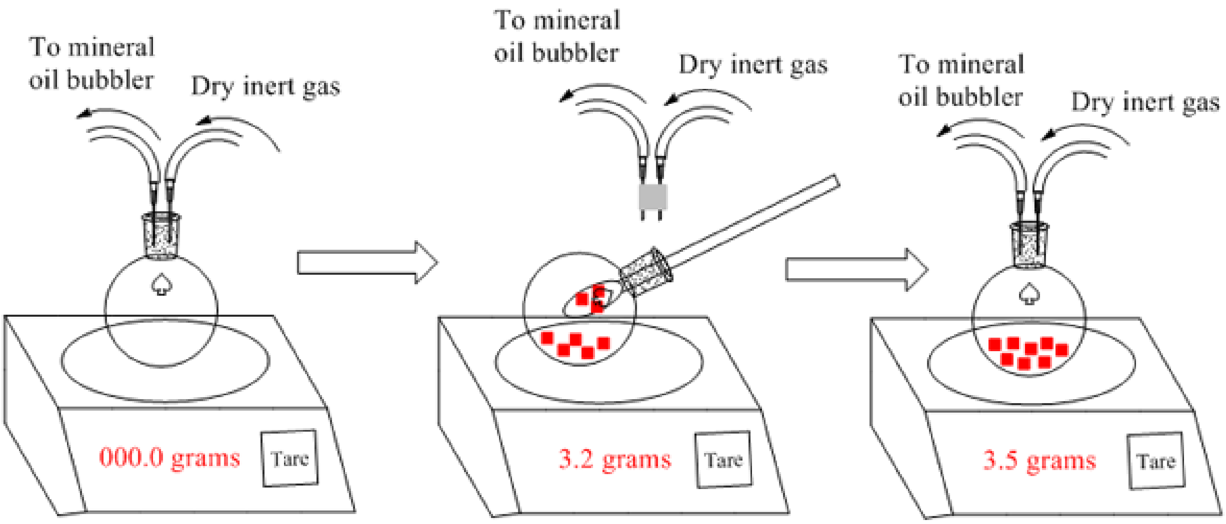
Warning: For highly air/water reactive solids use a glove box, the chemical dissolved in a liquid, or oil-coated dispersion form. Consult with DRS or an experienced researcher to determine the preferred technique to handle a specific chemical.

Septum Technique

1. Put the vessel (flame or oven dried) into which you intend to place the air/water reactive solid on a stable surface.
2. Place the vessel under an atmosphere of anhydrous and inert gas; use a septum inserted into a container/flask and two gas lines for inlet and outlet as shown below. Purge the vessel for a few minutes to remove all oxygen and moist air.
3. Open the vessel and quickly insert the spatula into the vessel, and add the air/water

reactive solid. Immediately close the vessel and place it under an inert environment. Again, purge the vessel of inert gas for a few minutes. Be sure to keep the original container of air/water reactive solid under an inert atmosphere by using this septum technique on it as well.

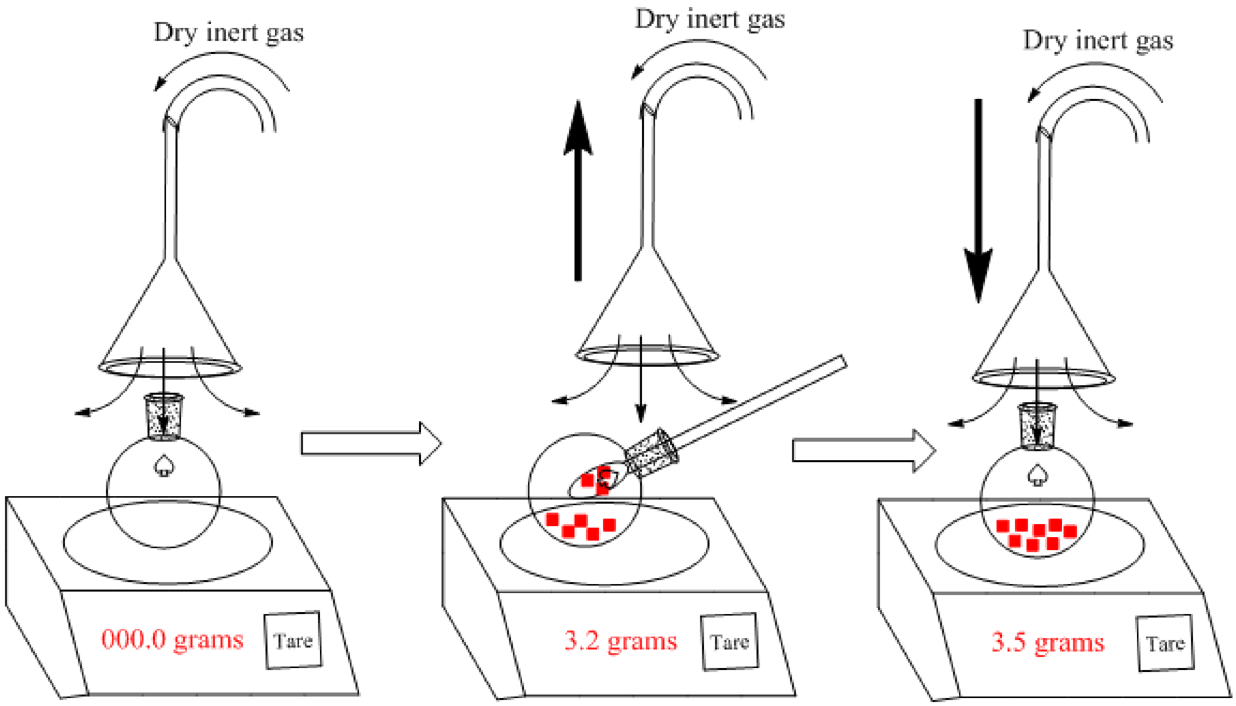
4. The air/water reactive solid is now ready for use.
5. Be sure to purge the original container of air/water reactive solid using anhydrous and inert gas. Then close and parafilm-seal the lid. Return it to storage.



Inverted Funnel Technique

1. Put the vessel (flame or oven dried) into which you intend to place air/water reactive solid on a stable surface.
2. Use a funnel with a mouth that covers the opening of the vessel into which the air/water reactive solid is to be weighed. Connect an inert gas line to it as shown in the illustration below.
3. Start the inert gas flowing, and adjust the flow to a moderately high rate. Be sure the inert gas is anhydrous and that the flow rate will create a blanket of inert gas over the air/water reactive solid as it is being manipulated.
4. Once the inverted funnel with inert gas is ready, place it over the vessel into which you intend to transfer the air/water reactive solid to purge the vessel of oxygen or moist air. Allow the vessel to be purged for a few minutes.
5. Once purged, very quickly lift up the inverted funnel and use a spatula or other transferring tool to transfer a portion of the air/water reactive solid into the vessel. Lower the inverted funnel immediately so that the air/water reactive solid remains under a blanket of inert gas. Be sure to also keep the original container of air/water reactive solid under an inert atmosphere by using the septum technique illustrated earlier!
6. Once the desired amount of air/water reactive solid is transferred, remove the inverted funnel and cover the vessel with a watch glass, lid, or parafilm. Keep the air/water reactive solid under an atmosphere of inert and anhydrous gas.
7. Be sure to purge the original container of air/water reactive solid with anhydrous and inert

gas. Then close and parafilm-seal the lid. Return it to storage.



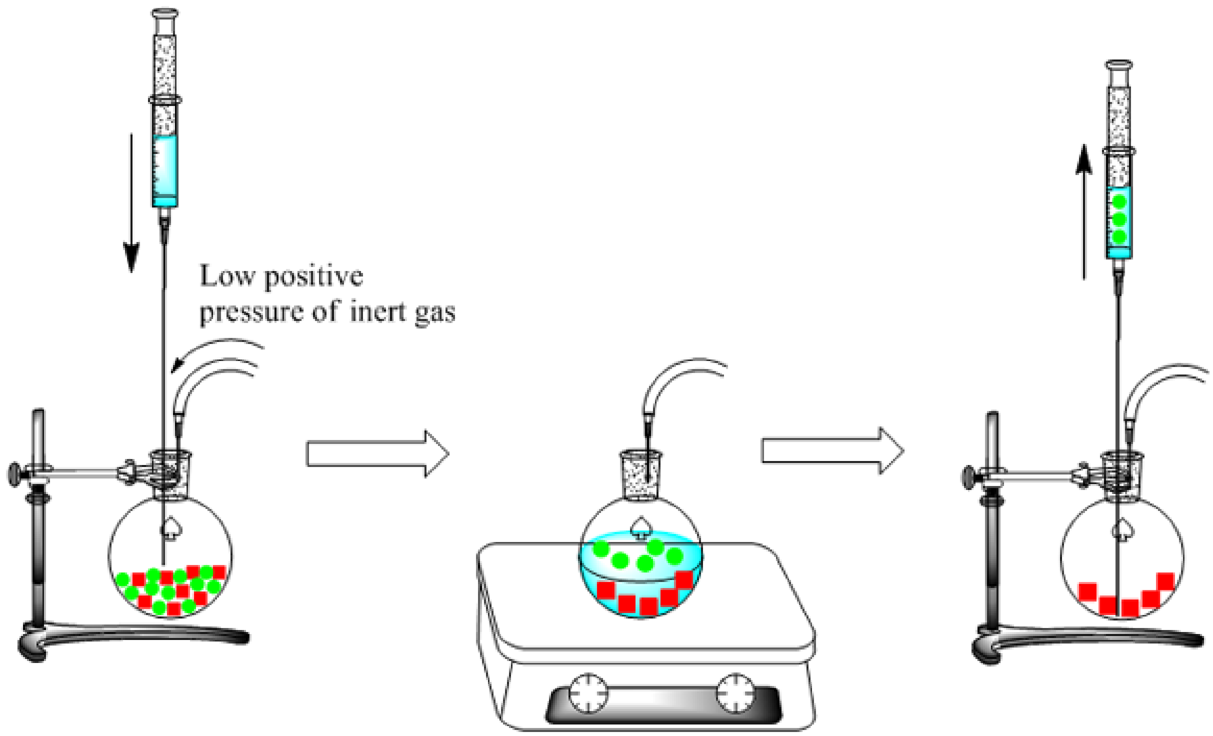
The inverted funnel technique can also be used for manipulating air/water reactive materials in odd-shaped and/or irregular pieces of equipment (e.g., pressure tubes, Büchner funnels, beakers, reaction kettles).

Oil-Coated Solid Technique

1. Put the vessel (flame or oven dried) into which you intend to place air/water reactive solid on a stable surface and insert a magnetic stir bar.
2. Place the vessel under an atmosphere of anhydrous and inert gas. Let it purge for a few minutes to remove all oxygen and moist air.
3. Open the vessel and quickly add the oil-coated air/water reactive solid. Immediately after adding the material, place the vessel under an inert environment. Again, purge it of inert gas for a few minutes. Be sure to also keep the original container of air/water reactive solid under an inert atmosphere by using the septa technique illustrated earlier!
4. While maintaining an inert atmosphere, inject solvent that will solubilize the oil with which the air/water reactive solid is coated (hexane is usually a good choice). Let it stir for a few minutes, and then stop the stirring so the solid can settle.
5. Using a long needle and syringe and without disturbing the settled air/water reactive solid, withdraw all of the solvent. (Using a cannula to decant the liquid is also an option)
6. Remove the syringe and needle. Eject the syringe contents into a large beaker and let it sit in the chemical fume hood for a week to slowly react with the air. Quenching the liquid can also be accomplished to cause residual material to react.
7. The air/water reactive solid is now ready for use. NOTE: Washing the air/water reactive solid multiple times in this manner may be necessary to remove all traces of the coating

oil. Some reactions may not require the removal of dispersion oil.

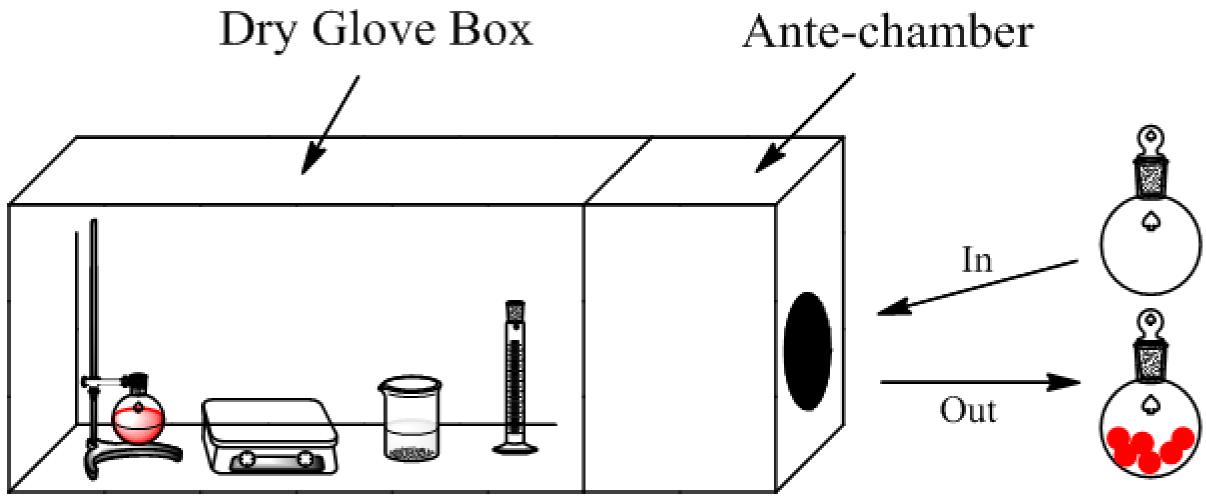
8. Be sure to purge the original container of air/water reactive solid with anhydrous and inert gas. Then close and parafilm-seal the lid. Return it to storage.



Glove Box Technique

1. Fill the main chamber of the glove box with inert gas according to the manufacturer's recommendations. This is usually done by performing three cycles of filling with inert gas and then evacuating.
2. Place the vessel (flame- or oven-dried) and lid into which you intend to place air/water reactive solid in the antechamber along with all the reagents and utensils you intend to use.
3. Close the antechamber door and evacuate the antechamber of atmospheric air as the manufacturer recommends. Fill the antechamber with inert gas.
4. Once the filling is complete, insert hands/arms into the glove port/sleeve assembly and open the interior antechamber door. Retrieve the vessel, reagents, and utensils you intend to use and place them in the main chamber.
5. Close and seal the interior antechamber door and start manipulating the air/water reactive solid as if it were a regular solid chemical.
6. Once finished, be certain the vessel containing the air/water reactive solid is closed and sealed. Clean up the main chamber accordingly.
7. Once you have confirmed the antechamber exterior door is still closed, open the interior antechamber door and place the vessel, reagents, and utensils in the antechamber.
8. Close the interior antechamber door and seal it.

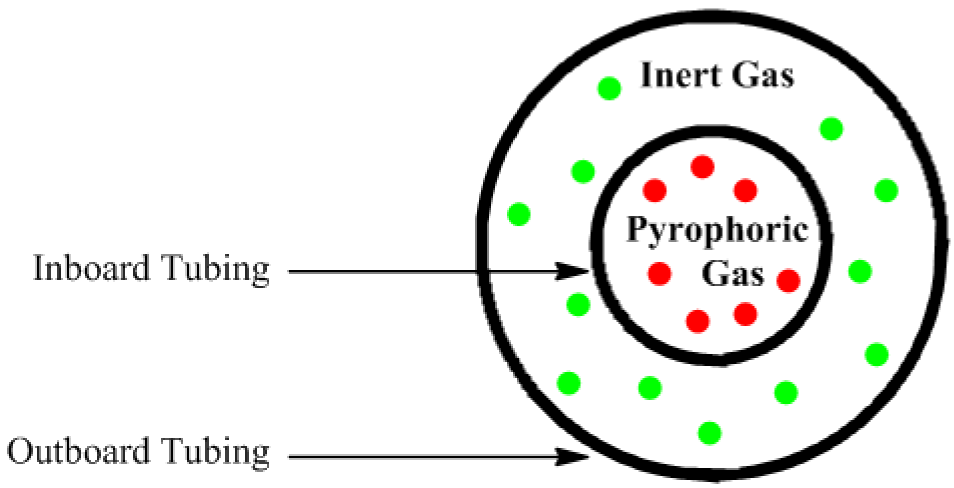
9. Open exterior antechamber door to retrieve your vessel of air/water reactive solid and other materials.
10. The vessel containing the air/water reactive solid is now ready for use.



Gases

A few extra notes of caution while working with air/water reactive gases:

- Pyrophoric compressed gas cylinders must be stored in a continuously ventilated gas cabinet with an operational fire suppression system inside the cabinet.
- Use tubing/plumbing that is compatible with the gas (e.g., stainless steel).
- Whenever possible, use outboard/inboard style tubing for air/water reactive gases.

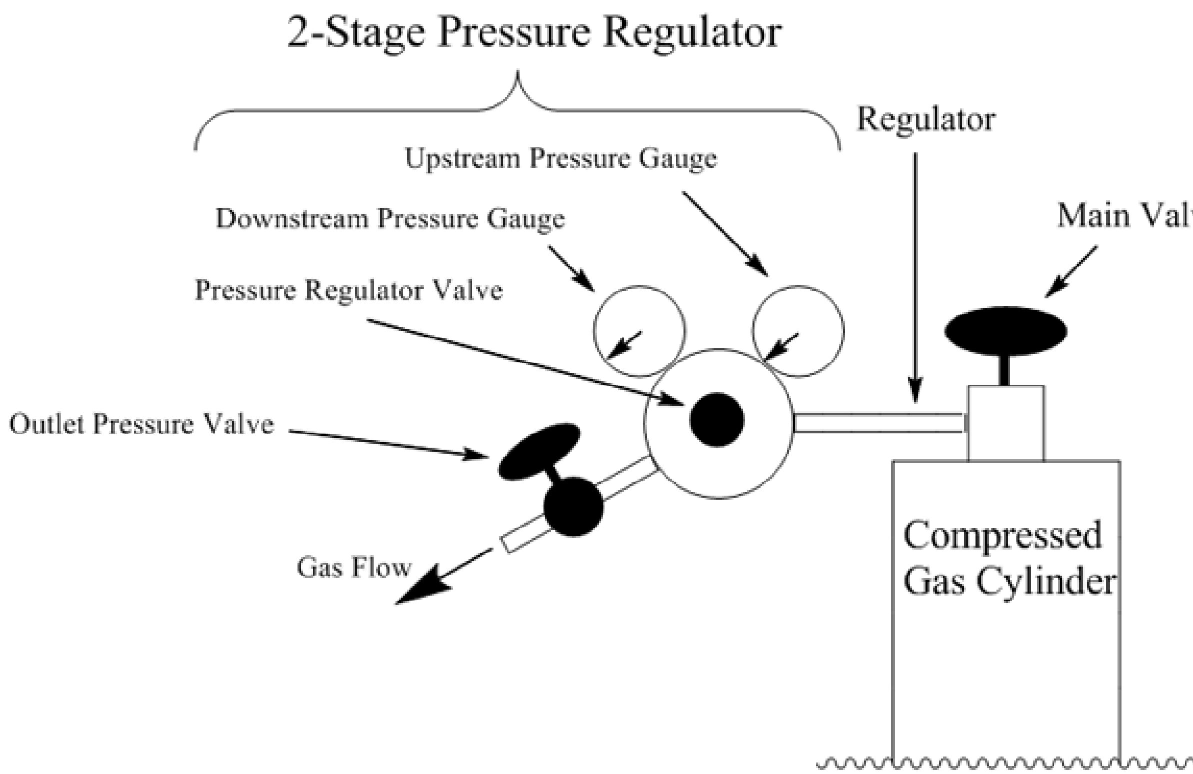


- A burnbox or scrubber device must be used when working with air/water reactive gases.
- A continuously ventilated engineering control (e.g., gas cabinet, chemical fume hood) must be used while working with air/water reactive gases.
- Leak detectors must be used while working with air/water reactive gases.

Regulator for flammable gas. Note notches on flats that indicate left-hand thread.

Air/water Reactive Gases

1. Secure the compressed gas cylinder against a bracket mounted to a solid structure inside of a gas cabinet or other engineering control.
2. Activate chemical leak detectors in the lab.
3. Unscrew and remove the compressed gas cylinder cap.
4. Install an appropriate CGA regulator using an adjustable wrench. NOTE: Do not use tools with teeth to install a regulator; this will destroy the flat surfaces on the regulator.
5. Install additional add-on pressure regulators/gauges as needed, and ensure all valves are closed (turned clockwise). Use Teflon tape as needed on screws, but do not use tape on regulator screws to be inserted into gas cylinder head.
6. Purge all in-board plumbing lines downstream of cylinder.
7. Ensure that the burnbox or scrubber is on and operational.
8. Open the compressed gas cylinder main valve by turning it counterclockwise until open.
9. Check leak integrity of connections using soapy water or “snoop.” If no leaks are detected, proceed; if leaks are detected, close all valves and proceed to correct leak connections with Teflon tape or by tightening connections.
10. If using pressure gauges, read the levels to ensure they are correct and nothing is amiss.
11. Continue opening valves (turning counterclockwise) further downstream, checking each connection for leaks until you reach the outlet pressure valve (last valve before tubing goes to the apparatus that is using the gas).
12. Attach tubing from the furthest downstream valve (outlet pressure valve or pressure regulator valve) to the apparatus or device to be used.
13. When the apparatus or device is ready, slowly open the outlet pressure valve (turning the knob counterclockwise) to a allow gas to flow. Use the outlet pressure valve or the valve on the pressure regulator (watching the downstream pressure gauge) to adjust gas flow rate to the desired level.



Clockwise Turning = Shutting Off/Reducing Gas Flow
Counter-Clockwise Turning = Opening/Increasing Gas Flow

Shut-down

1. Once gas is no longer needed, close the compressed gas cylinder main valve by turning it clockwise until it stops and gas flow stops (check gauges to confirm).
2. Purge all tubing lines and plumbing with inert gas.
3. Turn off the burnbox or disconnect the scrubber.
4. Disconnect tubing from the experimental device that is using air/water reactive gas, and remove tubing from the furthest downstream valve.
5. If the compressed gas cylinder is to be removed, close the main valve and open the other downstream valves to release internal pressure of inert gas in the plumbing.
6. Use an adjustable wrench to disconnect the pressure regulator from the compressed gas cylinder.
7. Place the cap back on the compressed gas cylinder and tighten it.
8. Unsecure the compressed gas cylinder from the bracket that is securing it and transport it back (via a cylinder cart) to storage.

Laboratory Specific Information

Prior Approval Required

☒ YES (describe): clearance with your PI and training with an experienced group member should be completed and documented.

Designated Area
☒ Other (describe): only inside fume hoods or gloveboxes.

Experimental Conditions of Use
Temperature Range: Ambient temperature
Pressure Range: Ambient pressure
Scale Range: 1 mg to 1 g scale
Other Relevant Details: Please follow chemical directions on lab storage and SOPs for proper waste disposal.

Standard Operating Procedure

Lithium- and sodium-ion cell disassembly

Overview

Lithium- and sodium-ion battery cells: defined as electrochemical devices comprising two solid-state electrodes: the cathode and anode, between which is an electrically insulating electrolyte (either a solid or a liquid). The anode and/or cathode may include lithium metal, sodium metal, transition metal fluorides, oxides, their solid solutions, polyanionic compounds, as well as many other possible chemistries. Here, the transition metals typically include manganese, iron, nickel, cobalt, vanadium and titanium. Electrolytes comprise either a glass fibre or polypropylene separator soaked in organic liquids (dimethyl carbonate, ethylene carbonate, propylene carbonate, fluoroethylene carbonate, diethyl carbonate and their mixtures) containing dissolved lithium hexafluorophosphate or sodium hexafluorophosphate salts; or solid transition metal oxides, sulphides and chlorides. These components are contained within a Swagelok-type stainless steel and/or polytetrafluorethylene (PTFE) body, a CR2032 coin cell, or a home-built electrochemical cell. The following process is described for disassembly of a Swagelok cell but the process is transferable to other cell types. Note that CR2032 coin cells are typically not disassembled following testing and rather taken by EH&S taped in clear plastic tape to prevent shorting. However, in cases where disassembly is required (e.g. for *ex situ* testing) the Li/Na metal should be disposed of using this method.

Process: disassembly involves exposing the contents of the cell (the cathode, anode and electrolyte) to air or a controlled argon atmosphere in a glovebox.

1. The stainless steel/PTFE body components are removed to leave the contents exposed either to ambient atmosphere inside a fume hood, or to the argon atmosphere in the glovebox (if components are desired for *ex situ* analysis). The body components should be stored in either a glass beaker, polypropylene bottle, or stainless steel cup, brought out of the glovebox (if relevant) and ethanol gradually added until the components are fully immersed.
2. If using the cathode, anode or electrolyte and separator for future experiments, retain in a glass or plastic vial for subsequent use. Else follow step 3 onwards.
3. Remove anode, cathode and separator/electrolyte from the glovebox, if appropriate. Obtain two beakers (sizes such that one beaker fits completely in the other) and a petri dish. Place the smaller beaker in the larger one.
4. Fill the smaller beaker to approximately one quarter of capacity with ethanol, then place anode, cathode and separator/electrolyte in the beaker. If lithium or sodium metal is present, leave soaking in the ethanol until effervescence ceases. Otherwise leave components soaking for at least five minutes. Ensure that no sodium/lithium contamination has occurred on any materials you may be working with (e.g. kim wipes, tweezers, etc.) If so, dispose of disposable components (e.g. kim wipes) in the lab's flame proof can (in the waste pick up area) and for non-disposables follow an analogous quenching procedure.
5. Slowly add water to the ethanol solution until the smaller beaker is approximately three-quarters full (i.e., add twice the volume of water to ethanol). Wait for at least two minutes.
6. Repeat steps 4 and 5 for the cell body components. If necessary, place the smaller beaker in an ultrasonic bath.

7. Dry the cell body components with paper towel and, if desired, place in drying oven.

Fire is the predominant risk with lithium and sodium metal containing cells, and can occur if water is added directly to the metals or if an ignition source is introduced. Risks can be mitigated by secondary containment (larger beaker), covering the secondary container with the petri dish to minimize oxygen exposure and slow addition of the ethanol and water to the components. All researchers handling these materials must receive thorough hands-on training and receive approval from the PI.

Special Handling and Storage Concerns

Personal Protective Equipment

- Flame resistant lab coat
- Safety goggles
- Nitrile gloves

Special Storage Requirements

Na or Li metal should be carefully handled across its entire life cycle in our labs. Prior to use, Na/Li should be handled solely in the glovebox. Na metal in particular should be stored under mineral oil in the glovebox. Following cell assembly, Na/Li metal waste generated within the glovebox during the cell making process should be retained in a plastic bottle (under mineral oil for Na) until ready for disposal. At that point, the bottle cap should be tightly closed and bottle sealed with parafilm. Coordinate with EH&S to minimize time (< 1 day) between removal of sealed waste bottles from glovebox and pick up. Following cell disassembly, this protocol should be followed for Na/Li neutralization to remove the hazards presented by these pyrophoric metals.

Engineering Controls

Glovebox: as much as possible, handle all components inside a glovebox

Fume Hood: when not handling inside a glovebox, handle all components in a fume hood. The fume hood should contain a bucket of sand, and the lab should be equipped with a class D metal fire extinguisher. In the MRL Battery Lab (MRL 1050), Fume hood #2 is the only designated fume hood for this use.

Testing: cells can be removed from the glovebox for testing with the Na/Li metal neutralized shortly after testing has been completed.

Special Handling Considerations

Quenching: Do not return anode, cathode or electrolytes to their original container. Unused pyrophoric materials should be disposed of as indicated in "Special Storage Requirements" above. To reiterate, following cell assembly, Na/Li metal waste generated within the glovebox during the cell making process should be retained in a plastic bottle (under mineral oil for Na) until ready for disposal. At that point, the bottle cap should be tightly closed and bottle sealed with parafilm. Coordinate with EH&S to minimize time (< 1 day) between removal of sealed waste bottles from glovebox and pick up. Quenching of pyrophorics is meant for used Na/Li metal from disassembled cells.

Never work alone when quenching these materials.

Decontamination

Do not use water. Consult material SDS for detailed hazard information.

Risk Management

Presence of Class D metal fire extinguisher in the lab, and all fume hoods contain a bucket of sand. In the MRL Battery Lab (MRL 1050), Fume hood #2 is the only designated fume hood for this use.

Waste Management

Dispose of the water/ethanol mixtures into aqueous basic waste. Any remaining solid materials should be appropriately disposed of in solid waste: label the container clearly, and contact EH&S for a prompt waste pickup. In Battery Lab, waste is picked up every Tuesday so aim to complete your work shortly before that day. If there is suspicion of pyrophoric material contamination on any waste product, dispose of in the lab's flame proof can-located in the waste pick up area.

First Aid and Emergencies

Spill and fire.

Within a Glove Box: quench the spilled material slowly with ethanol, absorb with a non-combustible absorbent, and dispose of as hazardous waste.

Outside of a Glove Box: Pyrophoric material will likely catch fire under these conditions. A Class D fire extinguisher may be used to extinguish a small fire. A Class ABC extinguisher should be used if a significant amount of organic solvent as caught fire as well. If you do not feel comfortable using a fire extinguisher, call 911. If the material has not caught on fire, notify others in the area of the spill. Evacuate the location. Call 911. Remain on-site, at a safe distance, to provide information to first responders.

Personnel Exposure

Consult material SDS for detailed hazard information.

Laboratory Specific Information

Prior Approval Required

> ☒ **YES (describe): clearance with your PI and training with a experienced user. Documentation must be done by way of a signed TNA indicating the training date on pyrophoric disposal. A hard copy should be kept in Battery Lab.**

Designated Area

> ☒ **Other (describe): only inside fume hoods or gloveboxes. In Battery lab, Fume hood #2 is the only designated fume hood for this use.**

Experimental Conditions of Use

> **Temperature Range: Ambient temperature**
> **Pressure Range: Ambient pressure**
> **Scale Range: 1 mg to 1 g scale**

Standard Operating Procedure

Battery material/Chemical waste disposal

Overview

Improper chemical waste disposal can lead to great risk to the labs and personals. When disposing chemicals, we must pay great caution. To be qualified for generating chemical waste in Battery Lab, one must fully understand the chemical properties of chemical waste, the compatibility between different chemicals, and the compatibility between chemicals and containers. All the group members who do lab work in Energy Lab ***MUST*** *get trained on this SOP.*

General Rules

⇒Do not dispose of chemicals via sink or trash cans.
⇒ Do not use fume hoods to intentionally evaporate chemicals.
⇒ Do not store waste outside the dedicated area.
⇒ Clearly label all the components in the chemical waste

Step by Step SOP

1. *Planning-Before starting any experiments, always think about whether chemicals waste will be produced. If the answer is yes, then your first step is to make plans for the chemical waste disposal.*
    a. *Identify the chemical waste and its physical and chemical properties and figure out which category that the waste belongs.*

Physical property*: Solid or Liquid*
Chemical properties:
- Halogenated organics
- Strong oxidizers
–Non-halogenated organics
- Peroxide-forming chemicals
- Acids of pH <2
- Alkaline solutions of pH > 12.
- Chemical carcinogens
- Alkali metals
- Unstable chemicals
- Heavy metal solutions and salts
- Other toxic materials

    b. Make the waste container available.

Different categories of chemicals need to be stored in different containers. Check if the lab already has the waste container for the certain category that your waste belongs to. If not, you need to get a new container that is compatible to your waste. If you cannot identify which category your waste belongs to, please use a new container.

When choosing containers for the chemical waste, **Compatibility** must be considered. Some general rules are:

i. **Do not use aluminum "ether" containers to store waste**. Do not store corrosives in metal steel containers.
ii. Do not store corrosive waste in the metal container (better to not use metal container for any waste)
iii. Fluorides (either solids or liquids) waste should be stored in plastic containers not glass containers.
iv. Non-halogenated organics better to be stored in Glass container. **Waste containing Acetone should be always stored in glass container.**

b. Create labels.
provide all the necessary information and fill the label. **Note: please use the full name of the chemicals rather than chemical formula or abbreviations.** Fill the State, chemical hazard classification(s), Date of accumulations. Label must be created before chemical waste is generated.
When the new chemical waste is compatible to already generated waste. You might use the same container and add the new chemical waste onto the existing label.

Once you finish the planning. You are good to start your chemical experiment.

2. Store the Chemical Waste

Once you transferred the chemical waste into containers. The containers need to be stored in a proper manner.
In MRL 1050, the bin for waste pick up is along the south wall by the chemical refrigerator on the floor. Please transfer material to be picked up into this bin on the day of pick up. Pick up is usually on Tuesdays. Otherwise, please leave material in an area with ventilations (inside fume hoods or the closet below the fume hoods). **Never leave the waste containers or bins outside the dedicated area for overnight!**

3. Arrange a pick up

Stop accumulating once Liquid waste is filled over 80% volume of the container. And ask for pick up.
Email Bruce Carter (bruce.carter@ehs.ucsb.edu) for waste pickup.

4. WHAT TO DO IN THE EVENT OF A SPILL

a. For emergencies involving chemical spills, call 9-911.
b. For technical assistance in non-emergencies involving chemical spills, call EH&S at ext. 3194 (24-hr. Hazardous Material Technical Assistance Line).
c. For chemical spills in which technical assistance is not needed, EH&S must be notified immediately for compliance purpose and to arrange pickup of the spilled materials.

Email Bruce Carter (bruce.carter@ehs.ucsb.edu) if you are unsure on anything.

Standard Operating Procedure

Carcinogens, Reproductive Toxins and Acute Toxins

(Cal/OSHA Particularly Hazardous Substances)

Overview

Three classes of hazardous chemicals are defined by Cal/OSHA as 'Particularly Hazardous Substances' (PHS):

- *Carcinogens* are materials that have the potential to cause cancer.
- *Reproductive Toxins* are materials that affect reproductive capabilities, including *mutagenesis* (causing chromosomal damage), *teratogenesis* (effects on the fetus), and adverse effects on sexual function and fertility.
- *Acute toxins* are substances that may be fatal as a result of a single exposure ($LD_{50} \leq$ 50 mg/kg (oral), 200 mg/kg (dermal), 500 ppm (inhaled).

If the carcinogen you are using is a Listed Carcinogen (8 CCR §5209), EH&S will contact you upon your ordering of that material to address safety requirements that go beyond this SOP.

Special Handling and Storage Concerns

Personal Protective Equipment

- Traditional lab coat. Flame resistant if material is flammable.
- Nitrile or Neoprene Gloves are adequate for possible incidental exposure in most cases. Consult a glove chart if the specific material in use is particularly hazardous, or if the risk of contact is high.
- ANSI Z87.1-compliant safety glasses. Safety goggles if a splash hazard is present.

Special Storage Requirements

Store Particularly Hazardous Substances away from other chemicals. Each container must include all applicable hazard warnings. It is recommended that the appropriate GHS pictogram also be on the container. The storage area must be within a PHS designated area, and all containers stored in secondary containment.

Engineering Controls

Fume Hood: All PHS *must* be handled in a fume hood. If this is not possible due to scale or equipment, contact EH&S to determine alternate ventilation approaches or respiratory protection needs.

Special Handling Considerations

Only use PHS in a designated area. This designated area may be the entire laboratory, or only a portion of it. Note that the information in this SOP describes the baseline requirements for PHS. You will need to generate or review a chemical-specific SOP if the material you are handling has:

- *Unique properties*: e.g. cyanide salts, where the risk of exposure varies greatly with pH.
- *Multiple hazards*: e.g. azide salts, which are highly toxic and potentially explosive.
- *Extreme hazards*: e.g. methyl mercury, which penetrates the skin and is lethal in tiny doses.

Decontamination

This SOP covers a wide range of materials. Consult the SDS for any possible special decontamination procedures.

Waste Management

Note that some PHS waste may be considered *Extremely Hazardous Waste* and should be handled as described in the UC Santa Barbara Chemical Hygiene Plan. This includes disposing of the emptied original container as hazardous waste through EH&S.

First Aid and Emergencies

Spill

Treat all spills of these materials as a major spill. Do not attempt to clean up the spill yourself. Notify others in the area of the spill, including your supervisor. Evacuate the area and call 911. Remain on-site at a safe distance to provide detailed response to first responders. Report any exposures to EH&S.

Fire

Standard measures apply.

Personnel Exposure

Skin or eye contact: Remove contaminated attire. Flush affected area with water for 15 minutes. If symptoms persist, get medical attention.

Inhalation: Move person to fresh air. Get medical attention immediately.

Ingestion: Rinse mouth with water. Get medical attention immediately.

Laboratory Specific Information

Prior Approval Required

- ☒ **NO**
- ☐ **YES (describe):**

Designated Area (required for Particularly Hazardous Substances)

- ☐ **Entire Laboratory Area**
- ☒ **Other (describe): MRL 1050**

Experimental Conditions of Use

Temperature Range: Room Temperature

Pressure Range: 1 atm

Scale Range: 1-4L

Other Relevant Details: Specific Chemicals used: Chloroform, Dichloromethane, Methanol

Standard Operating Procedure

Dichloromethane

(Methylene Chloride)

Overview

Dichloromethane is an anesthetic. Inhaling the vapor can cause light-headedness leading to unconsciousness and even death. Other symptoms of exposure include skin, eye and respiratory tract irritation. Strong evidence supports that dichloromethane is a human carcinogen upon chronic exposure. Its high volatility makes it imperative that it be handled in a fume hood or other vapor-capturing device. Unlike many organic solvents, dichloromethane is not flammable. Dichloromethane is considered a Particularly Hazardous Substance (PHS).

Special Handling and Storage Concerns

Personal Protective Equipment

- Standard Lab Coat.
- Butyl, Viton and polyvinyl alcohol gloves are recommended. Standard nitrile and neoprene lab gloves are NOT recommended.
- ANSI Z87.1-compliant safety glasses. Safety goggles if a large splash hazard is present.

Special Storage Requirements

Dichloromethane is a PHS. Each container must include all applicable hazard warnings. It is recommended that the appropriate GHS pictograms also be on the container. The storage area must be within a PHS designated area, and all containers stored in secondary containment.

Engineering Controls

Fume Hood: Dichloromethane *must* be handled in a fume hood. If this is not possible due to scale or equipment, contact EH&S to determine alternate ventilation/isolation approaches or respiratory protection needs.

Special Handling Considerations

Only use dichloromethane in a PHS in a designated area. This designated area may be the entire laboratory, or only a portion of it.

Decontamination

Standard decontamination procedures apply. Use great caution in avoiding exposure.

Waste Management

Standard waste disposal procedures apply.

First Aid and Emergencies

Spill

Treat all spills of benzene as major spills. Do not attempt to clean up the spill yourself. Notify others in the area of the spill, including your supervisor. Evacuate the area and call 911. Remain on-site at a safe distance to provide detailed response to first responders. Report any exposures to EH&S.

Fire

Dichloromethane is not itself flammable. Standard firefighting measures apply

Personnel Exposure
Skin or eye contact: Remove contaminated attire. Flush affected area with water for 15 minutes. If symptoms persist, get medical attention.
Inhalation: Move person to fresh air. Consult a physician is symptoms persist.
Ingestion: DO NOT induce vomiting. Rinse mouth with water. Consult a physician.

Laboratory Specific Information

Prior Approval Required

- ☒ **NO**
- ☐ **YES (describe):**

Designated Area

- ☐ **Entire Laboratory Area**
- ☒ **Other (describe): MRL 1050**

Experimental Conditions of Use

Temperature Range: Room Temperature

Pressure Range: 1 atm

Scale Range: >1L

Other Relevant Details:

Standard Operating Procedure

Peroxide-Forming Chemicals

Overview

Peroxide-Forming chemicals are flammable organic liquids which are capable of forming potentially explosive organic peroxides (R-O-O-R') upon exposure to air or other oxidizing impurities. Organic peroxides are among the most hazardous substances handled in the laboratory. They are sensitive to oxygen, heat, friction, light, strong oxidizers and reducing agents, and are far more shock sensitive than most primary explosives such as TNT. It is particularly dangerous to allow these materials to evaporate to dryness, such as during distillation. **ETHERS** are the peroxide-formers most common in the laboratory.

Special Handling and Storage Concerns

Personal Protective Equipment

- Flame Resistant Lab Coat.
- Nitrile or Chloroprene gloves are adequate for incidental exposure. Consult a glove chart if large splashes or immersion are possible.
- ANSI Z87.1-compliant safety glasses. Safety goggles if a large splash hazard is present.

Special Storage Requirements

Store in airtight containers, and in a flammable storage cabinet or refrigerator rated for flammable materials. Containers larger than 4 L are not recommended, due to the time-sensitivity of these materials. *Date containers upon receipt and opening*. As noted in the UC Santa Barbara Chemical Hygiene Plan:

- Class A peroxide formers must be discarded within 3 months of receipt or formation:
  (Divinyl ether, divinyl acetylene, isopropyl ether, sodium and potassium amide, potassium metal.)
- Class B peroxide formers must be discarded 6 months after opening, 12 months if they contain an inhibitor:
  (Diethyl ether, Furan, tetrahydrofuran, dioxane, etc.)
- Class C peroxide formers must be discarded after 5 days, 12 months if they contain an inhibitor:
  (Acrylic acid, ethyl acrylate, methyl methacrylate, styrene, vinyl acetate, vinyl chloride, vinyl pyridine)

Engineering Controls

Diethyl ether must be used in a fume hood at all times. Solvent mixtures for purification equipment that contain tetrahydrofuran or other higher boiling ethereal solvents must be prepared in the fume hood, but can be used in equipment outside the hood as long as the reservoir container is sealed.

Special Handling Considerations

Static Electricity Risk: Large containers of peroxide-forming chemicals are discouraged due to the time-sensitivity of these materials. If metal drums are used (≥20 L), they should always be grounded, and they should be bonded to the receiving container during transfer. Flammable storage cabinets are equipped with a grounding system that should be connected to a building ground. Transferring these materials between unbonded metal containers, or between plastic containers may lead to a fire hazard due to static electricity buildup.

Decontamination

This SOP covers a wide range of materials. Consult the SDS for any possible special decontamination procedures.

Laboratory Specific Information

Prior Approval Required

- ☒ **NO**
- ☐ **YES (describe):**

Designated Area

- ☐ **Entire Laboratory Area**
- ☒ **Other (describe): MRL 1043, MRL 1051, MRL 1050**

Experimental Conditions of Use

Temperature Range: Room Temperature

Pressure Range: 1 atm

Scale Range: 1-4L

Other Relevant Details: Specific Chemical Used: Tetrahydrofuran

Standard Operating Procedure

Flammables

Overview

A flammable solvent is defined by the National Fire Protection Agency (NFPA) as having a flashpoint below 100 °F (37.8 °C). The lower the flashpoint, the more easily the liquid can be ignited. Their presence in the laboratory in fairy large volumes greatly exacerbates the fire risk posed by these materials. The large volumes also increase the risk posed by any other hazards associated with the specific material (toxicity, carcinogenicity, etc.)

Special Handling and Storage Concerns

Personal Protective Equipment

- Flame Resistant Lab Coat.
- Nitrile or Neoprene Gloves are adequate for possible incidental exposure. Consult a glove chart if large splashes or immersion are possible.
- ANSI Z87.1-compliant safety glasses. Safety goggles if a large splash hazard is present.

Special Storage Requirements

Store in a flammable storage cabinet with self-closing hinges, or in a refrigerator rated for flammable storage. Any container larger than 1 gallon (4L) must be stored in a flammable storage cabinet at all times. *The maximum amount of flammable material allowed outside of these storage areas is 10 gallons*. Store flammable materials away from oxidizers and combustible materials. Flammable cabinets must be labeled clearly with the statement “Flammable – Keep Fire Away”.

Engineering Controls

If your protocol does not permit the handling of these materials in a **fume hood**, EH&S *must* be contacted to assess alternate ventilation options.

Special Handling Considerations

Static Electricity Risk: Large containers of flammable liquid (≥ 20L) should always be grounded, and should be bonded to the receiving container during transfer. Flammable storage cabinets are equipped with a grounding system that should be connected to a building ground. Transferring these materials between unbonded metal containers, or between plastic containers may lead to a fire hazard due to static electricity buildup.

Decontamination

This SOP covers a wide range of materials. Consult the SDS for any possible special decontamination procedures.

Waste Management

Segregate halogenated from non-halogenated organic solvent waste.

First Aid and Emergencies

Spill

A number of organic solvents are carcinogenic (e.g. benzene, methylene chloride, formaldehyde). Do not attempt to clean up a spill of these materials. Consult the SDS to confirm toxicity information, then call

EH&S for assistance.

Fire

Standard firefighting measures apply.

Personnel Exposure

Standard measures apply.

Laboratory Specific Information

Prior Approval Required

- ☒ **NO**
- ☐ **YES (describe):**

Designated Area

- ☐ **Entire Laboratory Area**
- ☒ **Other (describe): MRL 1043**

Experimental Conditions of Use

Temperature Range:

Pressure Range:

Scale Range:

Other Relevant Details:

Standard Operating Procedure

Corrosives

Overview

Corrosives are materials, acids and bases, that cause the destruction of exposed tissues and mucous membranes. Rapid damage can occur to eyes and skin, as well as to the respiratory tract (inhalation) and gastrointestinal tract (ingestion). Strong corrosive solutions have a pH <2.5 (strong acids) or >11 (strong bases) and cause damage via the reaction of hydroxide ions (OH^-) or hydronium ions (H_3O^+) with tissue. *This SOP <u>does not</u> cover oxidizing acids (e.g. Nitric acid, perchloric acid), or corrosives with other highly hazardous properties (e.g. hydrofluoric acid). If using, please see the SOP specific to these materials.*

Special Handling and Storage Concerns

Personal Protective Equipment

- Traditional white lab coat. Chemical-resistant apron when working with large volumes.
- Nitrile or neoprene gloves are adequate for possible incidental exposure. Consult a glove chart if large splashes are possible. *No latex gloves!*
- ANSI Z87.1-compliant safety glasses. Safety goggles or safety goggles plus face shield if a large splash hazard is present.

Special Storage Requirements

Acids and bases must be segregated in storage. Store in chemically-resistant secondary containers (e.g. polypropylene tubs). Store below eye level. Segregate from active metals such as sodium, potassium, magnesium, etc. Use a corrosives storage cabinet if available.

Engineering Controls

If your protocol does not permit the handling of these materials in a fume hood, assess the volatility of the material (e.g. hydrochloric acid) and contact EH&S if alternative ventilation options are necessary.
An eye wash/safety shower unit *must* be within a 10 second walk (about 35 feet) from where corrosives are being handled, with only a single intervening door, opening in the direction of travel.

Special Handling Considerations

When forming solutions/dilutions, to avoid serious splatter risk ***add the corrosive to water, and never the reverse***.
Acids can react with metals, releasing flammable hydrogen gas.

Waste Management

Segregate acids of pH $\leq$2, bases of pH $\geq$12.5, and oxidizing acids.

First Aid and Emergencies

Spill

It is best practice to keep acid and base neutralizers in the laboratory spill kit if corrosives are used (e.g. sodium bicarbonate, citric acid).

Personnel Exposure

Standard measures apply. Pay extra attention to flushing affected skin/eyes with water for a full 15 minutes using an eyewash/safety shower unit.

Laboratory Specific Information

Prior Approval Required

- ☒ **NO**
- ☐ **YES (describe):**

Designated Area

- ☐ **Entire Laboratory Area**
- ☒ **Other (describe): MRL 1043, MRL 1051**

Experimental Conditions of Use

Temperature Range: Room Temperature

Pressure Range: 1 atm

Scale Range: >1L

Other Relevant Details: Chemical specifically used: 0.1% solution of formic acid

Standard Operating Procedure

Compressed Gases

Overview

Chemicals in this category present hazards based on one or more of these characteristics:

- Pressurization
- Oxidizing ability
- Flammability*
- High Toxicity*

Many compressed gases are also considered to be simple asphyxiants due to their ability to displace oxygen in the event of a rapid release.
***Highly toxic and pyrophoric gases are some of the most dangerous materials found in the laboratory. A gas-specific Standard Operating Procedure must be developed for these materials in conjunction with the campus Chemical Hygiene Officer**.

Special Handling and Storage Concerns

Personal Protective Equipment

- Traditional white lab coat.
- Nitrile or neoprene gloves are adequate for possible incidental exposure.
- ANSI Z87.1-compliant safety glasses.

Special Storage Requirements
Proper mounting of gas cylinders is imperative. Follow all mounting requirements as described in the UC Santa Barbara Chemical Hygiene Plan Chapter 3, section 'Chemical Inventory, Storage and Transport'.
Corrosive gases: Store lecture bottles 6 months or less, cylinders 2 years or less.
Oxidizing gases: Store with 20 feet separation from, or non-combustible partition between, *flammable gases*.
Engineering Controls
Oxygen sensors: May be necessary in rooms where large quantities of compressed gases are stored or handled, or in areas with limited ventilation (closets, cold rooms).
Carbon monoxide sensors: Required for carbon monoxide use if the cylinder or any plumbing are outside of a fume hood or gas cabinet.
Special Handling Considerations
Be cautious when handling compressed gases in poorly ventilated areas such as cold rooms.
Inspect cylinders and valves for corrosion or other damage on a regular basis.
Transport:

- Disconnect regulators and other apparatus prior to transport.
- Always replace the valve safety cap before transporting cylinders.
- Cylinders must always be transported using a hand truck or cart designed for that purpose.
- Transport cylinders upright.

When transporting compressed gases on elevators, use service or freight elevators when available. In addition, when transporting compressed gases by elevator:

- Post a sign reading "DO NOT ENTER – GAS TRANSPORT" to exclude passengers. Send the elevator to the desired floor, but do not enter the elevator yourself.
- When possible, have someone send the elevator up while another person waits on the receiving floor to take the cylinder out of the elevator. If this is not possible, another plan should be devised to ensure that the cylinder is taken out of the elevator once it reaches the desired floor.

First Aid and Emergencies

Uncontrolled Release

In the event of an uncontrolled release, assume that an oxygen deficient atmosphere is present. Notify others in the area and evacuate the room until adequate oxygen levels can be confirmed.

Personnel Exposure

Move person to fresh air only if safe to do so. *If you suspect that a person has lost consciousness due to oxygen deprivation, call 911 and do not enter the room*. If symptoms persist, seek medical attention.

Laboratory Specific Information

Prior Approval Required

- ☒ **NO**
- ☐ **YES (describe):**

Designated Area

- ☒ **Entire Laboratory Area**
- ☐ **Other (describe):**

Experimental Conditions of Use

Temperature Range: Room Temperature

Pressure Range:

Scale Range:

Other Relevant Details:

Standard Operating Procedure

Vacuum Systems

Overview

Systems under vacuum and their associated equipment have a variety of hazards associated with them, including:

- Implosion and the associated flying debris, splattering chemicals and possibly fire.
- Condensation of liquid oxygen into a cold trap using liquid nitrogen as coolant. Liquid oxygen is an explosion hazard when warmed in a closed system, and when it comes in contact with organic material.
- Pinching extremities or catching clothing in the vacuum pump belt system.
- Exposure to hazardous material due to improper venting of pump exhaust.

These systems are typically quite complicated and require extensive hands-on training prior to use.
Related SOP:

- Cryogens

Special Handling and Storage Concerns

Personal Protective Equipment

ANSI Z87.1-compliant safety glasses or goggles. A face shield is recommended if the system is made of glass or other breakable material, and is not behind a fume hood sash or blast shield.

Engineering Controls

Special Handling Considerations

General Concerns

- Understand the type of vacuum pump being used, and ensure that it is appropriate for the application (e.g. evaporation of solvents vs. high vacuum).
- Prepare for power outages. Some valves close upon loss of power, some open. Understand the effects that a series of valve openings and closings will have upon the system's integrity.
- Always replace the pump belt guard to prevent catching fingers or clothing in the mechanism.
- Glass vessels that are evacuated should be round-bottomed and/or thick-walled and designed for low-pressure work. They should be regularly checked for star cracks and scratches.

Traps and Venting

- Mechanical vacuum pumps should be protected by cold traps – generally liquid nitrogen based. Cold traps are dangerous due to their ability to condense liquid oxygen. Therefore, operation of low these traps must be thoroughly understood. Both the cooling and warming phases deserve undivided attention, and the system tested for leaks regularly.
- If hazardous materials are used with the vacuum system they should be located in, and **vented** to, a fume hood.
- Dewar flasks are insulated by being under high vacuum and are therefore subject to implosion. They should be wrapped in tape or plastic sheathing and generally come that way.

Turning ON a High Vacuum System:

- Make sure all valves are closed.
- Turn on vacuum pump.
- Place Dewar around trap flask
- Submerge trap flask in liquid nitrogen. <u>Make sure system is under vacuum before cooling trap to avoid condensation of liquid oxygen.</u>

Turning OFF a High Vacuum System

- Remove all samples and experiments from vacuum line.
- Remove trap flask from Dewar. Allow to warm to room temperature
- Open vacuum system to atmosphere. <u>Do not do this while trap is cold to avoid condensation of liquid oxygen.</u>
- Turn off pump.

Chemical Hazards

- Mechanical pump oil can become contaminated with hazardous materials. During maintenance, proper protective equipment must be employed. A ventilated area should be used for changing pump oil, as harmful vapors may be released. Clean or contaminated pump oil must be disposed of as hazardous waste via EH&S.
- Mechanical pump exhaust may require suitable scrubbing for volatile highly toxic materials. This may involve a relatively simple filter or liquid bubbler.

Decontamination

Please see Large Laboratory Equipment Decontamination SOP for guidance on how to decontaminate vacuum pumps for repair or disposal

Waste Management

First Aid and Emergencies

Spill

Fire

Personnel Exposure

Laboratory Specific Information

Prior Approval Required

☐ **NO**

☐ **YES (describe):**

Designated Area

☐ **Entire Laboratory Area**

☐ **Other (describe):**

Experimental Conditions of Use

Temperature Range:

Pressure Range:

Scale Range:

Other Relevant Details:

Standard Operating Procedure

High-Pressure Reaction Vessels

Overview

Failure/explosion of a high-pressure reaction vessel creates a significant and immediate threat from flying debris and reaction constituents. Failure can be caused by a variety of factors including:

- Overloading
- Exceeding temperature/pressure rating
- Reagents incompatible with vessel material

Special Handling and Storage Concerns

Personal Protective Equipment

- Lab Coat, flame resistant if using flammable materials. Also, a chemical resistant apron if using corrosive materials.
- Nitrile or Neoprene Gloves are adequate for possible incidental chemical exposure. Consult a glove chart if extremely toxic or corrosive material is being handled.
- ANSI Z87.1-compliant safety goggles. Goggles and a face shield when performing manipulations while to vessel is at elevated pressure.

Special Storage Requirements

Keep a log of usage for each vessel. Information on the log should include temperature, pressure, reagents/solvents used, and any inspections and tests it has undergone.

Engineering Controls

Fume Hood: If your protocol does not permit the handling of these materials in a fume hood, EH&S *must* be contacted to assess alternate ventilation options.
Blast Shield: A portable blast shield should be used for small vessels being operated in a fume hood. Custom barricades/shields should be designed for vessels that are not operated inside a fume hood. These barricades/shields should protect in all directions that debris or reaction mixtures could fly in the event of a vessel failure.

Special Handling Considerations

Perform high-pressure operations only in special chambers equipped for this purpose. Commercially available high pressure reactor vessels are designed and manufactured to ensure safe operation when used within the temperature and pressure limits for which they are rated. **Any documentation and manuals that pertain to the reactor vessel in use must be thoroughly read, understood and consulted regularly.** However, in the end it is the user's responsibility to make sure that the selected vessel is compatible with the reagents and conditions to which it will be exposed during the experiment.

To this end, the user must:

- Select a vessel which has the capacity, pressure rating, corrosion resistance and design features that are suitable for its intended use.
- Operate the vessel within a suitable barricade/shield, if required.
- Establish training procedures to ensure that any person handling the equipment knows how to use it properly.

- Maintain the equipment in good condition, and test periodically per the vendor's instructions to ensure that the vessel is remains structurally sound.
- Complete a hazard assessment before initiating the experiment, including:
  - Assessment of any intermediates, side-products and products that may form and their behavior within the vessel, including their corrosive nature and their tendency to violently decompose at elevated temperature and pressure.
  - Determination of maximum temperature and pressure limits expected, taking into account the energetics of the reaction being conducted and any pathways that might cause the reaction to run out of control.
- Maintain adequate ventilation. This can be achieved by installing the reactor within a fume hood, attaching tubing to the rupture disk that extends to an appropriate exhaust such as the interior of a fume hood, or by ensuring that the lab area as a whole has adequate ventilation and that the reactor is installed near an exhaust fan (in the case of larger reactors).
- Run preliminary experiments using small quantities of reactants when starting work with new or unfamiliar materials.
- Use appropriate PPE, including safety glasses, chemical resistant gloves, a lab coat, and also a face shield for operations that present particular hazards.
- Keep a log of usage for each vessel. Information on the log should include temperature, pressure, reagents/solvents used, and any inspections and tests it has undergone.

Particular Hazards of Note

Potentially Explosive Material

There are a number of functional group categories whose presence within a structure is a common indication of explosive potential. Use of reagents containing these functional groups in a high-pressure reactor is contraindicated. These include but are not limited to: peroxides, perchlorates, azides, metal acetylides, etc.

Loading Limits

Overloading of a pressure vessel is a significant hazard. Dangerous pressures can develop suddenly and unexpectedly when a liquid is heated in a closed vessel if adequate head-space is not available to accommodate the expansion of the liquid. *This is particularly true of water and aqueous solutions, whose volume may increase up to a factor of three when heated to 374 °C.*

A vessel must **never** be filled to more than three-fourths of its available free space. Frequently, the maximum fill level must be reduced even more to insure safe operation. If a table of volume multipliers[1,2] is available for the solvent in use, use this data to calculate to maximum allowable loading using the formula:

Max. Loading Volume = (0.9)(Vessel Volume)/Volume Multiplier at Max. Temp.

Limitations of the Material of Construction

Pressure vessels of identical design but of differing materials of construction will have vastly

[1] **"Steam Tables : Thermodynamic Properties of Water Including Vapor, Liquid, and Solid Phases/With Charts"** Joseph H. Keenan, Frederick G. Keyes, Philip G. Hill , Joan G. Moore, Krieger Pub Co, 1992.

[2] Parr Instrument Company document No. 230M: **"Safety in the Operation of Laboratory Reactors and Pressure Vessels"**

different pressure and temperature limits, as well as differing corrosion resistance towards solvents and reagents (acids and bases in particular). The material of construction of the vessel must be known and its limitations understood before initiating an experiment. For commercial reactor vessels, the user's manual and other documentation is an excellent resource for this information.[2]

Decontamination

Laboratory Specific Information

Waste Management

First Aid and Emergencies

Spill

Fire

Personnel Exposure

Prior Approval Required

- ☐ **NO**
- ☒ **YES (describe): Training on vacuum ovens or microwave reactor needed**

Designated Area

- ☐ **Entire Laboratory Area**
- ☐ **Other (describe):**

Experimental Conditions of Use

- **Temperature Range:**
- **Pressure Range:**
- **Scale Range:**
- **Other Relevant Details:**

Section II: UC & UCSB policies, procedures and resources

Section III: Appendices: PI responsibilities, Self-Inspection checklist, GHS classification system details, and a list of Particularly Hazardous Substances (PHS)