# **Standard Operating Procedure (SOP) Title: Pyrophoric Materials**

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| Assessor:  | Joshua Linfoot | Location of work:  | MSRH 502 |
| Principal Investigator:  | Prof Alan Spivey |
| Date of approval:  | 13/09/2021 | Date for review: | 13/09/2022 |

## **Justifying the hazards:**

The usage of pyrophoric chemicals is required due to their unique reactivities.

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| Identify hazards with specific risk assessments and a College or a departmental approval process  |
| [Ionising radiation sources](https://www.imperial.ac.uk/safety/safety-by-topic/laboratory-safety/) | [ ]  | [Biological sources](https://www.imperial.ac.uk/safety/safety-by-topic/laboratory-safety/) (microorganisms, human/animal tissues, plants) | [ ]  |
| [Class 3R, 3B or 4 Lasers](https://imperiallondon.sharepoint.com/sites/fons/faculty/safety/lasers/SitePages/laserhome.aspx) | [ ]  | [Offsite work](http://www.imperial.ac.uk/safety/safety-by-topic/off-site-working/) | [ ]  |
| Confirm if [Lone working](https://www.imperial.ac.uk/safety/safety-by-topic/lone-working/) is permitted with this SOP? [ ]  If it is permitted, describe the control measures for lone workers: No Lone Working with this SOP! |

## **Preparing for the SOP:**

* **DO refer to the** [**2.33 Code of Practice Working safely with pyrophoric chemicals**](https://imperiallondon.sharepoint.com/sites/fons/faculty/safety/SitePages/Chemistry-Department-Approved-SOPs.aspx?Mode=Edit)(see the list of [Departmental SOPs](https://imperiallondon.sharepoint.com/sites/fons/faculty/safety/SitePages/Chemistry-Department-Approved-SOPs.aspx?Mode=Edit))
* **DON’T** start before designing a disposal/quenching scheme (never use water!) for residual materials.
* **DON’T** use acetone or 2-propanol cooling baths.
* **DO** check **FIRETRACE** indicator on FC (see SOP for Fumehood) and keep a container of sand near to the experiment for **immediate** use in event of ignition.
* **DO** use a **cannula technique** for large volumes (>12ml) and a **syringe** for smaller volumes. Syringe must be capable of containing **twice the volume you wish to transfer. Do not use syringes larger than 24 ml!**
* **DO** remove all sources of ignition and combustion from the area of reaction/ storage.
* **DO** turn the taps of the schlenk line SLOWLY!
* **DO** consider whether a less pyrophoric reagent may be used (e.g. NaH instead of KH, LDA or n-BuLi instead of tBuLi)

## **Procedure:**

# **Before the procedure:**

# Unpack the purchased box of material in a fume cupboard.

1. Pyrophoric liquids, or compounds dissolved in a liquid, must be stored in containers that are sealed with PTFE-lined septa to prevent exposure to the air.
2. Complete an appropriate **Reaction Risk Assessment form** and notify at least one of your colleagues with appropriate training about work you are about undertake. It is recommended to complete the [full COSHH form](http://www.imperial.ac.uk/safety/forms/).
3. Before use, allow the stock bottle to warm to room temperature in the fume cupboard to minimise condensation on both the outside of the bottle and within the bottle.
4. Set up apparatus using clean dry glassware from the desiccator; ensure a tight seal and rubber septa to the reaction flask for liquids withdrawal. Dry N2/Ar vacuum manifolds should release gas into hood via mineral bubbler for positive pressure and preventing air from entering the system (balloons of inert gas can also be used).
5. Ensure vacuum is attached to manifold via a cold trap to catch any contaminants.

# **Procedure:**

1. Ensure the reaction flask is under N2.
2. Add dry, degassed (oxygen free) solvent to the reaction flask. Purge the atmosphere in the flask by alternating N2 flushing with vacuuming a few times.
3. Once N2 is blanketing the solvent, and where a coolant is needed, the reaction vessel is placed in a coolant bath. Depending on the reaction, inert hydrocarbon solvents can be used such as heptane with dry ice.
4. Transfer the pyrophoric liquid using a cannula or syringe. Please see [**Best practice procedures for working with pyrophoric chemicals**](https://imperiallondon-my.sharepoint.com/%3Ab%3A/g/personal/fonssafety_ic_ac_uk/ETpD7LGGh3xJjry_MEqmEH0BaDfcQpGxhD2IDykumezbSA?e=OobXNg) for a more detailed procedure.

**When handling Pyrophoric Solids**

Weighing and transferring alkali metals: Cut the desired piece of alkali metal under packing oil using a scalpel. If rinsing of oil is required, use tweezers and transfer the cut metal to a flask containing toluene or heptane to rinse off oil. Use tweezers to transfer the (rinsed) metal to a weighed flask of toluene and measure weight to determine mass of metal. Use tweezers again to transfer to desired reaction flask.

Weighing and transferring granulated and powdered pyrophoric solids should occur under an inert atmosphere (refer to the relevant SDS, e.g. argon for Lithium).

# **After the procedure:**

1. After completing of the transfer, N2 is drawn into the syringe and the needle removed from the reaction flask, and then flushed three or four times with a 50:50 mixture of toluene and isopropanol.
2. Remove the plunger from the syringe barrel to allow contact with air and dry in the fume cupboard for 30 minutes. Dispose or clean and dry according to the syringe type.
3. Make sure you **leave the area and inside of the fume cupboard clean**.
4. Wash glassware following quenching procedure for any residues.

## **Disposal:**

As long as the bottle container and seal are intact, the stock bottle should be disposed of via the hazardous waste stream. Disposal of a 100 mL bottle of tert-butyllithium solution via the hazardous waste stream is only 50p (as of May 2020). Contact the Chemistry Research Technicians to arrange for disposal.

## The majority of the pyrophoric reagent should have been consumed during the reaction however, DO NOT assume that the risk from the reagent is low. **Dispose of as hazardous waste** in the lab using the available chemical waste disposal routes. If disposal is not appropriate, **quench any residual reagent with isopropanol under N2** (with a cooling bath in place for large-scale reactions or dry toluene solvent to absorb heat), methanol or ethanol and water subsequently to ensure the complete quenching of the pyrophoric material.

## **Personal Protective Equipment (PPE):**

Lab coat, appropriate gloves, safety glasses.

In some cases (for example if using >100 mL of a pyrophoric liquid or more than 5 g pf pyrophoric solids) additional personal protective equipment may be required, for example, a flame resistant lab coat, flame resistant gloves (a non-combustible neoprene, leather, Kevlar or Nomex Flight (fighter pilot) gloves), safety goggles and a face shield.

## **Risk Analysis of SOP and emergency procedures:**

### (In addition to [Safe Lab Practice](https://imperiallondon.sharepoint.com/sites/fons/faculty/safety/SitePages/Basic%20Laboratory%20Rules%20for%20All%20Laboratories%20in%20FoNS.aspx))

### **Always remember to include fire associated risks and control measures where appropriate**

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| Hazard | Raw risks | Current control measures | Residual risk(Low/Med/High) |
| **Pyrophoric chemicals** -Spontaneous ignition in air at a temperature of 54°C or below (gas) / within 5 minutes after coming into contact with air (liquids and solids) | Burns, inhalation, fire | 1. Reduction in the quantity of dangerous substances to a minimum
2. Work in a fume cupboard (or glove box) with access to an inert gas and vacuum system or purging methods should be used.
3. Always work with the FC sash lowered.
4. CHECK THE FIRETRACE GAUGE ON THE FUMEHOOD for a correct level, if present.
5. Ensure the fume cupboard or other enclosure is clear of chemicals and items not involved with the reaction, in particular, flammable solvents and other sources of ignition.
6. A bucket of sand (or other inert absorbent) must be immediately accessible (not CO2 extinguishers, which can react with some organolithium compounds and alkali metals and hydrides). If a powder extinguisher is required, this must be justified in the risk assessment, reviewed by the Faculty Safety Team and then approval given from the College Fire Office.
7. Ensure all glassware that will come into contact with liquid pyrophoric chemicals are clean and absolutely dry.
8. The N2/Ar used to create the inert atmosphere must be dry and high purity (oxygen free).
9. When pinch seal with needle/cannula is used, ensure needle’s edge remains above the level of pyrophoric liquid.
10. When transferring *via* syringe, ensure a small volume of solvent (under N2 with a septum) is available (50:50 toluene: isopropanol) for flushing needles and syringes of excess reagent after transfer. The isopropanol will inactivate any residual pyrophoric liquid while the toluene will act as a heat sink for the quench.
11. Use Luer lock syringes. For 1 mL syringes, protect the connection between syringe and the needle with PTFE tape.
12. Ensure another worker is with you and ready to assist if needed – never work alone.
13. Many pyrophoric solids are sold as solutions or dispersions in mineral oil, or are covered with hydrocarbon solvents to facilitate use.

When rinsing pyrophoric solid of the solvent, avoid using low boiling rinses such as ether and pentane, which tend to condense water upon evaporation. Use toluene or heptane. | Med |
| Glassware and glass parts | Cuts and splinters from broken glass  | Visually inspect glassware for cracks and other defects before and after use. If glassware damaged arrange for repair or dispose of. | Low |
| Sharp needles or cannulas | Puncture, Injection of hazardous materials into body/blood stream | Use the needle so that the point faces away from the operator and others around.If applying pressure to the syringe containing hazardous agents use a Luer lock or similar secure system to ensure the needle is firmly attached to the syringe.Ensure needles are never re-sheathed with their plastic cap.If a needle is dropped, it must be retrieved immediately. If it cannot be found, inform people of the danger, and search the area until it is found and disposed of.Do not leave needles exposed on benches or work surfaces, store in a suitable container.Clamp down rather than hold equipment in your other hand to avoid stabbing due to slippage. | Med |
| Hazardous materials | Exposure to hazardous reagents via inhalation or skin contact | No working with hazardous reagents outside of the FC. Always wear appropriate PPE.Ensure the FC and sample preparation areas are cleaned after each use.(Include hazards and controls of associated reagents in this or separate risk assessment) | Low |
| Organic solvents | Risks associated with specific solvent | Follow the control measures established in the COSHH assessment for the appropriate solvent. | Low |

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| **Additional control measures to minimise residual risks** | **Implementation date** |
| Consider placing a shield in front of the reaction to augment the fume cupboard sash until the reaction is complete. |  |

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| **Who may be harmed** |
| Staff / students [x]  | Cleaners / Engineers [x]  |
| Supporting staff [x]  | Others (specify):  |

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| **Emergency procedures** – describe the response(s) required by the user and lab members |
| **Pyrophoric** **fires** may be smothered with sand or powder extinguishers. If working with larger quantities of water/air sensitive solids (> couple of g) in one reaction, ensure the presence of specialised L2 powder extinguisher. If nobody is injured and nothing has ignited, use the appropriate **pyrophoric** **spill** control materials for pyrophoric chemicals (these are designed to be inert and will not react with the reagent (CaO)). Quench the spill by slowly adding isopropanol. After quenching is complete, collect and double bag spill residues and dispose as hazardous waste.Clear up **broken glass** using dustpan and brush, tweezers or other suitable equipment to prevent exposure to the glass, and place into the appropriate waste bin (clean or contaminated glassware).If anyone is injured while using the equipment contact first aider. If any **cuts or exposures** to hazardous substances, ensure affected area is held under running water for at least 15 mins and the wound is encouraged to bleed, ask for first aid assistance. If water is not available use alcohol free wipe from the First Aid Kit and dress the wound. Seek further medical attention if required.**Chemical spills, risks specific to hazardous substances** - dependant on the nature of the chemical. Specific procedures will be outlined on an individual basis on the relevant COSHH form.(Include emergency procedures associated with the use of hazardous substances if relevant) |

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| Recommended trainings and records: |
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| List of individuals competent to demonstrate safe work practice and train others (level 1 trainers): | Names of those that have been trained and can work unsupervised (level 2) and date training completed: |
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