**THIS IS A TEMPLATE/BASIC STARTING POINT. CUSTOMIZE THIS TEMPLATE WITH INFORMATION PERTINENT TO YOUR SETUP AND THE PROCEDURE YOU WILL BE USING/YOUR GROUP’S PERSONAL USE.**

STANDARD OPERATING PROCEDURE

Use this form to document the Health & Safety information associated with the procedure.

|  |  |
| --- | --- |
| **Procedure Title:** | Organolithium reagents |
|  |  |
| **Dept:** |  |  | **Bldg/Rm:** |  |  | **Supervisor:** |  |

**Procedure Overview:** (brief description of the project)

Organolithium reagents are pyrophoric organometallic compounds with the general formula RLi where R can be alkyls, aryls, and vinyls. Most organolithium reagents are highly pyrophoric substances and may spontaneously ignite in contact with air. These compounds have applications in organic synthesis since they are sufficiently strong bases to deprotonate many carbon acids, including benzene. In regard to molecular aggregation state, some of these compounds exist in clusters. These compounds can be used for the deprotonation of amines and activated C—H compounds.

**Health and safety information for materials used:** (briefly describe the hazards associated with the materials and/or equipment **OR** document your hazard assessment in Section II)

Always read and understand the safety data sheet (SDS) for a chemical before use or storage.

Organolithium reagents (such as methyllithium, *n*-butyllithium, *sec*-butyllithium, and *t*-butyllithium) are highly moisture sensitive and react violently with air and/or water. Upon reaction with water, these chemicals can ignite and/or liberate highly toxic gases; as such they are generally sold as solutions in an organic solvent. They must be handled under an inert atmosphere to minimize exposure to both air and moisture. They can cause severe burns and are highly corrosive. In addition to the high flammability, they can be harmful to the liver, kidneys, and nervous system.

**Hazard Control Measures:**

(Lab coat, eye and hand protection, and fully enclosed shoes must be selected as required by Section D of the ISU Laboratory Safety Manual.)

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| [ ]  | Latex gloves | [ ]  | Insulated gloves | [x]  | Face shield | [ ]  | Respirator |
| [x]  | Nitrile gloves | [ ]  | Safety glasses | [ ]  | Lab coat | [x]  | Fume hood |
| [ ]  | Neoprene gloves | [ ]  | Vented goggles | [ ]  | Apron | [ ]  | Biosafety cabinet |
| [ ]  | Vinyl gloves | [x]  | Splash goggles | [ ]  | Dust mask | [x]  | Glove box |
| [x]  | Fully enclosed shoes | [x]  | Flame resistant lab coat |

***Other Control Measures:***

Organolithium reagents should only be used in the fume hood and if applicable, an inert atmosphere glovebox.

**Special Handling Procedures and Storage Requirements:**

* Ensure the work area is clean and uncluttered.
* Do not use anyorganolithium reagent while working alone.
* Don appropriate personal protective equipment
* **For transfer of small amounts (<10-15 mL), you can use a syringe/needle:**
	+ All glassware and solvents should be dried prior to use with any organolithium reagent. Flame or oven-dried glassware which has been cooled under an inert atmosphere just before use is ideal.
	+ Upon charging a dry flask with dry reagents/solvents and reaching the desired temperature, the appropriate organolithium reagent can be added. Some organolithium reagents are stored at room temperature (i.e. methyllithium, n-butyllithium) and can be used as such, but some (e.g. t-butyllithium) are stored under refrigeration and must be warmed to room temperature prior to use.
	+ The organolithium reagent is contained in a Sure-Seal bottle, the septum of which can be pierced by a clean, dry needle fitted to an inert gas supply, used to keep the reagent under a blanket of inert gas during the entire process.
	+ The needle and syringe must be purged before use: A clean, dry reagent needle with the appropriate size syringe is used to pierce the septum and inert gas is drawn in to the syringe, then the needle is removed and the gas inside is expelled to the atmosphere. This process is repeated several times, piercing the same hole in the septum, to remove most air from the needle and syringe.
	+ After purging the needle appropriately, the tip is submerged below the level of reagent and the required amount is drawn up into the syringe. Be careful to hold the end of the plunger as well as the joint where the needle and syringe meet. If either of these comes apart the reagent will come out and may ignite on exposure to air. **DO NOT FILL SYRINGE MORE THAN 60% FULL**
	+ Once the desired amount of organolithium reagent has been drawn into the syringe, the tip of the needle is pulled above the level of reagent, and the plunger is pulled out slightly to draw a blanket of inert gas into the needle. The needle is then removed from the reagent bottle and quickly pierced into the septum of the reaction flask.
	+ Slowly dispense the reagent into the reaction flask, being careful to hold the needle and syringe together, as applying force to dispense the reagent can cause them to separate and expose the reagent to moisture.
	+ After the reagent has been dispensed, the needle can be rinsed with hexanes multiple times, then water. Clean the needle immediately after use to prevent clogging. Replace the cap and wrap the outside with parafilm to further ensure safe storage. If using a disposable syringe/needle, dispose of it appropriately into the sharps waste container.
* **For transfer of larger amounts (>10-15 mL), use a double-tipped needle process**, found in the [Sigma-Aldrich Technical Bulletin AL-134](https://www.sigmaaldrich.com/deepweb/assets/sigmaaldrich/marketing/global/documents/685/583/al_techbull_al134.pdf) “Handling Air-Sensitive Reagents”

**Waste Disposal Procedures:**

Fully quench all reagent before disposal. To quench excess organolithium, dilute significantly with an unreactive, dry, solvent. Slowly add isopropanol. The flask can be placed in a cooling bath to help control the temperature of the quenching process. Upon completion of quenching, add methanol, then water to ensure a complete quenching. Dispose of the quenched materials into the appropriate waste container.

**First Aid Procedures:**

**General advice**

Consult a physician. Show this safety data sheet to the doctor in attendance. Move out of dangerous area.

**If inhaled**

If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

**In case of skin contact**

Take off contaminated clothing and shoes immediately. Wash off with soap and plenty of water for 15 minutes. Consult a physician.

**In case of eye contact**

Rinse thoroughly with plenty of water for at least 15 minutes and seek medical attention. Continue rinsing eyes during transport to hospital.

**If swallowed**

Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

**All accidents and injuries occurring at work or in the course of employment must be reported to the employee's supervisor as soon as possible (even if no medical attention is required). Report all incidents and exposures here:** [**https://www.ehs.iastate.edu/services/occupational/accidents-injuries**](https://www.ehs.iastate.edu/services/occupational/accidents-injuries)

**Spill/Release Containment and Clean Up Procedures:**

Contain spillage and prevent further leakage or spillage if it is safe to do so. Avoid breathing vapors, mist, or gas. Ensure adequate ventilation. Removal all sources of ignition. Evacuate personnel to safe areas.

In case of a fire, use Metal-X, soda ash, or sand for small fires. For large fires, use an ABC fire

extinguisher. Do not use water or a CO2 extinguisher on the fire.

**Using Substances Requiring Special Procedures?** No [ ]  Yes [x]

(If Yes; identify authorized personnel, designate a use area and specify specialized safety precautions here. Refer to Section B in the ISU Laboratory Safety Manual for details.)

Use of organolithium reagents requires site specific training and the approval from the professor in charge.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Written By:** |  |  | **Date:** |  |
|  |  |  |  |  |
| **Approved By:** |  |  | **Date:** |  |

 (PI or Lab Supervisor)

**HAZARD ASSESSMENT**

Use the hierarchy of controls to document the hazards and the

corresponding control measure(s) involved in each step of the procedure.

Consider *elimination or substitution* of hazards, if possible.

*Engineering Control(s):* items used to isolate the hazard from the user (i.e. fume hood, biosafety cabinet).

*Administrative Control(s):* policies/programs to limit the exposure to the hazard (i.e. authorizations, designated areas, time restrictions, training).

*Required PPE*: indicate PPE including specific material requirements if applicable (i.e. flame resistant lab coat, type of respirator or cartridge).

|  |  |  |  |
| --- | --- | --- | --- |
| **Hazard** | **Engineering Control(s)** | **Administrative Control(s)** | **Required PPE**  |
|  Pyrophoric reagents – flammable and extremely reactive with air and water |  Fume hood with an inert gas line. Glove box |  Site specific training |  Flame resistant lab coat, goggles, face shield, gloves, fully enclosed shoes |
| **INSERT SPECIFIC HAZARDS/CONTROLS HERE** |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

1. **Training Record**

Use the following table to record the training associated with this Standard Operating Procedure.

|  |  |  |
| --- | --- | --- |
| **Print Name** | **Signature** | **Date** |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |

**Note: Attach to or file with written materials and methods**