



Quenching of Water Reactive Materials

H260 H261



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

Areas with blue text indicate that information must be provided or modified by researcher prior to the SOP approval.

This SOP is not a substitute for hands-on training.

Print a copy and insert into your laboratory SOP binder.

| | |
|---|---|
| Department: | Chemistry |
| Date SOP was written: | 11/22/16 |
| Date SOP was approved by PI/lab supervisor: | |
| Principal Investigator: | Name: Richmond Sarpong |
| | Signature: _____ |
| Internal Lab Safety Coordinator or Lab Manager: | Name: Melissa Hardy/Justin Jurczyk |
| | Lab Phone: 406-696-1225/412-728-1952 |
| | Office Phone: 510-642-6312 |
| Emergency Contact: | Name: Melissa Hardy/Justin Jurczyk |
| | Lab Phone: 406-696-1225/412-728-1952 |
| Location(s) covered by this SOP: | 831,832,834,836,837,838,839,842,844,847,849 |

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 Materials**” (WR) are covered by this hazardous operating SOP.

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 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 to specific Safety Data Sheets (SDS) of chemicals in use (See Section 11 – SDS Location).

3. Potential Hazards/Toxicity

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)

4. 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.

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 must be worn.

Skin Protection

- A. Flame-resistant lab coat (Nomex IIIA, NFPA 2112) must be worn when working with WR.
- B. 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.



C. Long pants, closed-toe/closed-heel shoes, covered legs, and ankles.

6. First Aid Procedures and Medical Emergencies

In the event of an injury, notify your supervisor immediately and EH&S within 8 hours.



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.

If inhaled

Move into fresh air.

Needle stick/puncture exposure

Wash the affected area with antiseptic soap and warm water for 15 minutes.

7. Special Handling and Storage Requirements

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.

Precautions for Safe Handling and Conditions for Safe Storage

Liquids may be safely transferred by employing techniques discussed in your laboratory "WR" Class SOP. Lab-specific information on handling and storage may be included in Section 12 - Protocol/Procedure section.

Disposal



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- 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.

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.
- 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. 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.

9. Cleaning and Decontamination

Lab-specific information on decontamination may be included in Section 12 - Protocol/Procedure.

- Wearing proper PPE, laboratory work surfaces must be cleaned at the conclusion of each procedure and at the end of each work day.



- Decontaminate 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

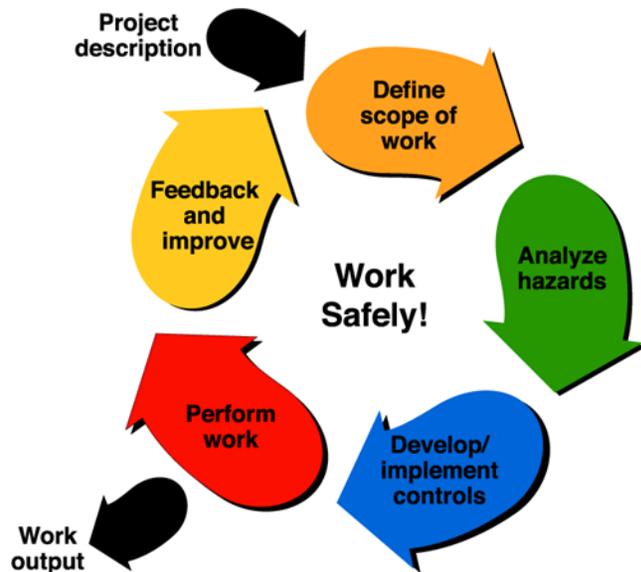
- Dispose of regularly generated chemical waste within 6 months.
- Call EH&S with questions.

11. Safety Data Sheet (SDS) Location

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



-Take Ownership of Your Safety-



Before starting any work, ask yourself:

- 1- **What will I be doing?**
- 2- **Do I know what the hazards are?**
- 3- **Do I have everything I need to do the job safely?**
- 4- **Am I doing the job safely?**
- 5- **What can we do better?**



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12. Protocol/Procedure - Quenching of Water Reactive Materials

Section 12 must be customized to your specific needs. Delete any procedure that does not apply to your laboratory.

| Procedure/Use | Scale | Engineering Controls/Equipment | PPE (eye, face, gloves, clothing) | Procedure Steps and Special Precautions for this Procedure |
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| <p>1. Quenching of inorganic hydrides such as LiAlH_4, NaBH_4, CaH_2, NaH, LiH, and metal powders (Li, Na) left after reactions.]</p> | <p>Up to 10g of material or up to 100 mL of a commercially acquired solution (3 M or lower).</p> <p>Minimal quantities will be left after reactions carried out at scales listed in your laboratory specific "WR" Class SOP.</p> <p>Larger quantities of WR chemicals can be disposed of as hazardous waste.</p> <p>Obtain PI approval if</p> | <p>Conduct in a clean and properly operating fume hood with the sash as low as possible.</p> | <p>Eye Protection: Wear tight-fitting safety goggles or safety glasses with side shields.</p> <p>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.</p> <p>Hand Protection: Confirm compatibility of glove material with chemical being used. Gloves must be inspected prior to use. Wash and dry hands after use.</p> <p>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</p> | <p><u>General Procedure:</u> Quenching procedures usually involve the reaction of the WR material with a reagent that has a reactive hydroxyl group.</p> <p>Any reaction or suspension containing these reagents MUST be quenched carefully!</p> <p>Addition of materials must be done SLOWLY and ensure 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.</p> <p>If you're quenching in a RB flask or any floating vessel in a bath, clamp it.</p> <p>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.</p> <p>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.</p> <p><i>For NaH, LiH, CaH₂, NaBH₄, small amounts LiAlH₄:</i> Start with the SLOW addition of isopropanol or ethanol, under adequate stirring until no more bubbling is observed.</p> <p>Repeat with methanol, and then repeat with water.</p> |



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| | <p>higher scale is necessary.</p> | | <p>Extremity Wear Nomex® Utility Liners.</p> <p>Clothing: Wear Nomex IIIA (NFPA 2112) lab coat; full length pants or equivalent; and close-toed and close-heeled shoes.</p> | <p>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.</p> <p>Stir for an additional 2 hours before disposing of the aqueous organic waste.</p> <p><i>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.</i></p> <p><u>Alternative Procedure:</u></p> <p>You can quench LiAlH_4 reactions containing x g lithium aluminum hydride via the Fieser-Fieser workup:</p> <ol style="list-style-type: none"> 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 |
| <p>Notes</p> | <p>Any deviation from this SOP requires approval from PI.</p> | | | |



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| Procedure/Use | Scale | Engineering Controls/ Equipment | PPE (eye, face, gloves, clothing) | Procedure Steps and Special Precautions for this Procedure |
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| <p>2. Quenching of Na or K. </p> | <p>Minimal quantities will be left after reactions carried out at scales listed in your laboratory "WR" Class SOP.</p> <p>Larger quantities of WR liquid chemicals can be disposed of as hazardous waste.</p> <p>Obtain PI approval if higher scale is necessary.</p> | <p>Conduct in a clean and properly operating fume hood with the sash as low as possible.</p> | <p>Eye Protection: Wear tight-fitting safety goggles or safety glasses with side shields.</p> <p>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.</p> <p>Hand Protection: Confirm compatibility of glove material with chemical being used. Gloves must be inspected prior to use. Wash and dry hands after use.</p> <p>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</p> | <p><u>General Procedure:</u></p> <p>Sodium/Potassium: Quenching procedures usually involve the reaction of the WR material with a reagent that has a reactive hydroxyl group.</p> <p>Any reaction or suspension containing these reagents MUST be quenched carefully!</p> <p>Addition of materials must be done SLOWLY and ensure 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.</p> <p>If you're quenching in a RB flask or any floating vessel in a bath, clamp it.</p> <p>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.</p> <p>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.</p> <p>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.</p> <p>Once addition is complete, let the solution stir for one hour.</p> |



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| | | | <p>Extremity Wear Nomex® Utility Liners.</p> <p>Clothing: Wear Nomex IIIA (NFPA 2112) lab coat; full length pants or equivalent; and close-toed and close-heeled shoes.</p> | <p>At this point, only the sodium is remaining in the flask and can be quenched as described above with isopropanol, methanol, and then water.</p> <p>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.</p> <p>Let stir for a couple of hours before disposing of the aqueous organic waste. </p> |
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| Procedure/Use | Scale | Engineering Controls/ Equipment | PPE (eye, face, gloves, clothing) | Procedure Steps and Special Precautions for this Procedure |
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| <p>3. Quenching of Na/K alloy.</p> | <p>Up to 10g of material or up to 100 mL of a commercially acquired solution (3 M or lower).</p> <p>Minimal quantities will be left after reactions carried out at scales listed in your laboratory specific "WR" Class SOP.</p> <p>Larger quantities of WR chemicals can be disposed of as hazardous waste.</p> <p>Obtain PI approval if higher scale is necessary.</p> | <p>Conduct in a clean and properly operating fume hood with the sash as low as possible.</p> | <p>Eye Protection: Wear tight-fitting safety goggles or safety glasses with side shields.</p> <p>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.</p> <p>Hand Protection: Confirm compatibility of glove material with chemical being used. Gloves must be inspected prior to use. Wash and dry hands after use.</p> <p>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.</p> | <p><u>General Procedure:</u> Quenching of Na/K requires extreme caution as it is highly reactive.</p> <p>Quenching procedures usually involve the reaction of the WR material with a reagent that has a reactive hydroxyl group.</p> <p>Any reaction or suspension containing these reagents MUST be quenched carefully!</p> <p>Addition of materials must be done SLOWLY and ensure 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.</p> <p>If you're quenching in a RB flask or any floating vessel in a bath, clamp it.</p> <p>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.</p> <p>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.</p> <p>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.</p> <p>Once addition is complete, let the solution stir for one hour.</p> |



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| | | | <p>Clothing: Wear Nomex IIIA (NFPA 2112) lab coat; full length pants or equivalent; and close-toed and close-heeled shoes.</p> | <p>At this point, only the sodium is remaining in the flask and can be quenched as described above with isopropanol, methanol, and then water.</p> <p>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.</p> <p>Let stir for a couple of hours before disposing of the aqueous organic waste.</p> |
| <p>Notes</p> | <p>Any deviation from this SOP requires approval from PI.</p> | | | |



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| Procedure/Use | Scale | Engineering Controls/Equipment | PPE (eye, face, gloves, clothing) | Procedure Steps and Special Precautions for this Procedure |
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| 4. Quenching of liquid WR. | <p>Up to 100 mL of WR in carrier solvent. See note for appropriate glassware size selection.</p> <p>Minimal quantities will be left after reactions carried out at scales listed in your laboratory "WR" Class SOP.</p> <p>As an alternative to quenching, remember that any quantities of WR can be disposed of as hazardous waste.</p> <p>Obtain PI approval if</p> | <p>Conduct in a clean and properly operating fume hood with the sash as low as possible.</p> | <p>Eye Protection: Wear tight-fitting safety goggles or safety glasses with side shields.</p> <p>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.</p> <p>Hand Protection: Confirm compatibility of glove material with chemical being used. Gloves must be inspected prior to use. Wash and dry hands after use.</p> <p>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</p> | <p>Note on Glassware Size: If quenching 100mL of WR solution, the WR 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.</p> <p>- Quenching in a FUME HOOD -</p> <p><u>General considerations:</u></p> <p>Quenching procedures usually involve the reaction of the WR, 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 MUST be quenched carefully! All transfers must be done under inert atmosphere using the syringe or cannula techniques.</p> <p>Addition of material must be done SLOWLY, 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 or other appropriate vent.</p> <p>If you're quenching in a RB 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 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> |



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| | <p>higher scale is necessary.</p> | | <p>Extremity Wear Nomex® Utility Liners.</p> <p>Clothing: Wear Nomex IIIA (NFPA 2112) lab coat; full length pants or equivalent; and close-toed and close-heeled shoes.</p> | <p>Before proceeding with the quenching of the WR solution, proceed with the cleaning of the empty bottle.</p> <p>Rinse the empty container 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, if any, and leave it 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 WR solution by adding isopropanol slowly 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>Be Very Careful with the addition of water! Even after methanol has been added, the residue 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. Syringes (plastic or glass) are ideal tools to make these additions.</p> <p>Once the addition of water is complete, let the solution temperature come back to room temperature while stirring.</p> |
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| | | | | <p>Stir for an additional 2 hours before disposing of the aqueous organic waste.</p> <p>- If quenching in a FUME HOOD using dry ice -</p> <p>Place dry ice in a container and add a stir bar. WR 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.</p> <p>Stir for an additional 2 hours before disposing of the aqueous organic waste.</p> <p>- If quenching in a GLOVE BOX -</p> <p><u>General considerations:</u></p> <p>Any reaction or suspension containing these reagents MUST be quenched carefully! Addition of solvents must be done SLOWLY with adequate stirring/mixing.</p> <p>If the glove box is equipped with a fridge, DO NOT 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 WR 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.</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> |
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| | | | | <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>Continue the quenching procedure in a clean fume hood with sash closed.</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. Syringes (plastic or glass) are ideal tools to make these additions.</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 must 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>Rinse the empty container three times with an inert dry COMPATIBLE solvent such as toluene or hexane.</p> <p>After the empty container is triple-rinsed, leave it open in back of a hood overnight.</p> <p>Solvent rinses must be disposed of as hazardous waste.]</p> |
| <p>Notes</p> | <p>Any deviation from this SOP requires approval from PI.</p> | | | |

