**Standard Operating Procedure**

**Pyrophoric Reagents Handling in Research Labs**

***This is an SOP template and is not complete until:*** *1) lab specific information is entered into the box below 2) lab specific protocol/procedure is added to the protocol/procedure section and
3) SOP has been signed and dated by the PI and relevant lab personnel.*

 Print a copy and keep with your
*Chemical Hygiene Plan* and/or *Lab Safety Resources Binder*

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| --- | --- |
| **Department:** | Click here to enter text. |
| **Date SOP was approved by PI/lab supervisor:** | Click here to enter a date. |
| **Principal Investigator:** | Click here to enter text. |
| **Lab Safety Coordinator/Lab Manager:** | Click here to enter text. |
| **Lab Phone:** | Click here to enter text. |
| **Office Phone:** | Click here to enter text. |
| **Emergency Contact:** | Click here to enter text. |
| *(Name and Phone Number)* |
| **Location(s) covered by this SOP:** | Click here to enter text. |
| *(Building/Room Number)* |

**Type of SOP:** X Process X Hazardous Chemical ☐Equipment

1. **Purpose**

This generic chemical safety guidance describes basic prudent safety practices for handling pyrophoric reagents in research labs. Pyrophoric reagents react with air or moisture in air; this reaction can readily lead to fire.

The principal investigator (PI) or the lab manager is responsible for developing and implementing standard operating procedures (SOPs) for the purchase, storage, and safe handling of pyrophoric reagents that are specific to the PI’s research.

1. **Hazards:**

Pyrophoric (pyrophorus or fire bearing) reagents such as organolithium compounds (alkyl-, alkenyl-, and alkynyl- lithium, etc.) can ignite spontaneously in air. Even small amounts of pyrophoric chemicals can initiate a lab fire.

Hazardous properties associated with pyrophoric reagents include: a) flammability, b) corrosivity, c) peroxide formation, d) teratogenicity, and e) target organ effects such as liver, kidneys, and central nervous system.

Pyrophoric reagents are not compatible with air, moisture, oxygen/oxidizer, and protic solvents including water, acids, alcohols, amines, mercaptans, etc.

1. **Training**

No researcher may work independently with the hazardous material described in this SOP until the Principal Investigator (or their designee) has ensured that the researcher:

* Has completed all required EH&S laboratory safety training programs
* Understands the hazards of the materials and risks of the processes involved
* Has read and understand the contents of this SOP and the lab's specific SOP
* Demonstrates the ability to execute their work according to the requirements in this SOP and the lab's specific SOP

All researchers should also be up-to-date on the Lab Safety Comprehensive training and must have taken Fire Extinguisher training.

***A. Review the safety procedures for handling highly reactive air-sensitive or moisture-sensitive reagents. If you wish to not require the review of any of the below options, you can replace these with training information required to your specific process.***

Everyone working with these compounds should be familiar with the Aldrich technical bulletins including:

1. [AL-134 Handling Air-Sensitive Reagents](https://www.sigmaaldrich.com/content/dam/sigma-aldrich/docs/Aldrich/Bulletin/al_techbull_al134.pdf)
2. [AL-164 Handling Pyrophoric Reagents](https://www.sigmaaldrich.com/content/dam/sigma-aldrich/docs/Aldrich/Bulletin/al_techbull_al164.pdf)
3. [Air-Sensitive Techniques](https://www.chemistryviews.org/details/education/3728881/Tips_and_Tricks_for_the_Lab_Air-Sensitive_Techniques_1.html) 1
4. [Air-Sensitive Techniques 2](https://www.chemistryviews.org/details/education/4308331/Tips_and_Tricks_for_the_Lab_Air-Sensitive_Techniques_2.html)
5. [Air-Sensitive Techniques 3](https://www.chemistryviews.org/details/education/4360441/Tips_and_Tricks_for_the_Lab_Air-Sensitive_Techniques_3.html)
6. [Transferring Air-Sensitive Reagents](http://research.unl.edu/docs/PyrophoricsWeb.pdf)

Recommended videos providing training on pyrophoric chemicals handling include:

1. The [University of California, San Diego (UCSD) video](http://chem-courses.ucsd.edu/CoursePages/Uglabs/143A_Weizman/EHS/EHS.html)
2. The Dartmouth Safety Video on Handling Pyrophoric Materials in Research Lab
3. Transfering <https://www.youtube.com/watch?v=WUHrzcEunNY>
4. Handling <https://www.youtube.com/watch?v=21iC4YEgOAs&t=52s>

*B.* ***Receive hands-on training***

All users of pyrophoric reagents in the laboratory must receive hands-on instruction from an experienced senior member of the laboratory (Principal Investigator or the person the PI delegates this training) and must be closely supervised until safe work practices are consistently demonstrated. This training must be documented so that proof of training is available upon request.

Researchers need hands-on training for the following:

* Emergency preparedness and response
* Use of Sure/Seal™ bottlesUse of Glove Box
* Storage requirements
* Process/Use of the chemical
1. **Safety Equipment**

*Location of nearest fire extinguisher:*

*Location of eyewash(s):*

*Location of safety shower:*

*Splash guard or safety shield (if reaction is preformed outside of a glove box):*

1. **Personal Protective Equipment (PPE)**

Wear PPE every time while working with pyrophoric chemicals. Exact PPE selection should depend on the severity of hazards associated with individual chemicals and handling processes.

*Proper Clothing:*

Avoid Wearing clothing made of flammable synthetic materials such as polyester, nylon, spandex, acetate and polypropylene under PPE.

A fire resistant (FR) lab coat is required. Blue FR lab coats are available via EH&S’s lab coat program.

Closed-toed shoes and long pants are required.

*Eye Protection*

Wear eye protection with chemical splash goggles or safety glasses with side shields and a face shield for additional protection as appropriate for the pyrophoric chemical reactions. If an explosive reaction is possible, a face shield and process safety shield are needed.

*Glove Use:*

Double nitrile gloves should be sufficient for handling research scale quantities of pyrophoric reagents in a research lab.

Wear flame-resistant gloves when handling large quantities of pyrophoric reagent in the lab. Nomex® flight gloves can be worn with thin 4 mil nitrile gloves as glove liners.

1. **Prudent Safety Practices**

*Pyrophoric reagents can be handled and stored safely if exposures to atmospheric oxygen and moisture are avoided.*

1. Do not work alone when handling pyrophoric reagents, especially organic lithium reagents and alkali metal hydrides.
2. Notify others in the laboratory prior to working with pyrophoric reagents.
3. Ideally, pyrophoric reagents should be stored under a blanket of an inert gas such as nitrogen before and after each dispensation from Sure/Seal™ bottles.
4. Do not store previously opened pyrophoric chemicals with other flammable materials or in a flammable solvents storage cabinet.
5. If refrigeration is needed, the fridge should be UL listed flammable or expolosion proof.
6. Use the smallest quantity of pyrophoric chemicals possible.
7. Perform all pyrophoric reagent transfers in a fume hood with the sash positioned at the lowest possible height with a splash guard or safety shield.
8. Utilize a splash guard or safety shield whenever possible.
9. Use the original Sure/Seal™ bottles for storing and dispending pyrophoric liquids by using a syringe or double-tipped needle. With proper handling and capping techniques, the Aldrich Sure/Seal™ cap that came with the original container can be used multiple times.
10. Conduct a hazard evaluation and process risks reduction assessment, and perform “dry runs” without pyrophoric reagent (e.g. using a pyrophoric reagent-free” solvent.
11. Deactivate or hydrolyze excess pyrophoric reagents and their residues using an appropriate hydroxyl solvent as per your lab SOP before discarding any empty containers.
12. Date Containers. Write on the container label the date that a pyrophoric material was received and the date the container was opened.
13. All glassware used for pyrophorics should be oven-dried and free of moisture.
14. Keep an appropriate fire extinguisher or extinguishing material close at hand.
15. Remove all other flammable material from the hood, as well as any clutter.
16. Secure the pyrophoric reagent bottle to a stand while in use.
17. Sigma Aldrich recommends the use of a long needle, 1-2 foot, and a syringe that is twice the volume of liquid to be transferred.
18. Further details, such as safe experiment setup diagrams, can be found in the references listed below.
19. **Process Description**

*Here you should input the proper steps for the process in which you are trying to use the pyroporic chemical. The process should be detailed and specific.*

1. **Storage**

Once opened, pyrophoric chemicals should be stored under an atmosphere of inert gas or under kerosene as appropriate. Avoid areas with heat/flames, oxidizers, and water sources. Containers carrying pyrophoric materials must be clearly labeled with the correct chemical name and hazard warning. Do NOT allow pyrophoric chemicals stored in solvent to dry out. Check periodically to ensure there is a visible amount of solvent in the bottle.

Never store in a non-explostion or flammable rated UL refrigerator. Once the chemical has been opened once, it should not be stored with other flammable or oxidizing materials.

1. **Emergency/Medical Treatment**

If pyrophoric reagents are not washed off immediately after the exposure, severe skin burn will occur. Even at very low concentrations, pyrophoric reagents will be irritating to the respiratory tract, eyes, and skin.

* Skin Contact: The affected skin area should be immediately rinsed off with copious amount of water at least for 15 to 20 minutes using the safety shower or eyewash as appropriate. This emergency procedure should be followed to ensure all pyrophoric chemicals are washed away from the affected skin area. Then seek medical attention
* Eye Contact: If a splash occurred near the eye, eyelid or eyelash area, irrigate eyes with water for at least 20 minutes and seek medical attention.
* If a person is exposed to fire or is on fire, the use of the “stop-drop and-roll” method, a safety shower, fire extinguisher are the most effective means of extinguishing fire that is on fire.
* Call 911 for medical emergencies.
1. **Spill Procedures:**

Keep spill response materials close to the pyrophoric work location. Pyrophoric specific spill adsorbents and fire extinguishing materials include dry sand, powdered soda ash (sodium carbonate), calcium oxide (lime), Celite® (diatomaceous earth), and clay-based kitty litter. Copious amounts of these materials should be used to completely cover/smother any pyrophoric spill in the research lab to minimize fire breakout. Once a pyrophoric material begins burning, it can be very difficult to extinguish.

1. A small beaker filled with sand can be used to safely extinguish small fires occurring at the tips of needles used to transfer liquid pyrophoric reagents.
2. The recommended fire extinguisher is a standard dry powder (ABC) type.
3. Class D extinguishers are recommended for combustible solid metal fires (such as sodium), but not for organolithium reagents. A CO2 fire extinguisher is not effective against pyrophoric reagents.
4. Seek additional help to clean up a large spill of pyrophoric chemicals. Turn off all ignition sources and vacate the laboratory immediately.
5. OSU Public Safety **must be notified of all fire related incidents.**

**Extinguish only if you can do so safely and quickly**

* After the fire is extinguished, call Public Safety – Dial.
* In case of emergency - Press 911.

**If the fire cannot be extinguished:**

* Confine the fire by closing the doors.
* Activate (pull) the nearest manual fire alarm, if there is one.
* Call the Fire Department - Press 911.
* Alert others in your area.
* Meet the Fire Department when they arrive.

After the use of a fire extinguisher, EH&S Fire Life Safety should be contacted to obtain a replacement.

If an accident happens the following documents must be completed:

* Online OSU HR Advocate Public Incident Reporting Form within 24 hours of the incident.
1. **Waste Disposal**

All materials that contain or are contaminated with pyrophoric reagents must be disposed of as hazardous waste by submitting the online request for Hazardous waste pick-up. Containers should be properly labeled for EHS pickup.

Before waste is created, submit a hazardous waste determintion so the OSU hazardous waste team can assist in proper disposal methods: <https://ehs.oregonstate.edu/waste>

*Disposal of Pyrophoric Reagents*

* A container with any residue of pyrophoric materials should never be left open to the atmosphere.
* Any unused or unwanted pyrophoric materials must be destroyed by transferring the materials to an appropriate reaction flask for hydrolysis and/or neutralization with adequate cooling.
* The essentially empty container should be rinsed three times with an inert dry solvent; this rinse solvent must also be neutralized or hydrolyzed.
* After the container is triple-rinsed and labeled as “EMPTY”, it should be left open in back of a hood or atmosphere at a safe location for at least a week. After the week, the container should then be rinsed 3 times again.
* Expired or unused reagent will be disposed of by EHS in the original commercial bottle. There is no need to empty, quench, or rinse these containers. Refer to earlier directions to add an appropriate solvent to containers when the solvent has dried out, prior to disposal by EHS.
* EHS will not accept pyrophoric chemicals for disposal when they are in an unstable condition or in a condition unsuitable for safe transportation. The laboratory may be responsible for the cost of stabilization if laboratory staff is unable to stabilize the chemicals or make them safe for transportation.

*Disposal of Pyrophoric Contaminated Materials*

* All materials – disposable gloves, wipers, bench paper, etc. - that are contaminated with pyrophoric chemicals should be disposed as hazardous waste.
* The contaminated waste should not be left overnight in the open laboratory but must be properly contained to prevent fires.
* Any container with residual pyrophoric chemicals must be stored under slightly positive nitrogen pressure in the headspace and these containers should NEVER be opened to the atmosphere.
* If a pyrophoric chemical that is stored in a solvent has dried out, then it must be CAREFULLY diluted within a fume hood with the solvent in which the reagent was originally stored. Use the laboratory’s specific SOP for dilution of pyrophoric reagents.
* For any significant amount of reagent remaining in the reagent bottle, first ensure that the bottle is purged with inert gas. Place a secondary container such as a sealable plastic bag (or the manufacturer supplied aluminum can in which the bottle was shipped) into an inert atmosphere and purge it with inert gas. Put the reagent bottle into this purged secondary container and seal for EHS pickup.
* With continuous purging of the headspace using inert gas flow, small amounts of liquid pyrophoric reagents and rinsates can be safely hydrolyzed/quenched by the drop-wise addition of reagents and rinsates into a reaction flask or a beaker containing isopropanol or isobutanol, while making sure that the container is kept in a cryogenic dry ice/isopropanol bath.
* If only trace amounts of the reagent remain in the reagent container, purge the container headspace with inert gas and triple rinse the reagent container using the solvent in which the reagent was originally stored. Rinsates can then be hydrolyzed as described above. Also, the above procedure should be followed for rinsing empty septa-sealed pyrophoric reagent bottles (such as Sure/Seal™ bottles) and rinsed off with water prior to disposal in a sharps container.
* Reaction mixtures containing pyrophoric reagents including BuLi, Grignard reagents, LiAlH4, NaBH4 and other reactive agents should be carefully and completely quenched with hydroxyl solvents before combining these with other waste solvents or packaging for disposal by EHS.
* Also, rinsate(s) resulting from quenching reactions should be collected in a waste solvent container.
1. **References**
* [Sure Seal™ Bottles – Standard Operating Procedure, University of Berkley](http://www.cchem.berkeley.edu/rsgrp/SOPs/Sure%20Seal%20bottles.doc)
* [Pyrophoric Chemicals Guide, University of Minnesota Environmental Health and Safety](http://www.dehs.umn.edu/PDFs/Pyrophoric_Chemicals_Guide.pdf)
* [Bretherick’s Handbook of Reactive Chemical Hazards Sixth addition Volume 1](file:///C%3A%5CUsers%5Cbamberma%5CDesktop%5Cbretherick-vol1.pdf)
* [Handling Pyrophoric Reagents](http://www.pnl.gov/main/publications/external/technical_reports/PNNL-18668.pdf) from U.S. Dept. of Energy and Pacific Northwest National Laboratory
* [Sigma-Aldrich Technical Bulletin AL-134](https://www.sigmaaldrich.com/content/dam/sigma-aldrich/docs/Aldrich/Bulletin/al_techbull_al134.pdf) – Handling Air-Sensitive Reagents
* [Sigma-Aldrich Technical Bulletin AL-164](https://www.sigmaaldrich.com/content/dam/sigma-aldrich/docs/Aldrich/Bulletin/al_techbull_al164.pdf) – Handling Pyrophoric Reagents
1. **Documentation of Training**

Prior to conducting any work with Pyrophoric Reagent Handling designated personnel must provide training to his/her laboratory personnel specific to the hazards involved in working with this substance, work area decontamination, and emergency procedures.

* The Principal Investigator must provide this SOP.
* The Principal Investigator must ensure that his/her laboratory personnel have attended appropriate laboratory safety training or refresher training.

**Principal Investigator SOP Approval**

By signing and dating here the designee certifies that the Standard Operating Procedure (SOP) for *Pyrophoric Reagent Handling* is accurate and effectively provides standard operating procedures for laboratory personnel.

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Signature Printed Name/Title Date

I have read and understand the content of this SOP:

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| --- | --- | --- |
| **Name** | **Signature** | **Date** |
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