**Standard Operating Procedure**

**Quenching and Disposal of t-ButylLithium (tBuLi) remaining in bottle or left after reactions**

Water-Reactive Materials, such as lithium, sodium, cesium, lithium aluminum hydride, potassium hydride, are designated by the following H codes: H260 and H 261. Please refer to the hazardous operation SOP “Quenching of Water Reactive Materials” for the chemicals.

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***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:** Process Hazardous Chemical Equipment

1. **Purpose**

This SOP covers the precautions and safe handling procedures for the Quenching and Dipsoal of t-ButylLithium (tBuLi) remaining in bottle or left after reactions.

If you have questions concerning the applicability of any recommendation or requirement listed in this procedure, contact the Principal Investigator/Laboratory Supervisor of your laboratory or EH&S.

1. **Physical & Chemical Properties/Definition of Chemical Group**

For physical and chemical properties on pyrophoric materials, please refer to specific Safety Data Sheets (SDS) of chemicals in use (See Section 11 – SDS Location).

1. **Potential Hazards**

When exposed to moisture or air, these reagents may ignite spontaneously. When quenching pyrophoric materials, the individual hazards of the pyrophorics and the solvent, as well as the hazard of the mixture, should all be considered, and procedures for safe quenching should reflect this consideration.

As defined by the Globally Harmonized System of Classification and Labeling of Chemicals (GHS), pyrophorics and self-heating substances are designated by one or more of the following H codes:

Pyrophorics

H250 Catches fire spontaneously if expose to air

Self-Heating Substances

H251 Self-heating; may catch fire

H252 Self-heating in large quantities; may catch fire

Please, review the SDS of any chemical before use (see Section 11 – SDS Location)

1. **Safety Data Sheet (SDS) Location**

Online SDS can be accessed at (<http://oregonstate.edu/ehs/sds>). A hard copy can be found at Oak Creek Building with Environmental Health & Safety.

1. **Engineering Controls**

The following is the set of engineering controls required when quenching pyrophoric materials:

* Work under an inert atmosphere (e.g., argon, nitrogen) using a Schlenk line, in a glove box, vacuum manifold, or any enclosed inert environment.
* Use a clean fume hood, preferably with the sliding sash windows or a glove box.
* If procedure is done in the fume hood, use the sash as a safety shield. For hoods with a horizontal sliding sash, position the sash all the way down, stand behind the sliding windows and reach around to perform the manipulations required. For hoods with vertical sliding sash, keep the sash as low as possible.
* Face shields are to be used when there is no protection from the hood sash or when the hood sash is open.
* Keep the materials under inert atmosphere when not in use.
* Remove any flammables (squirt bottles containing solvents, oil baths) and combustibles (Kimwipes, paper towels) from the area that will be used for the quenching.

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

At a minimum, the following PPE must be worn at all times:

**Eye Protection**

1. ANSI Z87.1-compliant safety glasses with side shields, or chemical splash goggles.

* Ordinary prescription glasses will NOT provide adequate protection unless they also meet ANSI standard and have compliant side shields.

1. If the potential for explosion/splashing exists, and adequate coverage is not provided by the hood sash, a face shield should be worn.

**Skin Protection**

1. Flame-resistant lab coat (Nomex IIIA, NFPA 2112) should be worn when working with pyrophoric and self-healing materials.
2. Gloves are required when handling hazardous materials. Refer to the specific chemical SDS for information on glove selection.
3. Long pants, closed-toe/closed-heel shoes, covered legs, and ankles. Cotton-based, non-synthetic clothing should be worn.
4. **Administrative Controls:**

* Anyone who uses Pyrophoirics is required to review this SOP and the attached Safety Data Sheet (SDS) prior to work.
* Keep quantities to a minimum and only order what you will be using.
* An eyewash must be available in the room with a safety shower accessible nearby.

1. **Best Practices for Safe Handling:**

Pyrophoric reagents can be handled and stored safely as long as all exposure to atmospheric oxygen and moisture or other incompatible chemicals is avoided. Never leave a container with a residue of a pyrophoric material open to the atmosphere.

Lab-specific information on handling and storage may be included in Section 12 - Protocol/Procedure section.

**Working alone**

OSU Environmental Health and Safety specifies not to work with pyrophorics alone or during off-hours when there are few people around to help.

**Handling and Storage of Pyrophoric Materials**

Process is to be performed under an inert atmosphere gas (use argon gas in the case of lithium metal).

Liquids may be safely transferred without the use of a glove box by employing techniques discussed in your laboratory “PYR” or “PYR, WR and SRA” Class SOP (syringe and cannula techniques).

Lab-specific information on handling and storage may be included in Section 13 Protocol/Procedure section.

1. **First Aid Procedures**

If an accident happens the following documents must be completed:

* Online OSU HR Advocate Public Incident Reporting Form within 24 hours of the incident
* If the employee’s incident resulted in the need for medical treatment, have the employee complete the worker section of the SAIF 801 Form and fax to risk management at 541-737-4855 within 24 hours.

**In case of skin contact**

If skin contact occurs, and/or skin or clothing are on fire, immediately drench in the safety shower with copious amounts of water for no less than 15 minutes to remove any remaining contaminants. If possible to do so without further injury, remove any remaining jewelry or clothing.

**In case of eye contact**

Rinse thoroughly with plenty of water using an eyewash station for at least 15 minutes, occasionally lifting the upper and lower eyelids. Remove contact lenses if possible.

<http://ehs.oregonstate.edu/sites/ehs.oregonstate.edu/files/pdf/si/eyewash_and_safety_shower_si.pdf>

**If swallowed**

Do NOT induce vomiting unless directed otherwise by the SDS. Never give anything by mouth to an unconscious person. Rinse mouth with water.

**Needle stick/puncture exposure**

Wash the affected area with antiseptic soap and warm water for 15 minutes. For mucous membrane exposure such as eyes, mouth and/or nose, flush the affected area for 15 minutes using an eyewash station.

**If inhaled**

Move into freshair.

1. **Chemical Spill**

**OSU Chemical Spill Safety Instruction**: <http://ehs.oregonstate.edu/sites/ehs.oregonstate.edu/files/pdf/si/spill_response-chemicals_si.019.pdf>

**Pyrophoric Spill Response**

* In the case of a spill, announce the situation loudly in the immediate area and have any nearby persons move to a safe location.
* Immediately eliminate/remove all nearby ignition sources.
* If spill occurs in a fume hood, cover with Met-L-X, dry sand, or other non-combustible material, close the hood sash and if present, press the red purge button.
* If a spill occurs outside a fume hood, cover with Met-L-X, dry sand, or other non-combustible material, and stand away from the spill.
* Locate and have a proper fire extinguisher (dry chemical-based) ready in case of ignition/fire.
* Use clean, non-sparking tools to collect absorbed material and place into loosely-covered metal or plastic containers ready for disposal.
* Do not use combustible materials (paper or cloth towels) to clean up a spill, as these may increase the risk of igniting the reactive compound.
* If you cannot assess the situation well enough to be sure of your own safety, do not approach the spill.
* Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).• Report the spill to 541-714-SAFE.

**Pyrophoric Fire Response**

* Call 911 for emergency assistance and for assistance with all fires, even if extinguished.
* If the spill ignites, and if you are trained and you feel comfortable to do so, consider extinguishing the fire with an appropriate fire extinguisher. Only dry chemical fire extinguishers should be used (classes ABC or D). Do not use a CO2 extinguisher.
* A can of Met-L-X or dry sand in the work area, within arm’s reach, might be helpful to extinguish any small fire as it can smother the flames.
* Do not use water to extinguish a pyrophoric chemical fire as it may enhance the intensity of the fire. An exception to this would be in the case of skin contact or ignited clothing/skin. In these cases rinsing any unreacted chemical off is of primary importance.
* Be AWARE: Small flames at the tip of the needles can be produced – always expect this to occur, and do not panic. The can of Met-L-X/sand is in the hood to quickly extinguish those small flames.

1. **Decontamination/Waste Disposal Procedure**

Lab-specific information on decontamination may be included in the Protocol/Procedure section.

* Wearing proper PPE, laboratory work surfaces should be cleaned at the end of each workday.
* Store liquid wastes in designated waste containers. Dispose of according the OSU EH&S hazardous waste guidelines.

**Disposal of Pyrophoric Reagents**

* Never leave a container with a residue of a pyrophoric material open to the atmosphere.
* Any unused or unwanted pyrophoric materials must be destroyed by following the specific destruction procedure(s) included in your “Hazardous Operation SOP – Quenching of Pyrophoric Materials”.
* Rinse the empty container three times with an inert dry COMPATIBLE solvent. The rinse solvent must be transferred in and out of the container under an inert atmosphere using the syringe or cannula technique.
* Solvent rinses must be disposed of as hazardous waste.

**Label Waste**

* Affix an EH&S hazardous waste label on all waste containers (<http://ehs.oregonstate.edu/sites/ehs.oregonstate.edu/files/pdf/hwlabelfull.pdf>) as soon as the first drop of waste is added to the container.

**Store Waste**

* Store hazardous waste in closed containers, in secondary containment and in a designated location. (<http://ehs.oregonstate.edu/sites/ehs.oregonstate.edu/files/pdf/si/waste_hazardous_disposal_si.pdf)>.
* Waste must be under the control of the person generating & disposing of it

**Dispose of Waste**

* Dispose of regularly generated chemical waste within 90 days
* Put in a waste request at: <http://ehs.oregonstate.edu/waste>

1. **References**

[Include any references useful to employees]

[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

University of Minnesota, Environmental Health and Safety – “[Pyrophoric Chemicals Guide](https://www.google.com/url?client=internal-uds-cse&cx=000747455861485679472:bqyjqaw6sp8&q=http://www.me.umn.edu/intranet/safety/doc/Pyrophoric%2520Chemicals%2520Guide.docx&sa=U&ved=2ahUKEwjQirSIl6HkAhXoFjQIHbAhCnAQFjAAegQIAxAB&usg=AOvVaw0pnB6xupRki_lEz4dnLxKZ)”

UCLA, Environmental Health and Safety – [“Pyrophoric Liquid Safety Video”](https://www.youtube.com/watch?v=21iC4YEgOAs)

University of Mass Amherst – [SOP Pyrophoric Liquids and Solids](https://ehs.umass.edu/sites/default/files/Pyrophoric%20Liquids%20or%20Solids%20SOP.pdf)

[UCSD - Transferring Pyrophoric Chemicals Video](https://www.youtube.com/watch?v=WUHrzcEunNY&feature=youtu.be)

[UCSD - Working with Pyrophoric Reagents Video Part 1](https://www.youtube.com/watch?v=3_cBVfYVAC8&feature=youtu.be)

1. **Protocol/Procedure for: Quenching of Water Reactive Materials**

**Preparation**

*[List any other particular preparation requirements needs for this procedure (e.g., location of spill kit or keep water or ignition sources away from procedure area]*

* Know the location of the nearest fire extinguisher, eyewash, and safety shower before beginning work.
* Have a small can of Met-L-X or dry sand in the work area, within arm’s reach.
* Solvents must be dry.
* Glassware must be dry before using. Either “flame” dry or dry in an oven at a minimum temperature of 100° C for at least two hours.
* Remove all other flammable/combustibles materials from the hood to reduce the hazard in case of a fire. Make adjacent lab workers aware that you will be working with pyrophoric chemicals.

**Quenching of t-ButylLithium (t-BuLi) remaining in bottle or left after reactions**

This procedure is to be used for up to 20 mL of liquid pyrophoric material as supplied in the reagent bottle. Quenching of t-BuLi in amounts greater than 20 ml at a time is not permitted.

See note for appropriate glassware size selection.

Note on Glassware Size: If quenching 20mL of pyrophoric solution, the pyrophoric material will be diluted in 400mL of inert solvent to start the quenching procedure (5% wt solution). In this case, the required size for the quenching vessel is 1L, no less.

- **Quenching in a FUME HOOD using a Schlenk line -**

General considerations:

Quenching procedures usually involve the reaction of the pyrophoric material, under inert atmosphere and on an ice bath, with a reagent that has a reactive hydroxyl group.

Any reaction or suspension containing these reagents MUST be quenched carefully! All transfers should be done under inert atmosphere using the syringe or cannula techniques.

Addition of material should be done SLOWLY, under adequate stirring/mixing.

Whenever quenching, be sure that it is not done in a sealed vessel as pressure will build up. The best situation is to have an inert gas flowing in with a small exit needle connected to a bubbler.

If you’re quenching in a round-bottom flask or any floating vessel in a bath, clamp it. If the solution level is low, you can dilute it with some inert solvent such as toluene or hexane first to less than 5% wt.

Procedure:

1. Transfer the t-BuLi solution to the quenching vessel, bring the temperature of the quench solution to -78°C using a dry ice/isopropanol bath, turn on the stirrer.
2. Before proceeding with the quenching of the pyrophoric solution, immediately proceed with the cleaning of the empty bottle.
3. The empty container should be rinsed three times with an inert dry COMPATIBLE solvent such as toluene or hexane. The rinse solvent must be transferred in and out of the container under an inert atmosphere using the syringe or the cannula technique.
4. After the empty container is triple-rinsed, remove the SureSeal cap and leave it open in the back of the fume hood overnight.
5. Dispose of the solvent rinse as hazardous waste.
6. Proceed with the quenching of the pyrophoric solution by adding isopropanol slowly to the quenching vessel using a syringe or an addition funnel, under adequate stirring, until no more bubbling Needlestick.
7. Keep the solution cool during the quenching process by controlling the feed rate of the alcohol.
8. Once you've added the isopropanol, add the following solvents in sequence, using the same step-wise caution: ethanol, methanol, and water.
9. Be Very Careful with the addition of water! Even after methanol has been added, the lithiate can still react violently with water, especially is there hasn't been sufficient mechanical stirring of the solution. Add water in small aliquots. The addition of water does not need to be conducted under an inert atmosphere. The use of a syringe (plastic or glass) is recommended.
10. Once the addition of water is complete, let the solution temperature come back to room temperature while stirring.
11. Stir for an additional 2 hours before disposing of the aqueous organic waste.

**- Quenching in a GLOVE BOX -**

General considerations:

Any reaction or suspension containing these reagents MUST be quenched carefully! Addition of solvents should be done SLOWLY with adequate stirring/mixing.

If the glove box is equipped with a fridge, DO NOT open the fridge during the quenching process.

Procedure:

1. Turn OFF the glove box circulation.
2. Remove bottle SureSeal cap or reaction vessel cap and add a stirrer if necessary.
3. Typically, a solution of less than 5%wt of pyrophoric in an inert solvent (such as dry and degassed hexane or heptane) is created, followed by the slow addition of dry and degassed isopropanol, under adequate stirring until no more bubbling is observed.
4. Place a cap on the quenching vessel and put the bottle/vessel in a zip-top bag before taking it out of the glove box.
5. Move the quenching vessel to a clean fume hood. Always place the quenching vessel in a secondary container for transfer to the fume hood.
6. Purge the glove box for 20-30 minutes.
7. Under adequate stirring, add water SLOWLY to the quenching solution until no more bubbling is observed. This step does not need to be conducted under an inert atmosphere. The use of a syringe (plastic or glass) is recommended.
8. Be Very Careful with the addition of WATER! Even after isopropanol has been added, the lithiate can still react violently with water, especially is there hasn't been sufficient mechanical stirring of the solution. Therefore the water should be added in small aliquots.
9. Stir for an additional 2 hours before disposing of the aqueous organic waste.
10. The empty container should be rinsed three times with an inert dry COMPATIBLE solvent such as toluene or hexane.
11. After the empty container is triple-rinsed, it should be left open in back of a hood overnight.
12. Solvent rinses should be disposed of as hazardous waste.

**Documentation of Training**

* Prior to conducting any work with pyrophorics, designated personnel must provide training to their 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 and a copy of the SDS (can be available online) available to all laboratory personnel.
* 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 *Quenching and Disposal of t-ButylLithium (tBuLi) remaining in bottle or left after reactions* is accurate and effectively provides standard operating procedures for laboratory personnel.

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Signature Printed Name/Title Date

I have read and understood the content of this SOP:

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