

## Quenching of Pyrophoric Materials

H250 H251 H252



Examples: tert-ButylLithium, sec-ButylLithium, n-Butyllithium, DiethylZinc, Organoaluminum compounds (as Et<sub>3</sub>Al, Et<sub>2</sub>AlCl, EtAlCl<sub>2</sub>, Me<sub>3</sub>Al), Raney Nickel catalyst

**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.

Department:	Chemistry
Date SOP was written:	March 23, 2015
Date SOP was approved by PI/lab supervisor:	March 25, 2015
Principal Investigator:	Name: Richmond Sarpong Signature: _____
Internal Lab Safety Coordinator or Lab Manager:	Name: Paul Leger Lab Phone: 510-643-2485 Office Phone: 510-642-6312
Emergency Contact:	Name: Richmond Sarpong Phone Number: 626-644-2407
Location(s) covered by this SOP:	834, 836, 837, 838, 839, 842, 844, 847, 849, 907

### 1. Purpose

This SOP covers the precautions and safe handling procedures for the Quenching of Pyrophoric Materials.

All pyrophoric materials mentioned in your laboratory “**Pyrophorics**” (PYR) or “**Pyrophoric, Water Reactive and Strong Reducing Agent Chemicals**” Class SOP (PYR, WR and SRA) are covered by this hazardous operations SOP.

Also, this SOP covers any materials synthesized using pyrophoric liquids or solids.

***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 the campus Chemical Hygiene Officer at [ucbcho@berkeley.edu](mailto:ucbcho@berkeley.edu).***

### 2. Physical & Chemical Properties



For physical and chemical properties on pyrophoric materials, please refer to your laboratory “PYR” or “PYR, WR and SRA” Class SOP and to specific Safety Data Sheets (SDS) of chemicals in use (See Section 11 – SDS Location).

### 3. 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 exposed to air

Self-Heating Substances

**H251** Self-heating; may catch fire

**H252** Self-heating in large quantities; may catch fire

### 4. 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 (spray bottles containing solvents, oil baths) and combustibles (Kimwipes, paper towels) from the area that will be used for the quenching.

### 5. Personal Protective Equipment

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

#### Eye Protection

- A. 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.
- B. 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



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

## 6. First Aid Procedures and Medical Emergencies

***In the event of an injury, notify your supervisor immediately and EH&S within 8 hours. Follow up with a call to 510-642-6060 to report the incident.***



***Go to the Occupational Health Facility (Tang Health Center, on campus); if after hours, go to the nearest emergency room (Alta Bates, 2450 Ashby Ave in Berkeley); or***



***Call 911 (from a cell phone: 510-642-3333) if:***

- ***it is a life threatening emergency; or***
- ***you not are confident in your ability to fully assess the conditions of the environment and/or the condition of the contaminated/injured person, or you cannot be assured of your own safety; or***
- ***the contaminated/injured person is not breathing or is unconscious.***

***Please remember to provide a copy of the appropriate manufacturer SDS (if available) to the emergency responders or physician. At a minimum, be ready to provide the identity/name of any hazardous materials involved.***

### **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.

### **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 fresh air.

## 7. Special Handling and Storage Requirements

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.

### **Working Alone**



The UC Berkeley Office of 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 12 - Protocol/Procedure section.

## 8. Chemical Spill and Fire Response

### 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 510-642-3073.

### Pyrophoric Fire Response

- Call **911** (from a cell phone: **510-642-3333**) 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.

## 9. Cleaning and Decontamination

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 work day.



- Dispose of contaminated materials in accordance with hazardous waste disposal guidelines referenced below.
- Clean all equipment before removing from a designated area.

## **10. Hazardous Waste Disposal**

### Label Waste

Label all waste containers. See the EH&S Fact Sheet, "Hazardous Waste Management" for general instructions on procedures for disposing of hazardous waste.

### Dispose of Waste

- Dispose of regularly generated chemical waste within 6 months.
- Call EH&S with questions.

## **11. SDS Location**

SDS can be accessed online at <http://ucmsds.com>



## 12. Protocol/Procedure for: Quenching of Pyrophoric Materials

<p>Preparation</p>	<p><i>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)</i></p> <ul style="list-style-type: none"><li>• Know the location of the nearest fire extinguisher, eyewash, and safety shower before beginning work.</li><li>• Have a small can of Met-L-X or dry sand in the work area, within arm's reach.</li><li>• Solvents must be dry.</li><li>• 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.</li><li>• 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.</li></ul> <p><b>NOTE: As an alternative to quenching, remember that pyrophoric chemicals can be disposed of as hazardous waste. If this option is chosen:</b></p> <ul style="list-style-type: none"><li>• <b>Carefully package and label the waste with current HWP labels.</b></li><li>• <b>Request waste pick up from EH&amp;S</b></li></ul>
<p><b>Procedure</b></p> <p>1</p> <p>2</p> <p>3</p>	<p>Quenching of t-Butyllithium (t-BuLi) remaining in bottle or left after reactions</p> <p>Quenching of pyrophoric liquids, such as sec-BuLi, n-BuLi, Grignard reagents, remaining in bottle or left after reactions</p> <p>Quenching of pyrophoric solids, such as finely divided metals (magnesium, zirconium, ...), non-metals (white phosphorus)</p>



## Hazardous Operation SOP

Procedure/Use	Scale	Engineering Controls/Equipment	PPE (eye, face, gloves, clothing)	Procedure Steps and Special Precautions for this Procedure
<p>1. Quenching of t-ButylLithium (t-BuLi) remaining in bottle or left after reactions</p>	<p>This procedure is to be used for up to <b>20 mL</b> of liquid pyrophor material as supplied in the reagent bottle. Quenching of t-BuLi in amounts greater than 20 ml at a time is not permitted.</p> <p><i>See note for appropriate glassware size selection.</i></p> <p>Minimal quantities will be left after reactions carried out at scales listed in your laboratory "PYR" or "PYR, WR and SRA" Class SOP.</p> <p>As an alternative to</p>	<p>Conduct in a clean fume hood with the sash closed using the Schlenk techniques, or an inert atmosphere glove box.</p> <p>If using outside an inert atmosphere glove box, ensure the receiving vessel is dry and a blanket of inert gas is kept over the air sensitive chemicals.</p>	<p><b>Eye Protection:</b> Wear fitted safety goggles or safety glasses with side shields.</p> <p><b>Face Protection:</b> Face shields are to be used when there is no protection from the hood sash or when the hood sash is open.</p> <p><b>Hand Protection:</b> Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Wash and dry hands after use.</p> <p>Hand Protection for <u>indirect contact with pyrophoric material</u> (closed-system procedures such as transfers via syringe or cannula): Use nitrile gloves of at least 10 mil thickness (double-glove if 5 mil thickness).</p> <p>Hand Protection for <u>direct contact with pyrophoric material</u> (open system procedures such as spill handling, wiping of residual pyrophorics): Provide for adequate employee hand protection by evaluating any additional risks via a hazard assessment and provide the appropriate hand protection.</p>	<p><b>Note on Glassware Size:</b> <i>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.</i></p> <p><b>- Quenching in a FUME HOOD using a Schlenk line -</b></p> <p><u>General considerations:</u></p> <p>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.</p> <p>Any reaction or suspension containing these reagents <b>MUST</b> be quenched carefully! All transfers should be done under inert atmosphere using the syringe or cannula techniques.</p> <p>Addition of material should be done <b>SLOWLY</b>, under adequate stirring/mixing.</p> <p>Whenever quenching, be sure that it is <u>not</u> 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.</p> <p>If you're quenching in a RB flask or any floating vessel in a bath, <b>clamp it</b>. If the solution level is low, you can dilute it with some inert solvent such as <b>toluene or hexane</b> first to less than 5% wt.</p> <p><u>Procedure:</u></p> <p>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.</p> <p>Before proceeding with the quenching of the</p>



## Hazardous Operation SOP

	<p>quenching, remember that any quantities of t-BuLi can be disposed of as hazardous waste.</p>		<p>Consider the use of fire resistant (FR) gloves or liners.</p> <p><b>Clothing:</b> Wear Nomex IIIA (NFPA 2112) lab coat; cotton based clothing; full length pants or equivalent; and close-toed and close-heeled shoes.</p>	<p>pyrophoric solution, immediately proceed with the cleaning of the empty bottle.</p> <p>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.</p> <p>After the empty container is triple-rinsed, remove the SureSeal cap and leave it open in the back of the fume hood overnight.</p> <p>Dispose of the solvent rinse as hazardous waste.</p> <p>Proceed with the quenching of the pyrophoric solution by adding isopropanol <b>slowly</b> to the quenching vessel using a syringe or an addition funnel, under adequate stirring, until no more bubbling is observed.</p> <p>Keep the solution cool during the quenching process by controlling the feed rate of the alcohol.</p> <p>Once you've added the isopropanol, add the following solvents in sequence, using the same step-wise caution: ethanol, methanol, and water.</p> <p><b>Be Very Careful with the addition of water!</b> Even after methanol has been added, the lithiate can still react violently with water, especially if 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.</p> <p>Once the addition of water is complete, let the solution temperature come back to room temperature while stirring.</p> <p>Stir for an additional 2 hours before disposing of the aqueous organic waste.</p>
--	---	--	---	--





## Hazardous Operation SOP

				<p><b>- Quenching in a GLOVE BOX -</b></p> <p><u>General considerations:</u></p> <p>Any reaction or suspension containing these reagents <b>MUST</b> be quenched carefully! Addition of solvents should be done <b>SLOWLY</b> with adequate stirring/mixing.</p> <p>If the glove box is equipped with a fridge, <b>DO NOT</b> open the fridge during the quenching process.</p> <p><u>Procedure:</u></p> <p>Turn OFF the glove box circulation.</p> <p>Remove bottle SureSeal cap or reaction vessel cap and add a stirrer if necessary.</p> <p>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 <b>slow</b> addition of dry and degassed isopropanol, under adequate stirring until no more bubbling is observed.</p> <p>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.</p> <p>Move the quenching vessel to a clean fume hood. Always place the quenching vessel in a secondary container for transfer to the fume hood.</p> <p>Purge the glove box for 20-30 minutes.</p> <p>Under adequate stirring, add water <b>SLOWLY</b> 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.</p> <p><b>Be Very Careful with the addition of WATER!</b> Even after isopropanol has been added, the lithiate can still react violently with water, especially if there hasn't</p>
--	--	--	--	--



## Hazardous Operation SOP

				<p>been sufficient mechanical stirring of the solution. Therefore the water should be added in small aliquots.</p> <p>Stir for an additional 2 hours before disposing of the aqueous organic waste.</p> <p>The empty container should be rinsed three times with an inert dry COMPATIBLE solvent such as toluene or hexane.</p> <p>After the empty container is triple-rinsed, it should be left open in back of a hood overnight.</p> <p>Solvent rinses should be disposed of as hazardous waste.</p>
Notes	Any deviation from this SOP requires approval from PI.			



## Hazardous Operation SOP

Procedure/Use	Scale	Engineering Controls/Equipment	PPE (eye, face, gloves, clothing)	Procedure Steps and Special Precautions for this Procedure
<p>2. Quenching of pyrophoric liquids, such as, but not limited to, sec-BuLi, n-BuLi, Grignard reagents, remaining in bottle or left after reactions</p>	<p>This procedure is to be used for up to <b>100 mL</b> of liquid pyrophor material as supplied in the reagent bottle. Quenching of pyrophoric materials in amounts greater than 100 ml at a time is not permitted.</p> <p><i>See note for appropriate glassware size selection.</i></p> <p>Minimal quantities will be left after reactions carried out at scales listed in your laboratory "PYR" or "PYR, WR and SRA" Class SOP.</p> <p>As an alternative to quenching,</p>	<p>Conduct in a clean fume hood with the sash closed using the Schlenk techniques, or an inert atmosphere glove box.</p> <p>If using outside an inert atmosphere glove box, ensure the receiving vessel is dry and a blanket of inert gas is kept over the air sensitive chemicals.</p>	<p><b>Eye Protection:</b> Wear fitted safety goggles or safety glasses with side shields.</p> <p><b>Face Protection:</b> Face shields are to be used when there is no protection from the hood sash or when the hood sash is open.</p> <p><b>Hand Protection:</b> Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Wash and dry hands after use.</p> <p>Hand Protection for <u>indirect contact with pyrophoric material</u> (closed-system procedures such as transfers via syringe or cannula): Use nitrile gloves of at least 10 mil thickness (double-glove if 5 mil thickness).</p> <p>Hand Protection for <u>direct contact with pyrophoric material</u> (open system procedures such as spill handling, wiping of residual pyrophorics): Provide for adequate employee hand protection by evaluating any additional risks via a hazard assessment and provide the appropriate hand protection.</p>	<p><b>Note on Glassware Size:</b> <i>If quenching 100mL of pyrophoric solution, the pyrophoric material will be diluted in 500mL of inert solvent to start the quenching procedure (20% wt solution). In this case, the required size for the quenching vessel is 1L, no less.</i></p> <p><b>- Quenching in a FUME HOOD using a Schlenk line -</b></p> <p><u>General considerations:</u></p> <p>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.</p> <p>Any reaction or suspension containing these reagents <b>MUST</b> be quenched carefully! All transfers should be done under inert atmosphere using the syringe or cannula techniques.</p> <p>Addition of material should be done <b>SLOWLY</b>, under adequate stirring/mixing.</p> <p>Whenever quenching, be sure that it is <u>not</u> 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.</p> <p>If you're quenching in a RB flask or any floating vessel in a bath, <b>clamp it</b>. If the solution level is low, you can dilute it with some inert solvent such as <b>toluene or hexane</b> first to less than 20% wt.</p> <p><u>Procedure:</u></p> <p>Transfer the diluted solution to the quenching vessel, place an ice under the flask, and turn on the stirrer.</p> <p>Before proceeding with the quenching of the pyrophoric solution, proceed with the cleaning of the</p>



## Hazardous Operation SOP

	<p>remember that any quantities of t-BuLi can be disposed of as hazardous waste.</p>		<p>Consider the use of fire resistant (FR) gloves or liners.</p> <p><b>Clothing:</b> Wear Nomex IIIA (NFPA 2112) lab coat; cotton based clothing; full length pants or equivalent; and close-toed and close-heeled shoes.</p>	<p>empty bottle.</p> <p>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.</p> <p>After the empty container is triple-rinsed, remove the SureSeal cap and it should be left open in back of a hood overnight.</p> <p>Dispose of the solvent rinse as hazardous waste.</p> <p>Proceed with the quenching of the pyrophoric solution by adding isopropanol <b>slowly</b> to the quenching vessel using a syringe or an addition funnel, under adequate stirring, until no more bubbling is observed.</p> <p>Keep the solution cool (50°C or below) during the quenching process by controlling the feed rate of the alcohol.</p> <p>Once you've added the isopropanol, add the following solvents in sequence, using the same step-wise caution: ethanol, methanol, and water.</p> <p><b>Be Very Careful with the addition of water!</b> Even after methanol has been added, the lithiate can still react violently with water, especially if 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.</p> <p>Once the addition of water is complete, let the solution temperature come back to room temperature while stirring.</p> <p>Stir for an additional 2 hours before disposing of the aqueous organic waste.</p>
--	--	--	---	---



## Hazardous Operation SOP

				<p>- <b>If quenching in a FUME HOOD using dry ice -</b></p> <p><i>Place dry ice in a container and add a stir bar. Pyrophoric liquids in this section can be quenched by slowly adding the dilute solution directly onto dry ice, then adding a mildly reactive quenching agent such as methanol. Once the addition of the dilute solution is complete, let the solution temperature come back to room temperature under stirring.</i></p> <p>Stir for an additional 2 hours before disposing of the aqueous organic waste.</p> <p>- <b>If quenching in a GLOVE BOX -</b></p> <p><u>General considerations:</u></p> <p>Any reaction or suspension containing these reagents <b>MUST</b> be quenched carefully! Addition of solvents should be done <b>SLOWLY</b> with adequate stirring/mixing.</p> <p>If the glove box is equipped with a fridge, <b>DO NOT</b> open the fridge during the quenching process.</p> <p><u>Procedure:</u></p> <p>Turn OFF the glove box circulation.</p> <p>Remove bottle or reaction vessel cap and add a stirrer if necessary.</p> <p>Typically, a solution of less than 20%wt of pyrophoric in an inert solvent (such as dry and degassed hexane or heptane) is created , followed by the <b>slow</b> addition of dry and degassed isopropanol, under adequate stirring until no more bubbling is observed.</p> <p>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.</p> <p>Move the quenching vessel to a clean fume hood. Always place the quenching vessel in a secondary</p>
--	--	--	--	---



## Hazardous Operation SOP

				<p>container for transfer to the fume hood.</p> <p>Purge the glove box for 20-30 minutes.</p> <p>Continue the quenching procedure in a clean fume hood with sash closed.</p> <p>Under adequate stirring, add water <b>SLOWLY</b> 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.</p> <p><b>Be Very Careful with the addition of WATER!</b> Even after isopropanol has been added, the lithiate can still react violently with water, especially if there hasn't been sufficient mechanical stirring of the solution. Therefore the water should be added in small aliquots.</p> <p>Once the addition of water is complete, stir for an additional 2 hours before disposing of the aqueous organic waste.</p> <p>The empty container should be rinsed three times with an inert dry COMPATIBLE solvent such as toluene or hexane.</p> <p>After the empty container is triple-rinsed, it should be left open in back of a hood overnight.</p> <p>Solvent rinses should be disposed of as hazardous waste.</p>
Notes	Any deviation from this SOP requires approval from PI.			



## Hazardous Operation SOP

Procedure/Use	Scale	Engineering Controls/Equipment	PPE (eye, face, gloves, clothing)	Procedure Steps and Special Precautions for this Procedure
<p>3. Quenching of pyrophoric solids</p>	<p>Up to <b>10g</b> of material.</p> <p><i>See note for appropriate glassware size</i></p> <p>Minimal quantities will be left after reactions carried out at scales listed in your laboratory “PYR” or “PYR, WR and SRA” Class SOP.</p> <p>As an alternative to quenching, remember that any quantities of t-BuLi can be disposed of as hazardous waste.</p>	<p>Conduct in a clean fume hood with the sash closed using the Schlenk techniques, or an inert atmosphere glove box.</p> <p>If using outside an inert atmosphere glove box, ensure the receiving vessel is dry and a blanket of inert gas is kept over the air sensitive chemicals.</p>	<p><b>Eye Protection:</b> Wear fitted safety goggles or safety glasses with side shields.</p> <p><b>Face Protection:</b> Face shields are to be used when there is no protection from the hood sash or when the hood sash is open.</p> <p><b>Hand Protection:</b> Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Wash and dry hands after use.</p> <p>Hand Protection for <u>indirect contact with pyrophoric material</u> (closed-system procedures such as transfers via syringe or cannula): Use nitrile gloves of at least 10 mil thickness (double-glove if 5 mil thickness).</p> <p>Hand Protection for <u>direct contact with pyrophoric material</u> (open system procedures such as spill handling, wiping of residual pyrophorics): Provide for adequate employee hand protection by evaluating any additional risks via a hazard assessment and provide the appropriate hand protection.</p>	<p><b>Note on Glassware Size:</b> <i>If quenching 100mL of pyrophoric solution, the pyrophoric material will be diluted in 500mL of inert solvent to start the quenching procedure (20% wt solution). In this case, the required size for the quenching vessel is 1L, no less.</i></p> <p><b>- If quenching in a FUME HOOD using a Schlenk line -</b></p> <p><u>General considerations:</u></p> <p>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.</p> <p>Any reaction or suspension containing these reagents <b>MUST</b> be quenched carefully! All transfers should be done under inert atmosphere using the syringe or cannula techniques.</p> <p>Addition of material should be done <b>SLOWLY</b> and ensure adequate stirring/mixing.</p> <p>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.</p> <p>If you’re quenching in a RB flask or any floating a vessel in a bath, <b>clamp it.</b></p> <p><u>Procedure:</u></p> <p>Typically, a suspension of less than 20%wt of pyrophoric in an inert solvent (such as hexane or toluene) is created, followed by the <b>slow</b> addition of isopropanol using a syringe or an addition funnel, under adequate stirring until no more bubbling is observed.</p> <p>During the quenching process, keep the solution cool,</p>



## Hazardous Operation SOP

			<p>Consider the use of fire resistant (FR) gloves or liners.</p> <p><b>Clothing:</b> Wear Nomex IIIA (NFPA 2112) lab coat; cotton based clothing; full length pants or equivalent; and close-toed and close-heeled shoes.</p>	<p>50°C or below, by application of an external cooling bath (ice bath) and by controlling the feed rate of the alcohol.</p> <p>Once you've added the isopropanol, add the following solvents in sequence, using the same step-wise caution: ethanol, methanol, and water.</p> <p><b>Be Very Careful with the addition of water!</b> Even after methanol has been added, the lithiate can still react violently with water, especially if 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.</p> <p>Once the addition of water is complete, let the solution temperature come back to room temperature while stirring.</p> <p>Stir for an additional 2 hours before disposing of the aqueous organic waste.</p> <p>The empty container should be rinsed three times with an inert dry COMPATIBLE solvent.</p> <p>After the empty container is triple-rinsed, it should be left open in back of a hood overnight.</p> <p>Solvent rinses should be disposed of as hazardous waste.</p> <p><b>- If quenching in a GLOVE BOX -</b></p> <p><u>General considerations:</u></p> <p>Any reaction or suspension containing these reagents <b>MUST</b> be quenched carefully! Addition of solvents should be done <b>SLOWLY</b> with adequate stirring/mixing.</p> <p>If the glove box is equipped with a fridge, <b>DO NOT</b> open the fridge during the quenching process.</p>
--	--	--	---	--





## Hazardous Operation SOP

			<p><u>Procedure:</u></p> <p>Turn OFF the glove box circulation.</p> <p>Remove bottle or reaction vessel cap and add a stirrer if necessary.</p> <p>Typically, a suspension of less than 20%wt of pyrophoric in an inert solvent (such as dry and degassed hexane or heptane) is created, followed by the <b>slow</b> addition of dry and degassed isopropanol, under adequate stirring until no more bubbling is observed.</p> <p>Place a cap on the quenching vessel and put the bottle/vessel in a Ziploc bag before taking it out of the glove box.</p> <p>Move the quenching vessel to a clean fume hood. Always place the quenching vessel in a secondary container for transfer to the fume hood.</p> <p>Purge the glove box for 20-30 minutes.</p> <p>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.</p> <p>Be Very Careful with the addition of WATER! Even after isopropanol has been added, the lithiate can still react violently with water, especially if there hasn't been sufficient mechanical stirring of the solution. Therefore the water should be added in small aliquots.</p> <p>Once the addition of water is complete, stir for an additional 2 hours before disposing of the aqueous organic waste.</p> <p>The empty container should be rinsed three times with an inert dry COMPATIBLE solvent.</p>
--	--	--	--



### Hazardous Operation SOP

				After the empty container is triple-rinsed, it should be left open in back of a hood overnight. Solvent rinses should be disposed of as hazardous waste.
Notes	Any deviation from this SOP requires approval from PI.			



**13. Documentation of Training (signature of all users is required)**

- Prior to conducting any work with pyrophoric materials, 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 his/her laboratory personnel with a copy of this SOP and a copy of the pyrophoric material MSDS provided by the manufacturer.

I have read and understand the content of this SOP:

Name	Signature	Identifier	Date
