**Pyrophoric Materials**

**STANDARD OPERATING PROCEDURE (SOP)**

**Type of SOP:** ☐ Process ☐ Hazardous Chemical ☒ Hazardous Class

**All personnel subject to these SOP requirements must review a completed SOP and sign the associated training record. Completed SOPs must be kept in the laboratory’s safety binder or be otherwise readily accessible to laboratory personnel. Electronic access is acceptable. SOPs must be reviewed, and revised where needed, as described in the** [**SJSU Chemical Hygiene Plan**](http://www.sjsu.edu/fdo/departments/ehs/lab/Chemical%20Hygiene%20Plan_20191017-final.pdf)**. Note that not all hazardous chemicals are appropriately addressed in a single Hazard Class SOP, and some chemicals are subject to several Hazard Class SOPs.**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Date SOP Written:  | **4/2/2020** |  | Approval Date: | **4/3/2020** |
| SOP Prepared by: | **Prof. Jane Doe** |
| SOP Reviewed and Approved by (name/signature): | **Prof. Jane Doe** |
| Department:  | **Chemistry** |
| Principal Investigator/Laboratory Supervisor:  | **Prof. Jane Doe** | Phone:  | **408-924-XXXX** |
| Emergency Contact(s):  | **Prof. Jane Doe** | Phone:  | **(personal contact number/cell number)** |
|  | **Randy Kirchner** |  | **408-924-5004** |
|  | **Prof. John Smith** |  | **408-924-XXXX** |
|  |  |  |  |
|  |  |  |  |
| Location(s) covered by SOP: | Building: | **Duncan Hall** | Lab Phone: | **408-924-XXXX** |
| Room #(s): | **435** |

1. **HAZARD OVERVIEW**

Pyrophoric materials are substances that can ignite spontaneously upon exposure to oxygen or air. They can also be water-reactive, where heat and a flammable gas are produced. For Pyrophoric materials, oxidation of the compound by oxygen or moisture in air proceeds so rapidly that ignition occurs.

1. **HAZARDOUS CHEMICAL(S)/CLASS OF HAZARDOUS CHEMICAL(S)**

Typical Pyrophorics include, but are not limited to:

1. Metal hydrides (e.g. sodium hydride, diisobutylaluminum hydride, yttrium trihydride);
2. Some finely divided metal powders depending on particle size (e.g. Raney nickel, aluminum powder, zinc powder);
3. White phosphorus and some phosphine compounds (e.g. diphenylphosphine, trimethylphosphine);
4. Alloys of reactive materials (e.g. neodymium-iron-boron alloy)
5. Some organoborane and silane compounds (e.g. bromodimethylborane, trichlorosilane)
6. Some Grignard reagents (e.g. octylmagnesium bromide, allyl magnesium bromide); and
7. Some organometallic compounds, including alkyllithium and alkylzinc reagents (e.g. butyllithium, dimethylzinc).

Materials that spontaneously auto-ignite can be identified using the Safety Data Sheet by the Globally Harmonized System (GHS) using the Hazard Code H250 (Catches fire spontaneously if exposed to air).

**REQUIRED:** List (or attach) the applicable chemical(s) for your laboratory, and describe important properties and signs/symptoms of exposure. The chemical’s Safety Data Sheet (SDS) and [PubChem](https://pubchem.ncbi.nlm.nih.gov/)’s Laboratory Chemical Safety Sheet (LCSS) are excellent sources for this information.

*List of pyrophoric chemicals in the Dr. Jane Doe Lab*

*All of the following materials are defined as pyrophoric by the GHS classification system (Hazard code H250). The following list is to be used as a reference only; users should consult the SDS before use of any pyrophoric chemical. Many of the chemicals on this list also have GHS codes for water reactives (H260/261)*

***Tri-tert-butylphosphine*** *[13716-12-6] - Catches fire spontaneously if exposed to air. Keep away from heat, sparks, open flames and hot surfaces. Causes severe skin burns and eye damage.*

***Triethylphosphine*** *[554-70-1] – Highly flammable liquid and vapor that catches fire spontaneously if exposed to air. Keep away from heat, sparks, open flames and hot surfaces. Causes severe skin burns and eye damage. If exposed, user may experience nausea, headache and vomiting.*

***n-Butyllithium*** *[109-72-8] – Highly flammable liquid and vapor that catches fire spontaneously if exposed to air. In contact with water generates butane gas, which is also flammable. Causes severe skin burns and eye damage. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Symptoms of exposure include lung irritation, chest pain, edema, headache, dizziness, drowsiness, incoordination, slowed reaction time, slurred speech, giddiness, and unconsciousness. Prolonged or repeated exposure to skin causes defatting and dermatitis. Contact with eyes can causes redness and blurred vision.*

***tert-Butyllithium*** *[594-19-4] - Extremely flammable liquid and vapor that catches fire spontaneously if exposed to air. In contact with water generates isobutane gas, which is also flammable. Causes severe skin burns and eye damage. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Symptoms of exposure include lung irritation, chest pain, edema, headache, dizziness, and drowsiness.*

***Diethylzinc*** *[557-20-0] - In contact with water generates ethane gas, which is also flammable. Causes severe skin burns and eye damage. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Symptoms of overexposure include cough, shortness of breath, headache, and nausea.*

***Methylmagnesium chloride*** *[676-58-4] - Highly flammable liquid and vapor that may catch fire spontaneously if exposed to air. In contact with water generates methane gas, which may ignite spontaneously. Causes severe skin burns and eye damage. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Symptoms of exposure include lung irritation, chest pain, edema, headache, dizziness, and drowsiness.*

***Phenylethynyl magnesium bromide*** *[6738-06-3] - Highly flammable liquid and vapor that may catch fire spontaneously if exposed to air. In contact with water generates flammable gas, which may ignite spontaneously. Causes severe skin burns and eye damage. Suspected carcinogen. Symptoms of exposure include central nervous system depression, cough, chest pain, and difficulty in breathing. Exposure to high airborne concentrations can cause anesthetic effects.*

***Allylmagnesium bromide*** *[1730-25‑2] Highly flammable liquid and vapor that may catch fire spontaneously if exposed to air. In contact with water releases flammable gases which may ignite spontaneously. Causes severe skin burns and eye damage. Keep away from heat, sparks, open flames and hot surfaces. May cause drowsiness or dizziness.*

***Trichlorosilane*** *[10025-78-2] – Highly flammable liquid and vapor that may catch fire spontaneously if exposed to air. Toxic if inhaled. Avoid breathing vapors, mist or gas. Inhalation causes severe irritation of respiratory system. Causes severe skin burns and eye damage. Keep away from heat, sparks, open flames and hot surfaces. Take measures to prevent buildup of electrostatic charge. Reacts violently with water. Contact with water liberates toxic gas.*

1. **ENGINEERING/VENTILATION CONTROLS**

**The following is a general plan for all Pyrophorics:**

* Work under an inert and dry atmosphere (argon, nitrogen) in an enclosed glove box; or
* Work inside a properly functioning certified chemical fume hood using air-free (e.g., Schlenk) technique when handling Pyrophoric materials. Work with the sash as low as possible.

**Always:**

* Work away from water sources or potential water splash;
* Remove adjacent ignition sources and unneeded flammable/combustible materials;
* Use fresh, dry solvents; and
* If materials or side products are prone to rapid decomposition, use a portable blast shield.

**REQUIRED:** Describe the lab-specific engineering or ventilation controls and equipment safety features (if applicable) that will be used to reduce the risk of Pyrophoric chemical exposure.

*All work with Pyrophoric materials is carried out in a certified chemical fume hood.**Work shall be conducted with an inert solid (e.g. dry sand or MgO) within arm’s reach to extinguish potential fires.*

1. **ADMINISTRATIVE CONTROLS**

The following elements are required:

1. Complete laboratory safety training prior to working in the laboratory;
2. Complete laboratory-specific safety orientation and training on laboratory-specific safety equipment, procedures, and techniques to be used, including the location of laboratory safety equipment (emergency eyewash, safety shower, fire extinguisher);
3. Demonstrate competency to perform the procedures described in this SOP to the Principal Investigator (PI) or trainer;
4. Be familiar with the location and content of any Safety Data Sheets (SDSs) for the chemicals used (online SDSs are available from [MSDS online](https://msdsmanagement.msdsonline.com/8511b604-100d-449a-9a6b-366eff19da04/ebinder/?nas=True));
5. Inspect all equipment and experimental setups prior to use;
6. Follow best practices for the movement, handling, and storage of hazardous chemicals (see Chapters 5 and 6 of [Prudent Practices in the Laboratory](http://ucanr.edu/sites/ucehs/files/133892.pdf) for more detail). An appropriate spill cleanup kit must be located in the laboratory. Chemical and hazardous waste storage must follow an appropriate segregation scheme and include appropriate labeling. Hazardous chemical waste must be properly labelled, stored in closed containers, in secondary containment, and in a designated location;
7. Do not deviate from the instructions described in this SOP without prior discussion and approval from the PI; and
8. Notify the PI of any accidents, incidents, near-misses, or unexpected outcomes involving the Pyrophorics described in this SOP.

**For Pyrophorics, the following are also required:**

1. Never work alone. All work involving Pyrophorics must be performed in the presence of at least one safety buddy. The safety buddy must be a person who has been trained in the use of Pyrophorics and who is proficient with the Pyrophoric emergency protocols set forth in this SOP. Furthermore, the safety buddy must be within audible and visible range of the person that is handling Pyrophorics at all times, and must not be concurrently working with Pyrophorics or any other compound or process that cannot be easily and safely abandoned;
2. Keep quantities of Pyrophorics used as small as possible, especially when trying new experiments. Increase reaction scale with caution, and only when you have run the reaction on a small scale first. Never use Pyrophorics in a greater scale than has been approved by the PI;
3. Liquids may be safely transferred without a glovebox by employing certain syringe or cannula techniques. While a syringe is appropriate for the transfer of small volumes (less than 10 mL), larger volumes should only be transferred via cannula or in a glovebox. Never transfer more than one aliquot using the same syringe. Never transfer more than half of the volume contained by a syringe (e.g., do not transfer more than 5mL of liquid in a 10 mL syringe). Before transferring, make sure that the material is at the appropriate temperature [see SDS];
4. Ensure all equipment is dry, damage-free, air-free, clean, and appropriate for the task;
5. Be sure to have a quenching scheme for residual materials prior to beginning work;
6. Clear the area of unrelated and incompatible hazards;
7. Clear the area of clutter, especially flammable materials such as organic solvents and paper;
8. Know the location of eye wash/safety shower. Only use Pyrophorics in specific laboratory if the area is properly equipped with this safety equipment located within ten seconds of travel;
9. You **must** have an appropriate extinguishing agent (dry sand, Met-L-X, soda ash, or lime) for the Pyrophoric material you are using immediately available adjacent to your workspace;
10. Pyrophorics should have their own dedicated storage with secondary containment and shall be segregated from incompatibilities. Pyrophoric gases shall be stored in compliance with California Fire Code requirements. Please contact alexi.ball-jones@sjsu.edu for storage recommendations and requirements; and
11. Minimize your purchases of Pyrophoric materials to quantities that will be used within one year. The date of receipt and date of opening should be written onto the container. Contact alexi.ball-jones@sjsu.edu for the storage restrictions for your specific laboratory/building.

**REQUIRED:** Insert any laboratory-specific restrictions on maximum quantities of Pyrophorics to be used and stored. Include lab-specific transfer procedure(s), volume threshold for syringe vs. double cannula transfer methods, and any equipment restrictions.

1. *No more than 45 mmol of a pyrophoric reagent may be used without consulting Prof. Jane Doe.*
2. *No more than 10 mL of a pyrophoric material may be transferred at a time with a syringe. For larger quantities, use a cannula to transfer.*
3. *Tert-butyllithium presents an added hazard and all use must be approved by Prof. Jane Doe. Tert-butyllithium should always be transferred with a cannula using Schlenk techniques (not balloons).*

Storage Considerations for Pyrophorics:

* When appropriate, store under inert (e.g. N2, Ar) gas;
* Store all pyrophoric materials in the manufacturer’s container;
* Avoid heat/flames, ignition sources, oxidizers, protonating substances (e.g., acids, alcohols, etc.), and water sources; and
* Storage requirements of the individual materials must be considered (e.g. alkyllithium species should not be stored near strong Lewis acids despite the similar storage requirements for the two species).

**REQUIRED:** Insert descriptions of lab-specific storage, segregation, and any special handling requirements for each chemical or group of chemicals.

* *Organolithium compounds and trichlorosilane must be stored in the -20 C flammable materials storage freezer in Rm 435.*
* *All other Pyrophorics are stored in secondary containment in the pyrophoric materials dessicator in Rm 435.*
* *Allylmagnesium bromide is a peroxide forming material. The chemical must be used or disgarded within one year of opening. Never order more than will be used in one year.*
* *All pyrophoric materials are stores under dry argon or dry nitrogen. Dessicator is charged with dry inert gas for storage.*
1. **PERSONAL PROTECTIVE EQUIPMENT (PPE)**

At a minimum, long pants (covered legs and ankles) and shoes that cover the entire foot are required to enter a laboratory or technical area where hazardous chemicals are used or stored.

In addition to the minimum attire required upon entering a laboratory, the following PPE is required for all work with Pyrophorics:

1. **Eye Protection** (must be ANSI Z87.1-compliant)**:**
	1. At a minimum, safety glasses are necessary.
	2. Splash goggles may be substituted for safety glasses, and are required for processes where splashes are foreseeable or when generating aerosols.
	3. Ordinary prescription glasses are not acceptable eye protection and cannot be used in lieu of proper safety eyewear.
2. **Body Protection:** At a minimum, a flame-resistant (FR) laboratory coat that is NFPA 2112-compliant laboratory coat that fully extends to the wrist is necessary.
	1. Clothing worn under PPE should not be made from synthetic materials;
	2. For chemicals that are corrosive and/or toxic by skin contact/absorption additional protective clothing (e.g. face shield, chemically-resistant apron, disposable sleeves, etc.) are required where splashes or skin contact is foreseeable.
3. **Hand Protection:** Hand protection is needed for the activities described in this SOP. Use of Pyrophorics outside of an inert atmosphere glove box including, but not limited to, movement or handling of reagent bottles, reagent transfer, reagent quenching, and any spill cleanup activities requires:
	1. Gloves that provide sufficient protection from the specific chemicals being used, and ideally do not support a flame (e.g. neoprene is a better option than nitrile, such as Ansell NeoTouch disposable gloves).

**REQUIRED:** Insert lab-specific descriptions of PPE and hygiene practices used with Pyrophorics, including any specialized PPE needed for a procedural step or specific task.

*See considerations above to guide PPE selection based on scale of reaction and other reagents. Safety glasses, FR lab coat, and neoprene disposable gloves shall be worn, at a minimum. Do not wear clothing made of synthetic materials while working with pyrophorics.*

1. **SPILL AND EMERGENCY PROCEDURES**

Do not attempt to clean up a chemical spill unless you have been trained and feel comfortable doing so. Contact the College Safety Team or Environmental Health & Safety (EH&S), for help with cleaning up a small chemical spill. For a large spill of Pyrophoric materials, confine the spill within the fume hood or room, evacuate everyone from the lab, and call 911 (or 408-924-2222 from a non-campus phone).

**Once spilled, liquid or solid Pyrophoric chemicals may ignite.**

The primary emergency response is to extinguish Pyrophoric fires using an appropriate extinguishing agent (dry sand, Met-L-X, soda ash or lime). Primary emergency response to extinguish Pyrophoric fires on a person would be to use an emergency eyewash/safety shower. Additional considerations for a fire involving Pyrophoric materials includes:

1. If possible, immediately isolate the fire from any potential flammable material;
2. If you are not trained/comfortable with the fire extinguishing methods, close the hood completely to keep the fire contained (without additional fuel, small pyrophoric fires will self-extinguish as they are quickly combusted);
3. To extinguish the fire, use an appropriate extinguishing agent for the Pyrophoric material (dry sand, Met-L-X, soda ash or lime);
4. Using a dry chemical Class ABC fire extinguisher may be helpful to manage a collateral fire; and
5. Fire extinguishers containing water (or that may develop water over time), carbon dioxide, or halons are not suitable for fires involving Pyrophoric compounds as they react violently.

**REQUIRED:** Insert description of who to call in case of Pyrophoric spill in the lab.

*In case of Pyrophoric chemical spill, immediately call* ***Prof. Jane Doe at (personal cell phone contact #)*** *and* ***Randy Kirchner at 408-924-5004****.*

**REQUIRED:** Describe suitable extinguishing agent to be available in the lab that should be used to extinguish the Pyrophoric chemical spill.

***All Pyrophoric Material users:***

* *In case of a Pyrophoric material spill, announce the situation loudly in the immediate area and have any nearby persons move to a safe location.*
* *For small pyrophoric spills (spills less than 10 mL) cover the spill area with an inert extinguishing agent (e.g. dry sand) ONLY IF IT IS SAFE TO DO SO.*
* *For large pyrophoric spills in the fume hood (spills greater than 10 mL OR any spill that has ignited a fire), close the hood sash, evacuate the lab, call 911, and alert Prof Jane Doe and Randy Kirchner (phone numbers listed above).*

***For those trained to clean up a pyrophoric chemical spill****, use the following procedure for SMALL SPILLS ONLY. Students SHALL NOT attempt spill cleanup without Prof. Jane Doe’s help and supervision.*

*1) Cover the spill area with an inert extinguishing agent (e.g. dry sand)*

*2) Using non-sparking tools, transfer the pyrophoric/sand mixture to a large beaker*

*3) Quench the pyrophoric material with the following quenching scheme: slowly add first hexanes, then isopropanol, then brine, in a dropwise manner and each time waiting until no reaction is observed. Use a cooling bath if necessary.*

*4) Alert all emergency contacts listed on page 1 of this SOP.*

*Please note that if at any point during this process a fire ignites, follow the procedure for large pyrophoric spills and close the hood sash, evacuate, and call 911.*

If there is an unusual or unexpected occurrence when using Pyrophorics, the occurrence must be documented and discussed with the Principal Investigator and others who might be using the material(s). Unusual or unexpected occurrences might include a fire, catastrophic failure, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers. It is also essential that “Lessons Learned” and “Near Misses” incident reports be maintained and shared within the research group.

1. **WASTE MANAGEMENT AND DECONTAMINATION**

**Waste Management:**

Hazardous waste must be managed as outlined in [SJSU’s Chemical Hygiene Plan](http://www.sjsu.edu/fdo/departments/ehs/lab/Chemical%20Hygiene%20Plan_20191017-final.pdf), and must be [properly labeled](http://www.science.sjsu.edu/safety/HazWasteForm.pdf). In general, hazardous waste must be removed from your laboratory within 9 months of the accumulation start date.

**Decontamination:**

Carefully inspect work areas to make sure no Pyrophoric materials remain. Clean contaminated work areas with wipers moistened with a dry, non-polar solvent before using polar solvents to clean the area. Be sure all ignition sources are secured before beginning cleaning up with flammable liquids. Be certain that the appropriate quenching procedure is complete before adding materials to a hazardous waste container.

**REQUIRED:** Insert the lab-specific quenching protocol(s) for the Pyrophoric(s) described in this SOP.

*To quench pyrophoric materials, place container in cooling bath. In a dropwise fashion add the following sequentially: hexanes, isopropanol, and brine, adding each until no reaction is observed.*

**REQUIRED:** Insert description(s) of decontamination procedures for equipment, glassware, and/or controlled areas (e.g. gloveboxes, restricted access hoods, or designated portions of the laboratory).

*Ensure that all materials that have come in contact with the Pyrophoric materials have been quenched according to the quenching procedure above, including the fume hood work surface (though using water instead of brine for final surface wash).*

Upon completion of work with Pyrophorics and/or decontamination of equipment, remove gloves and wash hands with soap and water. Upon leaving the laboratory or designated Pyrophoric work area, remove all PPE worn and wash hands and forearms as needed. Contaminated PPE should not be worn outside of the laboratory. Soiled lab coats should be sent for professional laundering. Grossly contaminated clothing/PPE, and disposable gloves must not be reused and should be disposed of as hazardous waste.

1. **DESIGNATED AREA**

Designated area(s) for the use and storage of Pyrophorics shall be established where limited access, special procedures, knowledge, and work skills are required. Signage indicating the corresponding [Globally Harmonized System (GHS) pictogram(s)](https://www.osha.gov/Publications/HazComm_QuickCard_Pictogram.html) should be visible at the entrance of the designated area (e.g. postings on the exterior of the laboratory door).

**REQUIRED:** Insert description(s) of the designated area(s) for Pyrophorics in your laboratory. The entire laboratory, a portion of the laboratory, a fume hood, etc. can be designated.

*Laboratory room 435 is designated for pyrophorics use. All manipulation of pyrophorics outside the storage container must occur in a properly functioning certified fume hood. Fume hoods in which pyrophorics are being actively used should be indicated with a sign that reads: "DANGER - PYROPHORICS IN USE"*

1. **DETAILED PROTOCOL**

**REQUIRED:** Insert the lab-specific protocol for the process, hazardous chemical(s), or hazard class described in this SOP. Include any relevant resources such as journal articles, patents, etc. as desired.

*Lab workers using Pyrophorics must demonstrate competence to Prof. Jane Doe by being able to 1) identify the hazards and list any particularly hazardous handling techniques involved in the proposed reaction (e.g. use of a schlenk line, cannula transfer, extremes of pressure or temperature, etc.); 2) list the foreseeable emergency situations; 3) describe the proper response to the emergency situations; and 4) know the control measures to minimize the risks.*

*When working in the lab with a Pyrophoric material, a laboratory worker must:*

*1. Never work alone;*

*2. Be cognizant of all the SDS and safety information presented in this document;*

*3. Find/follow a literature experimental procedure describing the use of the needed Pyrophoric reagent in a related chemical transformation. If a pertinent literature protocol cannot be found, the researcher MUST discuss the planned experiment with Prof. Jane Doe prior to using the Pyrophoric reagent;*

*4. Not deviate from the literature experimental protocol mentioned in (3) in either temperature or pressure without PRIOR APPROVAL from Prof. Jane Doe;*

*5. Follow all related SOPs in the laboratory SOP bank (e.g. process-specific such as work within a glovebox, or hazard-specific such as working with Carcinogens).*

*Discuss ALL issues or concerns regarding Pyrophorics with Prof. Jane Doe prior to their use.*

***Transportation of pyrophoric materials*** *from storage location to use location in the laboratory will be accomplished in the following way:*

* *Bottles will be transported in bottle carriers or other non-combustible secondary containment.*
* *As much as is feasible, transportation will not occur alone. It is highly recommended that two people transport pyrophoric materials. Transport of >50 mL requires a buddy.*

***Proper syringe/needle transfer is as follows:***

1. *All glassware and solvents should be dried prior to the treatment with any Pyrophoric chemical. Flame-dried glassware which has been cooled under inert atmosphere just before use is ideal. Upon charging a dry flask with dry reagents/solvents and reaching the desired temperature, the appropriate reagent can be added.*
2. *The Pyrophoric is contained in a Sure-Seal bottle, the septum of which can be pierced by clean, dry needle fitted to a Schlenk line which is supplying an inert gas. If the bottle does not have a Sure-Seal, seal the top of the bottle with a septum. This line is used to keep the reagent under a blanket of inert gas during the entire process. The inert gas flow is adjusted by monitoring the oil bubbler on the Schlenk line.*
3. *A clean, dry reagent needle (<16 gauge) with the appropriate size syringe is used to pierce the septum and inert gas is drawn into the syringe, then the needle is expelled to the atmosphere. This process is repeated several times, piercing the same whole in the septum, to remove most air from the needle and syringe.*
4. *Upon 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.*
5. *Once the desired amount of Pyrophoric has been drawn into the syringe, the tip of the needle 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.*
6. *At this point the Pyrophoric reagent is dispensed, usually slowly to control heat evolution Be 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. Once the addition of reagent is complete, the needle can be removed from the reaction flask.*
7. *The excess reactive reagent can be expelled into a beaker of sand or onto a chunk of dry ice. After the excess reagent has been expelled, the needle can be rinsed with hexanes multiple times, then water.*
8. *Clean the needle immediately after use to prevent clogging. To close the reagent bottle, simultaneously place a piece of tape over the hole formed by the needle while removing it from the bottle. This should seal the bottle and keep air and moisture from entering. Replace the cap and wrap the outside with parafilm to further ensure safe storage. Quench hexane wash with isopropanol.*

***Proper transfer by cannula for volumes greater than 10 mL:***

1. *Pressurize the Sure/Seal bottle with nitrogen and then insert the double tipped needle through the septum into the headspace above the reagent. Nitrogen will pass through the needle.*
2. *Insert the other end through the septum at the calibrated addition funnel on the reaction apparatus which must be equipped with a gas line to a bubbler.*
3. *Push the needle into the liquid in the Sure/Seal reagent bottle and transfer the desired volume, then withdraw the needle above the liquid level. Allow nitrogen to flush the needle.*
4. *Remove the needle first from the reaction apparatus and then from the reagent bottle.*
5. *Alternatively, for an exact measured transfer, convey from the Sure/Seal bottle to a dry nitrogen flushed graduated cylinder fitted with a double-inlet adapter. Transfer the desired quantity and then remove the needle from the Sure/Seal bottle and insert it through the septum of the reaction apparatus.*
6. *Apply nitrogen pressure as before and the measured quantity of reagent is added to the reaction flask. To control flow rate: fit a Luer lock syringe valve between two long needles.*
7. *Needles used with pyrophoric reagents must be cleaned immediately to avoid clogging. Flush double-tipped needles with hexane and then quench hexane wash in isopropanol.*

***Final notes:***

* *Eliminate all potential incompatibles and combustibles from the work and potential spill area.*
* *Your reaction isn’t done until everything is quenched appropriately and according to the procedures described above for needles or in Section 7.*
* *Hazardous waste shall be managed according to its type (organic, aqueous, solid). Never mix wastes.*
* *Never work alone!!!*

**TEMPLATE REVISION HISTORY**

|  |  |  |  |
| --- | --- | --- | --- |
| **Version** | **Date Implemented** | **Author** | **Revision Notes:** |
| **1.0** | **4/3/2020** | **Alexi Ball-Jones** | **New template** |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

**LAB-SPECIFIC REVISION HISTORY**

|  |  |  |  |
| --- | --- | --- | --- |
| **Version** | **Date Approved** | **Author** | **Revision Notes:** |
| **1** | 4/3/2020 | **Dr. Jane Doe** | New Template |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |
|  |       |  |       |

**Documentation of Standard Operating Procedure Training**

*(Signature of all users is required)*

* Prior to use of **Pyrophorics**, laboratory personnel must be trained on the hazards involved in working with this SOP, how to protect themselves from the hazards, and emergency procedures.
* Ready access to this SOP and to a Safety Data Sheet for each hazardous material described in the SOP must be made available.
* The Principal Investigator (PI), or the Laboratory Supervisor if the activity does not involve a PI, must ensure that their laboratory personnel have attended appropriate laboratory safety training or refresher training within the last three years.
* Training must be repeated following any revision to the content of this SOP.

**Designated Trainer:** *(signature is required)*

I have read and acknowledge the contents, requirements, and responsibilities outlined in this SOP:

|  |  |  |  |
| --- | --- | --- | --- |
| **Name** | **Signature** | **Trainer Initials** | **Date** |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |