**Standard Operating Procedure**

**Quenching and Disposal of Water Reactive Materials**

**Examples: lithium, sodium, cesium, lithium aluminum hydride, calcium hydride, potassium hydride, Grignard reagents**

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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 [x] Hazardous Chemical [ ] Equipment

1. **Purpose**

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

Also, this SOP covers any material synthesized using water-reactive chemicals.

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

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

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

1. **Potential Hazards**

When quenching water-reactive materials, the hazards of the mixture, the WR chemical and the solvent, must be considered together and procedures for safe quenching must reflect the hazard properties of both solvent and solute.

As defined by the Globally Harmonized System of Classification and Labeling of Chemicals (GHS), water-reactive materials are defined as “substances and mixtures which, in contact with water, emit flammable gases” and are designated by one or more of the following H codes:

H260 In contact with water releases flammable gases which may ignite spontaneously

H261 In contact with water releases flammable gases

It is the Principal Investigator’s responsibility to ensure activity-specific laboratory procedures and/or processes are taken into account when using this Hazardous Operation Class SOP.

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 WR chemicals:

* Use a clean fume hood, preferably with the sliding sash windows.
* 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.
* Remove any flammables (squirt bottles, solvents, oil bath) and combustibles (KimWipes, paper towel) from 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:**

Water-reactive chemicals can be handled and stored safely as long as all exposure to moisture or other incompatible chemicals is minimized.

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

Any unused or unwanted water-reactive materials must be destroyed by following the Quenching of Water Reactive Chemicals SOP. If you have large quantities of unreacted water-reactive reagent material contact EH&S for guidance on disposal options.

• Waste materials generated must be treated as a hazardous waste.

• The empty container must be rinsed three times with a COMPATIBLE solvent; leave it open in the back of the hood overnight.

• The empty container, solvent rinses and water rinse must be disposed of as hazardous waste.

• Do not mix with incompatible waste streams.

• Decontamination of containers in order to use them for other purposes is not permitted.

**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.

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>

**General Guidelines Water Reactive 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.

**Water Reactive 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. Use only dry chemical fire extinguishers (classes ABC or D).

• 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 WR 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.

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.

**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)](http://ehs.oregonstate.edu/sites/ehs.oregonstate.edu/files/pdf/si/waste_hazardous_disposal_si.pdf%29).
* Double-bag dry waste using transparent bags
* 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]

[UCSD - Working with Reactive Metals Video](https://www.youtube.com/watch?v=ozmddj0fIpk&feature=youtu.be)

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

Quenching of inorganic hydrides such as LiAlH4, NaBH4, CaH2, NaH, LiH, and metal powders (Li, Na) left after reactions.

Up to 10g of material or up to 100 mL of a commercially acquired solution (3 M or lower).

Larger quantities of WR chemicals can be disposed of as hazardous waste.

Engineering controls

Conduct in a clean and properly operating fume hood with the sash as low as possible.

Personel Protection Equipment (PPE)

Eye Protection: Wear tight-fitting safety goggles or safety glasses with side shields.

Face Protection: Face shields are to be used when there is no protection from the hood sash or when the hood sash is in open position.

Hand Protection: Confirm compatibility of glove material with chemical being used. Gloves must be inspected prior to use. Wash and dry hands after use.

General guidance : For indirect contact (closed-system procedures such as transfers via syringe or cannula) AND direct contact (open system procedures such as spill handling, wiping of residual pyrophorics) with pyrophoric material - a combination of fire resistant (FR) liners, covered with a pair of chemical-resistant disposable gloves (e.g. nitrile gloves or those specified in the specific SDS), must be worn AT ALL TIMES. The following products are Approved FR Liners: Ansell Kevlar® Goldknit® Lightweight 70-200 and Hanz Extremity Wear Nomex® Utility Liners.

Clothing: Wear Nomex IIIA (NFPA 2112) lab coat; full-length pants or equivalent; and close-toed and close-heeled shoes.

**Procedure Steps and Special Precautions for this Procedure**

**1. Quenching of inorganic hydrides such as LiAlH4, NaBH4, CaH2, NaH, LiH, and metal powders (Li, Na) left after reactions.**

General Procedure:

Quenching procedures usually involve the reaction of the WR material with a reagent that has a reactive hydroxyl group.

Any reaction or suspension containing these reagents MUST be quenched carefully!

Addition of materials must be done SLOWLY and ensure adequate stirring/mixing.

Whenever quenching be sure that it is not done in a sealed vessel as pressure will build up.

If you’re quenching in a RB flask or any floating vessel in a bath, clamp it.

Typically, a suspension of less than 20 wt% of WR in an inert solvent (such as hexane or toluene) is created, followed by the slow addition of isopropanol, under adequate stirring until no more bubbling is observed. To avoid vigorous bubbling and any signs of overheating during the quenching process, keep the solution cool by controlling the feed rate of the alcohol. If the solution begins to warm up, stop and allow it cool down before continuing the addition of the quenching agent.

For NaH, LiH, CaH2, NaBH4, small amounts LiAlH4: Start with the SLOW addition of isopropanol or ethanol, under adequate stirring until no more bubbling is observed.

Repeat with methanol, and then repeat with water.

Be Very Careful with the addition of WATER! Even after methanol has been added, the water-reactive agent can still react violently with water, especially is there hasn't been sufficient mechanical stirring of the solution. So add in small aliquots.

Stir for an additional 2 hours before disposing of the aqueous organic waste.

If you are filtering out a solid residue containing a water-reactive chemical, be sure there is no active residue on the filter paper before disposing of it in the solid waste. Wash the solid residue with some methanol to quench any remaining WR material.

Alternative Procedure:

You can quench LiAlH4 reactions containing x g lithium aluminum hydride via the Fieser-Fieser workup:

1. Either dilute with ether and cool to 0°C or cool to 0°C and slowly add EtOAc

2. Slowly add x mL water

3. Slowly add x mL 15 % aqueous sodium hydroxide

4. Add 3x mL water

5. Warm to RT and stir 15 min

6. Add some anhydrous magnesium sulfate

7. Stir 15 min and filter to remove salts

**2. Quenching of Na or K.**

Minimal quantities will be left after reactions carried out at scales listed in your laboratory “WR” Class SOP.

Larger quantities of WR liquid chemicals can be disposed of as hazardous waste.

Engineering Controls

Conduct in a clean and properly operating fume hood with the sash as low as possible.

Personel Protection Equipment (PPE)

Eye Protection: Wear tight-fitting safety goggles or safety glasses with side shields.

Face Protection: Face shields are to be used when there is no protection from the hood sash or when the hood sash is in open position.

Hand Protection: Confirm compatibility of glove material with chemical being used. Gloves must be inspected prior to use. Wash and dry hands after use. General guidance : For indirect contact (closed-system procedures such as transfers via syringe or cannula) AND direct contact (open system procedures such as spill handling, wiping of residual pyrophorics) with pyrophoric material - a combination of fire-resistant (FR) liners, covered with a pair of chemical-resistant disposable gloves (e.g. nitrile gloves or those specified in the specific SDS), must be worn AT ALL TIMES. The following products are Approved FR Liners: Ansell Kevlar® Goldknit® Lightweight 70-200 and Hanz Extremity Wear Nomex® Utility Liners.

Clothing: Wear Nomex IIIA (NFPA 2112) lab coat; full-length pants or equivalent; and close-toed and close-heeled shoes.

Procedure Steps and Special Precautions for this Procedure

General Procedure:

Sodium/Potassium: Quenching procedures usually involve the reaction of the WR material with a reagent that has a reactive hydroxyl group.

Any reaction or suspension containing these reagents MUST be quenched carefully!

Addition of materials must be done SLOWLY and ensure adequate stirring/mixing.

Whenever quenching be sure that it is not done in a sealed vessel as pressure will build up.

If you’re quenching in a RB flask or any floating vessel in a bath, clamp it.

A typical procedure involves suspending the Na/K alloy in DRY toluene to less than 20 wt%. Slowly add the toluene to the stirring Na/K. To avoid vigorous bubbling and any signs of overheating during the quenching process, keep the solution cool by controlling the feed rate of the alcohol. If the solution begins to warm up, stop and let it cool down before continuing the addition of the quenching agent.

Once all toluene has been added, allow to stir for 5-10 minutes then add DRY ethyl acetate SLOWLY to the stirring solution. The ethyl acetate will quench the potassium.

Once addition is complete, let the solution stir for one hour.

At this point, only the sodium is remaining in the flask and can be quenched as described above with isopropanol, methanol, and then water.

Be Very Careful with the addition of WATER! Even after methanol has been added, the alloy can still react violently with water, especially is there hasn't been sufficient mechanical stirring of the solution. So add in small aliquots.

Let stir for a couple of hours before disposing of the aqueous organic 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 of Water Reactove Materials* 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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