Quenching of Water-Reactive (WR) Materials

H260  H261

Examples: lithium, sodium, cesium, lithium aluminum hydride, calcium hydride, potassium hydride

1. Purpose

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

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

Also, this SOP covers any materials 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 of your laboratory or the campus Chemical Hygiene Officer at ucbcho@berkeley.edu.

2. Physical & Chemical Properties

For physical and chemical properties on water-reactive materials, please refer to your laboratory “WR and SRA” or “PYR, WR and SRA” Class SOP and to specific SDSs of chemicals in use (See Section 11 – SDS Location).
3. Potential Hazards/Toxicity

Water reactive substances are dangerous when wet because they can undergo a chemical reaction with water. This reaction may release a gas that can be flammable and/or toxic. In addition, the heat generated when water contacts such materials is often enough to spontaneously combust or explode. When quenching WR materials, the hazards of the mixture, the WR chemical and the solvent, should be considered together and procedures for safe quenching should reflect the hazard properties of both solvent and solute.

As defined by the Globally Harmonized System of Classification and Labeling of Chemicals (GHS), WR 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

4. Engineering Controls

The following is the set of engineering controls that are required when quenching WR chemicals:

- Use a clean fume hood, preferably with the sliding sash windows or a glove box.
- 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 a vertical sliding sash, keep the sash as low as possible.
- Remove any flammables (squirt bottles, solvents, oil bath) 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**

- 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.
- 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**

- Flame-resistant lab coat (Nomex IIIA, NFPA 2112) should be worn when working with water-reactive materials.
- Gloves are required when handling hazardous materials. Refer to the specific chemical SDS for information on glove selection.
- 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. 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
WR chemicals can be handled and stored safely as long as all exposure to moisture or other incompatible chemicals is minimized. Never leave a container with a residue of a WR material open to the atmosphere.

Working Alone
The UC Berkeley Office of Environmental Health and Safety specifies not to work with WR chemicals alone or during off hours, when there are few people around to help.

Handling and Storage of Water-Reactive Materials
Liquids may be safely transferred by employing techniques discussed in your laboratory “WR and SRA” or “PYR, WR and SRA” Class SOP. Lab-specific information on handling and storage may be included in Section 12 - Protocol/Procedure section.

8. Chemical Spill and Managing Any Subsequent Fire

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.
Hazardous Operation SOP

- 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.

Water-Reactive 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).
- 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.

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. Safety Data Sheet (SDS) Location
SDS can be accessed online at http://ucmsds.com

<table>
<thead>
<tr>
<th>Preparation</th>
<th>List any other particular preparation requirements needed for this procedure (e.g., location of spill kit or keep water or ignition sources away from procedure area)</th>
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<tbody>
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<td>• Know the location of the nearest fire extinguisher, eyewash, and safety shower before beginning work.</td>
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<td>• Have a small beaker or can of Met-L-X or DRY sand in the work area, within arm's reach.</td>
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<td>• Solvents must be dry.</td>
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<td>• Glassware must be dry before using. Either “flame” dry or dry in an oven at a minimum temperature of 100 °C for about 2 hours.</td>
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<td>• Remove all other flammable materials from the hood to reduce the hazard in case of a fire.</td>
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<td>• Make adjacent lab workers aware that you will be working with WR chemicals.</td>
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**NOTE:** As an alternative to quenching, remember that water-reactive chemicals can be disposed of as hazardous waste. If this option is chosen:

- Carefully package and label the waste with current HWP labels.
- Request waste pick up from EH&S

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Procedure Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Quenching of inorganic hydride (LiAlH₄, NaBH₄, NaH, LiH, CaH₂)</td>
</tr>
<tr>
<td>2</td>
<td>Quenching of Na or K</td>
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<tr>
<td>3</td>
<td>Quenching of Na/K alloy</td>
</tr>
<tr>
<td>Procedure/Use</td>
<td>Scale</td>
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</table>
| 1. Quenching of inorganic hydrides such as LiAlH₄, NaBH₄, CaH₂, NaH, LiH, and metal powders (Li, Na) left after reactions |       | Conduct in a clean fume hood with the sash closed. | **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.  
**Hand Protection:** 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.  
**Hand Protection for indirect contact with WR material** (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).  
**Hand Protection for direct contact with WR material** (open system procedures such as spill handling, wiping of residual WR chemical): Provide for adequate employee hand protection by evaluating any additional risks via a hazard assessment and provide the appropriate hand protection. Consider the use of fire resistant (FR) gloves or liners.  
**Clothing:** Wear Nomex IIIA (NFPA 2112) lab coat; cotton based | General considerations:  
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 should 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**.  
**Procedure:**  
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 if 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. It is recommended to wash the solid residue with some methanol to quench any remaining WR material.*
### Procedure/Use

<table>
<thead>
<tr>
<th>Procedure/Use</th>
<th>Scale</th>
<th>Engineering Controls/Equipment</th>
<th>PPE (eye, face, gloves, clothing)</th>
<th>Procedure Steps and Special Precautions for this Procedure</th>
</tr>
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<tbody>
<tr>
<td>2. Quenching of Na or K</td>
<td>This procedure is to be used for up to 10 g of material. Quenching of WR in amounts greater than 10 g at a time is not permitted. Small amounts will be left after reactions carried out at scales listed in your laboratory specific “WR and SRA” or “PYR, WR and SRA” Class SOP. As an alternative to quenching, remember that any quantities of WR can be disposed of as hazardous waste.</td>
<td>Conduct in a clean fume hood with the sash closed.</td>
<td><strong>Eye Protection:</strong> Wear tight-fitting safety goggles or safety glasses with side shields. <strong>Face Protection:</strong> Face shields are to be used when there is no protection from the hood sash or when the hood sash is in open position. <strong>Hand Protection:</strong> 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. <strong>Hand Protection for indirect contact with WR material</strong> (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). <strong>Hand Protection for direct contact with WR material</strong> (open system procedures such as spill handling, wiping of residual WR chemical): Provide for adequate employee hand protection by evaluating any additional risks via a hazard assessment and provide the appropriate hand protection. Consider the use of fire resistant (FR) gloves or liners. <strong>Clothing:</strong> Wear Nomex IIIA (NFPA 2112) lab coat; cotton based clothing; full length pants or equivalent; and close-toed and close-heeled shoes.</td>
<td>Sodium: A ceramic flower pot (see picture below) is half filled with dry fine-grained sand. Sodium residues are placed on the dry sand. On top of the sodium a further layer of dry sand is placed. Finally the flower pot is placed in a large porcelain tray or dish. Water is added into the dish to raise the level of water to roughly 3cm. After a few minutes water is drawn by capillary network into the sand. It is important that during the reaction, there is always enough water in the tray. After 1-2 days all sodium is converted into NaOH and hydrogen. The sand is washed with water to remove NaOH and, after drying, the sand can be used again. <strong>Potassium:</strong> WARNING! Potassium is often encrusted with KO₂. Explosions are observed when this material is cut with a knife or destroyed by alcohol. Therefore it is recommended to place potassium encrusted with KO₂ very gently on sand and to cover it very gently with sand. Follow the same procedure as for Na.</td>
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*The photo shows the flower pot placed in a porcelain beaker. The flower pot is filled with dry sand and sodium before the water is added into the beaker.*
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| 3. Quenching of Na/K alloy | This procedure is to be used for up to 10 g of material. Quenching of WR in amounts greater than 10 g at a time is not permitted. Small amounts will be left after reactions carried out at scales listed in your laboratory specific “WR and SRA” or “PYR, WR and SRA” Class SOP. As an alternative to quenching, remember that any quantities of WR can be disposed of as hazardous waste. | Conduct in a clean fume hood with the sash closed. | **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.  
**Hand Protection:** 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.  
Hand Protection for indirect contact with WR material (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).  
Hand Protection for direct contact with WR material (open system procedures such as spill handling, wiping of residual WR chemical): Provide for adequate employee hand protection by evaluating any additional risks via a hazard assessment and provide the appropriate hand protection. Consider the use of fire resistant (FR) gloves or liners.  
**Clothing:** Wear Nomex IIIA (NFPA 2112) lab coat; cotton based | **General considerations:**  
Quenching of Na/K requires extreme caution as it is highly reactive.  
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 should 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.  
**Procedure:**  
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 toluene. 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. |
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<th>clothing; full length pants or equivalent; and close-toed and close-heeled shoes.</th>
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<tr>
<td>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 if 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.</td>
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13. Documentation of Training (signature of all users is required)

- Prior to conducting any work with water-reactive 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 water-reactive material MSDS provided by the manufacturer.

I have read and understand the content of this SOP:

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<tr>
<th>Name</th>
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