



Requirements for Pyrophoric Research

RevDate: 08/01/2023

DOCUMENT USE:

This document is chemical specific information provided for the safe handling, use, and disposal of pyrophoric's. This information is required to be addressed and included as part of an experiment specific risk assessment for the purposes of completing a written standard operating procedure (SOP). Visit the EHS website for additional information/guidance: [SOPs for Hazardous Chemicals](#).

A Brief Hazard Summary/Overview

Pyrophoric materials have the ability to spontaneously ignite upon exposure to oxygen at temperatures of 130 °F (54 °C) or below without the influence of heat or fire. The high level of reactivity associated with these chemicals requires them to be handled in inert atmospheres, free of ignition sources. In addition to reactivity and flammability, many of these chemicals are also classified as acutely toxic, corrosive, reproductive toxins, peroxide-forming agents or capable of damaging the liver, kidneys, and central nervous system.

The use of appropriate engineering controls and personal protective equipment, together with good laboratory housekeeping, will help to minimize the severity and impact of an accidental fire. Failure to follow proper handling techniques could result in serious injury or even death to the person handling the materials or to others in close proximity

Quantity/Concentration Hazards

Pyrophoricity scales significantly with concentration. Exercise greater caution when working with more concentrated solutions. Do not attempt to syringe transfer volumes greater than 15 mL; perform multiple, smaller volume transfers instead.

Routes of Exposure

Skin contact, absorption & inhalation.

Types

Pyrophoric materials may be in the solid, liquid or gas phase. Pyrophoric liquids are often stored in flammable solvents such as ether, THF or a hydrocarbon. Pyrophoric solids are often stored under oil or kerosene. Additional examples of pyrophoric reagents are metal hydrides, finely divided metal powders, nonmetal hydride and alkyl compounds, white phosphorus, alloy of reactive metals, and selected organometallic compounds; including alkyllithiums.

Most of pyrophoric reagents fall into the following categories:

- Alkyl aluminum reagents
- Alkyl zinc reagents
- Alkylphosphines
- Alkylsilanes
- Sodium/Potassium Alloy (NaK)
- Grignard Reagents: RMgX
- Metal alkyls and aryls: tert-butyllithium, n-butyllithium, phenyllithium
- Metal carbonyls: Lithium carbonyl, nickel tetracarbonyl, iron pentacarbonyl
- Metal hydrides: Sodium hydride, potassium hydride, diisobutylaluminum hydride
- Nonmetal hydrides: Diethylarsine, diethylphosphine
- Non-metal alkyls: R3As, R3B, R3P, R2Cd

Work Practice Controls

1. Review the Safety Data Sheets (SDS), technical bulletins ([Aldrich Technical Information Bulletin AL-134](#)), and guidance documents to understand how to manage the hazards.
2. Principal investigators must develop and implement Standard Operating Procedures (SOP's) for work practices and procedures involving pyrophoric chemicals as well as other highly reactive chemicals.
3. Whenever possible, principal investigators should consider the use of less hazardous, alternative chemicals.
4. Use pilot reactions to confirm the appropriate procedures, processes, and safe guards are in place prior to scaling up.
5. Given the significant hazards presented by pyrophoric chemicals, any researcher performing an experiment with this class of chemicals **MUST NOT WORK ALONE**. Experiments should be planned so that this work is not done after normal business hours.
6. ALWAYS wear the appropriate personal protective equipment.
7. Remove all excess and nonessential chemicals and equipment from the fume hood or glove box where pyrophoric or water reactive chemicals will be used. This will minimize the risk if a fire should occur.
8. Keep the amount of pyrophoric materials present in the lab to the smallest amount practical. Use and handle the smallest quantity practical. It is better to do multiple transfers of small volumes than attempt to handle larger quantities at once.
9. Remove any potential ignition sources. Handle all reactive chemicals with extreme care, and store them away from incompatible chemicals.
10. Ensure that lab personnel know the locations of the eyewashes and safety showers and know how to activate them in the event of an emergency.

Training

No researcher may work independently with the hazardous materials described in this document until the Principal Investigator (or their designee) has ensured that the researcher:

- Complete EHS online Laboratory Safety training: OH102, 101,202 and 201, available through UT Learn (<https://ehs.utexas.edu/training/lab-training-requirements>).
- Other EHS training requirements (e.g., fire extinguisher use, compressed gas) as appropriate.
- Complete training on operation of specialized equipment prior to use (e.g., ultracentrifuge, hydrogenation apparatus, glovebox).
- Understand the hazards of the materials and risks of the processes involved.
- Has read and understand the contents of the labs pyrophoric SOP.
- Demonstrates the ability to execute their work according to the requirements in the SOP.

All users of pyrophoric reagents in the laboratory must receive hands-on instruction from an experienced senior member of the laboratory and must be closely supervised until safe work practices are consistently demonstrated. This training must be documented in the SOP.

Engineering Controls

All manipulation of liquid or solid pyrophoric materials must be conducted inside an inert glove box or properly functioning fume hood with the sash level at the lowest height possible to perform the required operations. **Refer to the methods outlined in APPENDIX A & B.** Before starting work, clear the fume hood or glove box of any unnecessary equipment or chemicals. A suitable eye wash station, safety shower, and appropriate fire extinguisher are required to be readily accessible.

1. Fume Hood

- Many pyrophoric chemicals release noxious or flammable gases, and some pyrophoric materials are stored under kerosene or mineral oil. These materials must be handled in a laboratory hood with sash in the lowest feasible position. Verify that your fume hood has been checked in the last 12 months.

2. Safety shielding

- If the potential exists for explosion of a high thermal reaction, additional shielding should be utilized. Portable shields may also be used for additional protection.

3. Glove box

- Glove boxes are an excellent device to control pyrophoric chemicals when inert or dry atmospheres are required. The PI is responsible for ensuring personnel are trained and competent in using a glove box, and that ergonomic considerations for glove box use are addressed.

Personal Protective Equipment (PPE)

Gloves (nitrile), fire resistant lab coat, and safety glasses / goggles. With more concentrated or neat solutions, a face shield and fire-resistant gloves are required.

1. Eye Protection

- Chemical splash goggles or safety glasses that meet the ANSI Z.87.1 standard and must be worn whenever handling pyrophoric chemicals. Goggles must be worn to protect from splash, and when appropriate, a face shield is needed for added protection.

- A face shield, worn over safety eyewear, is required any time there is a risk of explosion, large splash hazard, or a highly exothermic reaction. All manipulations of pyrophoric chemicals that pose this risk should be carried out in a fume hood with the sash in the lowest feasible position.

2. Skin Protection

- Gloves must be worn when handling pyrophoric chemicals. Be sure to use adequate protection to prevent skin exposures. Sigma-Aldrich recommends the use of nitrile gloves underneath neoprene gloves. Flame retardant gloves should be used for handling large quantities of these chemicals.
- A lab coat made from fire retardant material is recommended for labs using these reagents routinely. Lab coats need to be buttoned and fit properly to cover as much skin as possible.
- Appropriate shoes that cover the entire foot (closed toe) and long pants must be worn.
- Avoid wearing clothing made out of highly flammable synthetic fabrics such as acrylic or polyester.

Transportation Procedures

Newly received packages of pyrophoric chemicals should be transported through our buildings in the same packaging designed for DOT transport. Containers should not be unpacked until they can be transferred into a desiccator or other appropriate storage area. Materials should be unpacked for use and then transferred to a dry box or a chemical hood and used as instructed.

Storing Pyrophoric Reagents

It is **highly** recommended that pyrophoric reagents are purchased in quantities that will be used in each experiment to avoid the storage of excessive amounts. Never return excess chemicals to the original container. If small amounts of impurities are introduced into the container, it may cause a fire or explosion.

If storage is needed, consult the SDS for any special storage considerations or incompatibilities. Containers carrying pyrophoric materials must be clearly labeled with the complete chemical name and hazard warning. If cold storage is required the refrigerator must be designed for the storage of flammable liquids, and must be designated for the storage of pyrophoric liquids.

Waste Disposal Procedures

Small amounts of residual pyrophoric liquids must be carefully and completely quenched as part of your experimental procedure before they are collected for waste disposal. In addition to the general practices described below, follow procedures established by the PI in the lab-specific SOP and chemical-specific SDS.

- A container/contaminated waste with any residue of pyrophoric materials should never be left open to the atmosphere but must be properly contained to prevent fires.
- Transfer the materials to an appropriate reaction flask under inert atmosphere for hydrolysis and/or neutralization with adequate cooling.

- To the flask add an inert dry solvent, such as toluene or THF, to dilute the reactive reagent under an atmosphere of dry nitrogen or argon.
- The stirred solution should be neutralized by the drop-wise addition of an ice-chilled protic solvent, such as isopropyl alcohol. Never add water or a protic solvent directly to a pyrophoric reagent.
- Then slowly add methanol, as a stronger quenching agent, until no further reaction is observed.
- Finally, add water dropwise until no further reaction is observed, ensuring there is no residual pyrophoric material.
- Once emptied, the flask should then be triple rinsed with isopropyl alcohol and left to dry in the back of a fume hood for a 24-hour period.
- The original container should be triple-rinsed with a dry inert solvent under a flow of inert gas. These rinses should be then treated as described above. The triple-rinsed container should be left open in back of a hood exposed to the ambient atmosphere for at least a week to ensure all remaining residue has been quenched. After a week, the container should then be triple rinsed again with isopropyl alcohol.

Dispose of the resulting waste according to existing protocols for hazardous chemical waste. If you have large quantities of unreacted pyrophoric reagent material, contact EHS for guidance on disposal options.

Store Waste

- Hazardous waste tags must be placed on the hazardous waste container upon the start of accumulation.
- Hazardous waste containers must be kept closed, except when adding waste.
- Hazardous waste containers must be stored in secondary containment to adequately contain all of the contents of the container.
- Hazardous waste containers must be inspected weekly for signs of leaks, corrosion, or deterioration.
- Fill out and submit the online “Request for Chemical Waste Disposal” form in EMS. More information is available [HERE](#).

Emergency Procedures

1. Eyewash/ Safety Shower

A combination eyewash/safety shower should be within 10 seconds travel time from any location where pyrophoric chemicals are used. Bottle type eyewash stations are not acceptable.

2. Spills or Releases:

Extreme caution must be taken with all spills due to the risk of spontaneous combustion. Carefully remove all flammable materials from the immediate area and cover the spill with sand or powdered lime. Carefully quench / neutralize the resulting mixture as described in the

Waste Disposal section, and dispose of the resulting waste according to existing protocols for hazardous chemical waste.

- **DO NOT** use water on a pyrophoric reagent fire, as it can make the incident even worse.
- Do not use combustible materials like paper towels to clean up a spill, as these may increase the risk of ignition with a pyrophoric reagent. Soda ash (powdered lime) or dry sand may cover and contain any small spill that occurs.
- Assess the extent of danger. Help contaminated or injured persons if safe to do so. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

3. Chemical Spill on Body or Clothes

Remove contaminated clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. **Notify supervisor and EHS at 512-471-3511 immediately.**

4. Small Spills (<100 g)

Even small spills of a pyrophoric material, especially liquids, can be extremely hazardous, especially if other flammable compounds or solvents are in the lab or fume hood. If the fire can be readily contained and you have had training, you may assist in the clean-up effort. Use appropriate personal protective equipment and clean-up materials for chemical spilled. The spill should be completely covered with soda ash (lime) or dry sand followed by a slow quenching with isopropanol. Once the material has been completely quenched and all reactions have ceased, the mixture should be placed in a sealed container for disposal. Double bag neutralized waste in clear plastic bags or approved container(s), label and take to the next chemical waste pick-up.

5. Large (>100 g)

If a large spill occurs, evacuate the area, close the doors, dial 911 and contact EHS at: 512-471-3511.

6. Medical Emergency

- Life Threatening Emergency – Dial 911 if the condition is LIFE THREATENING or REQUIRES IMMEDIATE MEDICAL ATTENTION
- Non-life-threatening emergency – Notify your supervisor and EHS if condition is not life threatening.
- Needle stick/puncture exposure (as applicable to chemical handling procedure) – Wash the affected area with antiseptic soap and warm water for 15 minutes. For mucous membrane exposure, flush the affected area for 15 minutes using an eyewash station.

All serious injuries must be reported to EHS at 512-471-3511.

7. Fire

DO NOT use a carbon dioxide fire extinguisher or water to attempt to extinguish a pyrophoric material fire as these types of extinguishers can actually enhance the combustion of some pyrophoric materials.

- The recommended fire extinguisher is a standard dry powder (ABC) type and should be within immediate reach in the event of an incident. Cover the residue with sand or powdered lime to prevent reignition.
- Class D extinguishers are recommended for combustible solid metal fires (e.g. sodium, LAH), but not for organo-lithium reagents.
- A small beaker of dry sand or soda ash (lime) in the work area is useful to extinguish any small fire that occurs at the syringe tip and to receive any last drops of reagent from the syringe.
- For larger fires, contact emergency services for assistance and evacuate the area.

8. Signs and Symptoms of Exposure

Burning sensation or flames.

9. Exposures

Wash affected area with copious amounts of water. If on fire, extinguish in lab safety shower and contact emergency services for immediate medical treatment.

Procedure Review

All pyrophoric SOPs must be reviewed annually by the PI or laboratory manager.

PROCEDURE MODIFICATIONS/REVISIONS

Current Date	Modifications or Revisions	Name
05/08/2020	Original document	Naween Dahal
9/9/2022	Re-Formatted	Andrea McNair
9/30/2022	Editing and rearrangement	Rudy Guerrero
10/3/2022	Review and edits	Naween Dahal, Andrea McNair, Rudy Guerrero
8/1/2023	Review and Edits	Naween Dahal, Andrea McNair, Rudy Guerrero

APPENDIX A

Pyrophoric Solids

Although pyrophoric solids are more stable to air than pyrophoric liquids, caution must be exercised during handling, storage, and disposal. Finely divided pyrophoric solids must be transferred under an inert atmosphere in a glove box. In general, pyrophoric reagents are usually handled in a glovebox or Schlenk line under an inert gas. Some pyrophoric solids, such as lithium and sodium, are sold as dispersions in mineral oil to reduce the rate of oxidation to oxides/hydroxides and the pyrophoric character of the reagents. Removal of the mineral oil is accomplished by cutting a piece of the alkali metal and transferring it to a flask containing a hydrocarbon solvent such as hexane to rinse off the oil. Other mildly pyrophoric solids, such as dispersions in oil of sodium hydride (NaH) and lithium aluminum hydride (LiAlH₄), can be handled in air for short periods of time. The dry solid forms should be handled under inert atmosphere. In addition, the containers must be kept under a blanket of inert gas during storage.

Sodium Metal

Sodium reacts violently with water. Store in mineral oil in a sealed container kept away from water. Do not touch the metal directly. Handle with tweezers/tongs which have been washed with hexanes, and cut into smaller pieces with a razor blade which has also been rinsed with hexanes. Rinse the mineral oil off the desired amount using hexanes. Work away from any source of water. Keep away from heat sources.

In case of fire, never use water. Do NOT use CO₂ or other fire extinguishers. Cover the flame with dry sand and let the fire extinguish itself, adding additional sand as needed. Wear sufficient protective gear clothing/gloves/goggles at all times.

WARNING!

If sodium amalgam (Na[Hg]) needs to be prepared and used, use extreme caution. Dissolving sodium into liquid mercury results in a very exothermic reaction; producing the intermetallic compound NaHg₂ with enough heat to cause localized boiling of the mercury. The preparation of sodium amalgam should be performed in a hood under an inert atmosphere. The benefit of obtaining sodium amalgam is that it has the reducing potential of sodium, a larger surface area, and is more air stable.

Note: Potassium metal is more reactive than lithium and sodium. During prolonged storage, potassium can be oxidized to form a potassium superoxide (KO₂) coating; a yellow, shock sensitive peroxide that can explode on handling.

Lithium Aluminum Hydride

This is a strong reducing agent and water reactive substance. It is incompatible with oxidizing agents, air, water, ketone---based solvents (acetone/ethyl acetate/etc). Reacts violently with water and moisture in the air, producing hydrogen gas. Causes severe burns in all cases of exposure. Store under nitrogen. Light and heat sensitive. Wear sufficient

protective clothing/gloves/goggles at all times. For solutions in THF, keep nitrogen or argon balloon pressure, and draw up the appropriate amount with a glass syringe. When your reaction is complete, quench excess LAH by cooling solution to -78°C with acetone/dry ice and adding water drop wise slowly. This will produce precipitates of aluminum oxides, as well as LiOH. Saturated solutions of ammonium chloride can be used to aid in neutralization. Do NOT use acidic solutions to quench LAH.

In case of fire, use dry sand. Never use water or CO_2 fire extinguishers.

APPENDIX B

Transferring Liquid Pyrophoric Reagents outside the Inert Atmosphere of a Glovebox

If an inert atmosphere glovebox is not available and a pyrophoric reagent such as t-butyl lithium solution needs to be used, the solution can be transferred either by a syringe or double tipped needle (cannula). If the transfer takes place outside the glovebox the procedure must perform under an inert atmosphere using proper handling techniques such as the methods a-c outlined below.

The following list contains important key elements for transferring pyrophoric reagents outside the atmosphere of the glovebox.

- Do not work *alone* when handling pyrophoric reagents. Laboratory policy recommends that a colleague be present (the Buddy System) while working with hazardous chemicals. For example, if you become unable to respond to a situation due to an injury, the colleague can provide immediate assistance and make the calls to your emergency contact and building manager who can make the contact with your immediate manager.
- Fire resistant apparel (PPE) and safety glasses must be worn at all times when working with pyrophoric reagents. Fire-retardant laboratory coats are mandatory, and fire-retardant gloves are highly recommended. Note: Fire resistant gloves can also absorb hazardous liquids and consideration should be given to using the gloves in conjunction with appropriate chemically-resistant gloves. In other words, extreme caution must be exercised to mitigate skin exposure or spillage when handling pyrophoric reagents. Avoid wearing synthetic clothing while working with pyrophoric reagents as they do not provide the required protection. Natural fiber clothing tends to char instead of melt when exposed to flames or high temperatures and is preferable to synthetic fibers.
- Be prepared for the worst when handling pyrophoric reagents. These reagents can catch fire very easily when exposed to air. It is therefore advisable that extreme caution be used. Know the location of the safety shower, a blanket, a sand bucket, and how to use a fire extinguisher in case of emergency.
- If uncertain of how to handle pyrophoric reagents, users must consult a knowledgeable staff member prior to performing the experimental task, read the manufacturer's SDS and understand the technical information associated with handling the materials.
- Perform the work inside a fume hood with the sash down as far as possible for protection against chemical splashes and unexpected ignition events.
- Remove all other flammable materials from the hood to reduce the hazard in case of a fire.
- Secure the pyrophoric reagent bottle to a stand with a clamp before use to prevent it from tipping over. Controlling the bottle with one hand while handling the syringe with the other hand is strongly discouraged.
- Use a long needle to reach the solvent level. Do not invert the bottle as such action dislodges sediments that may clog the needle.
- Use a wide bore needle of 18-gauge or larger. The use of a smaller bore needle can slow down the transfer process and cause the needle to clog.

- Use a luer lock syringe with long needle when possible and be sure that the assembly does not leak. Make sure that the needle is secured in the luer-lock and does not separate from the barrel during the transfer.
- When disposable plastic syringes are used, it is highly recommended that the transfer is performed within the confinement of a glovebox. The syringes must be rinsed with non-reacting solvent, neutralized as appropriate, and disposed of safely at the conclusion of the transfer.
- All equipment, such as syringes and glassware, should be free of moisture and purged with an inert gas prior to use.
- Slowly add the pyrophoric reagent to the reaction vessel in a cooling bath to control reaction rate and heat dissipation.
- A container with residual pyrophoric reagents must never be opened. The containers must be rinsed with inert solvent and neutralized with adequate cooling prior to disposal. If uncertain of how to safely neutralize residual pyrophoric reagents, consult with an experienced staff member prior to neutralization.
- Products from pyrophoric reagents should be handled as potentially highly reactive materials.
- If a person is splashed with chemicals and catches fire, the use of the stop, drop, and roll method, safety shower, a fire blanket, or fire extinguisher (H₂O, CO₂ or A, B, C) are the most effective means of controlling clothing on fire. If a safety shower is available, keep the person under the shower for at least 15-20 minutes to make sure that all chemicals are washed away. Call your emergency contact.

When a syringe is used, the best practice for transferring a pyrophoric reagent is shown in method A below.

Note: When a syringe is used, extreme caution must be exercised to prevent the plunger from separating from the syringe. Use luer-lock, airtight syringes for the transfer. Avoid the transfer of a large volume (> 10 mL) from the pyrophoric reagent bottle when using syringes. For transferring larger volumes, used the double-tipped needle methods described below. At the conclusion of the transfer, the syringe needs to be clean to minimize the chance of the plunger from sticking/freezing in the barrel. The residue should be diluted with non-reacting solvent and remain under an inert atmosphere until rinsed with non-reacting solvent and the rinse and other residues are neutralized as appropriate.

1. Method A

Figure 1.1 shows the complete assembly of reagent transfer using an airtight glass syringe and a bubbler, for pressure release, under an inert atmosphere. The volume of reagent to be transferred is not to exceed 10 mL.

- Make sure that the syringe and the needle attached to the inert gas line needle, equipped with a bubbler and a shut-off valve, are purged prior to reagent transfer.

- Insert the needle, connected to the inert gas line (bubbler), through the septum into the Headspace above the reagent to maintain a slight positive pressure inside the Sure/Seal™ pyrophoric reagent bottle.
- Insert the long needle of a luer-lock, airtight, dry syringe through the septum into the reagent.
- Pull the plunger back slowly to fill the syringe with the required volume of reagent. Always keep the plunger in your grasp and avoid pulling back the plunger quickly as this action causes leaks and creates gas bubbles.
- Once the required volume is attained, slowly pull up the syringe needle from the pyrophoric reagent to the overhead space above the liquid.
- Pull the plunger up slowly and allow the inert gas to push the reagent trapped in the needle into the syringe.
- Shut the inert gas line off and slowly pull the needle out from the assembly to complete the transfer.
- At the conclusion of the transfer, the syringe and needle need to be rinsed with a non-reacting solvent and the residue quenched as appropriate under an inert atmosphere (see below).

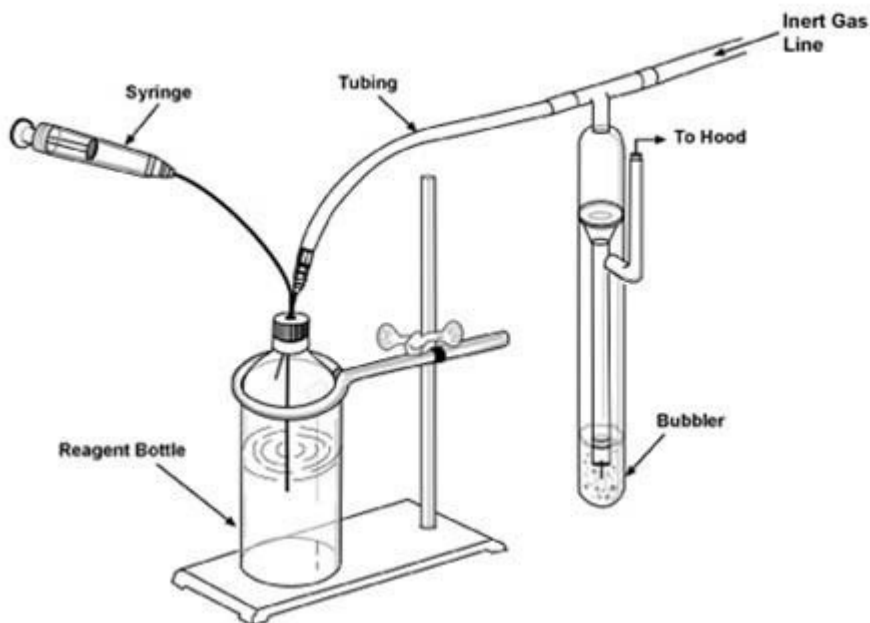


Figure 1.1. Syringe Transfer Assembly Equipped with an Inert Gas Line and a Bubbler during Reagent Transfer

When a Double-Tipped Needle is used, the Best Practices for transferring pyrophoric reagents are shown in Methods B or C.

Note: At the conclusion of the transfer, the needle needs to be clean to minimize clogging. The residue should be diluted with non-reacting solvent and remain under an inert atmosphere until the rinse and other residues are neutralized as appropriate.

2. Method B

Figure 2.1 shows the complete assembly for reagent transfer using a double-tipped needle under low pressure (< 1 PSI) from an inert gas line. Note that a bubbler is connected to the reaction vessel to avoid pressure build up in the assembly.

- Make sure that the glassware being used is dry, and the assembly is purged and maintained under an inert atmosphere prior to reagent transfer.
- Insert one end of the double-tipped needle through the septum into the reaction vessel or addition funnel in order to flush the needle.
- Insert the other end of the double-tipped needle through the septum of the reagent bottle into the headspace above the liquid.
- A needle attached to a very low-pressure inert gas line (< 1 PSI) is inserted through the septum into the headspace and kept above the solution of the Sure/Seal™ pyrophoric reagent bottle.
- Transfer the liquid from the reagent bottle into the reaction vessel or the addition funnel by pushing down the end of the double-tipped needle in the reagent bottle into the liquid. Note: At all times during the transfer, the reaction vessel must be vented through a mineral oil bubbler and kept under a positive pressure of an inert atmosphere to prevent air from entering the system.
- Once the required volume is transferred, pull up slowly on the end of the double-tipped needle in the pyrophoric reagent so that it is no longer in the liquid, but still through the septum and in the headspace above the liquid. Allow the inert gas to flow through the needle to push the trapped reagent in the needle to reaction vessel or the addition funnel.
- At the conclusion of the transfer, the double-tipped needle needs to be rinsed with non-reacting solvent and the residue quenched as appropriate under an inert atmosphere (see below)

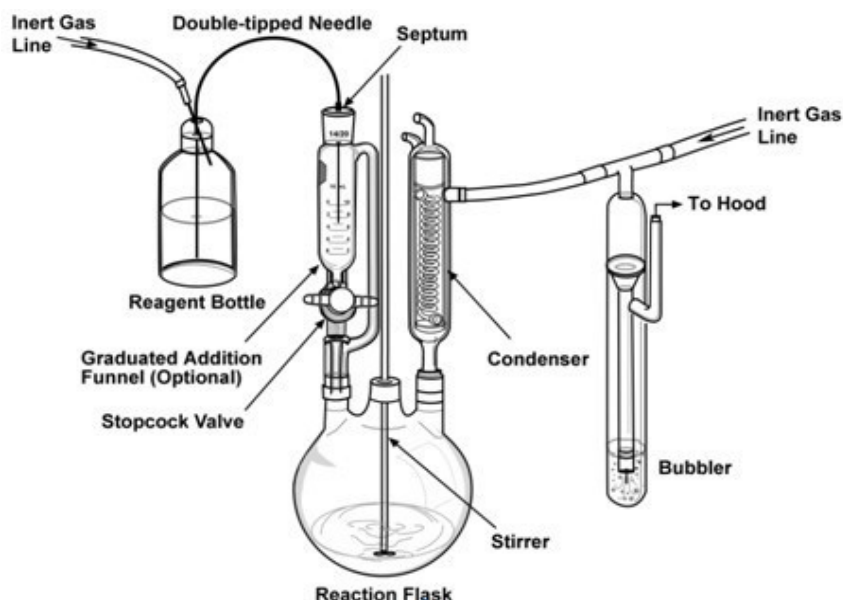


Figure 2.1 Double-Tipped Needle Assembly Equipped with a Bubbler and Kept Under an Inert Atmosphere during Reagent Transfer.

3. Method C:

Figure 3.1 shows the complete assembly for reagent transfer using the double-tipped needle under slight vacuum and connected to an inert gas line through a bubbler.

- Make sure that the reaction vessel being used is dry, and that the assembly is purged and maintained under an inert atmosphere prior to reagent transfer.
- Insert the double-tipped needle through the septum into the reaction vessel or addition funnel to purge the needle with inert gas.
- Insert the other end of the double-tipped needle into the head space above the liquid of the pyrophoric reagent bottle.
- Connect a vacuum line with a shutoff valve to the reaction vessel (as shown below).
- Insert the needle connected to the inert gas line (bubbler) through the septum into the headspace of the Sure/Seal™ reagent bottle to keep the space above the solution under a blanket of an inert gas.
- Make sure that the assembly is in proper configuration, and the inert gas is flowing through the bubbler.
- Push the end of the double-tipped needle in the reagent bottle down into the pyrophoric reagent.
- Apply a slight vacuum to the reaction vessel assembly by opening the vacuum valve slowly to transfer the desired volume from the reagent bottle to the reaction vessel through the double-tipped needle. Caution: The vacuum line will only need to be opened intermittently in order to transfer the pyrophoric reagent. High and continuous vacuum may allow air to enter the system through the bubbler.
- Once the required volume is transferred, pull up slowly on the end of the double-tipped

- needle in the pyrophoric reagent so that it is no longer in the liquid, but still through the septum and in the headspace above the liquid. Allow the inert gas to flow through the double-tipped needle to push the trapped reagent in the needle to reaction vessel.
- Close the vacuum valve and connect the reaction flask to an inert gas line.
- At the conclusion of the transfer, the double-tipped needle needs to be rinsed with non-reacting solvent and the residue quenched as appropriate under an inert atmosphere (see below).

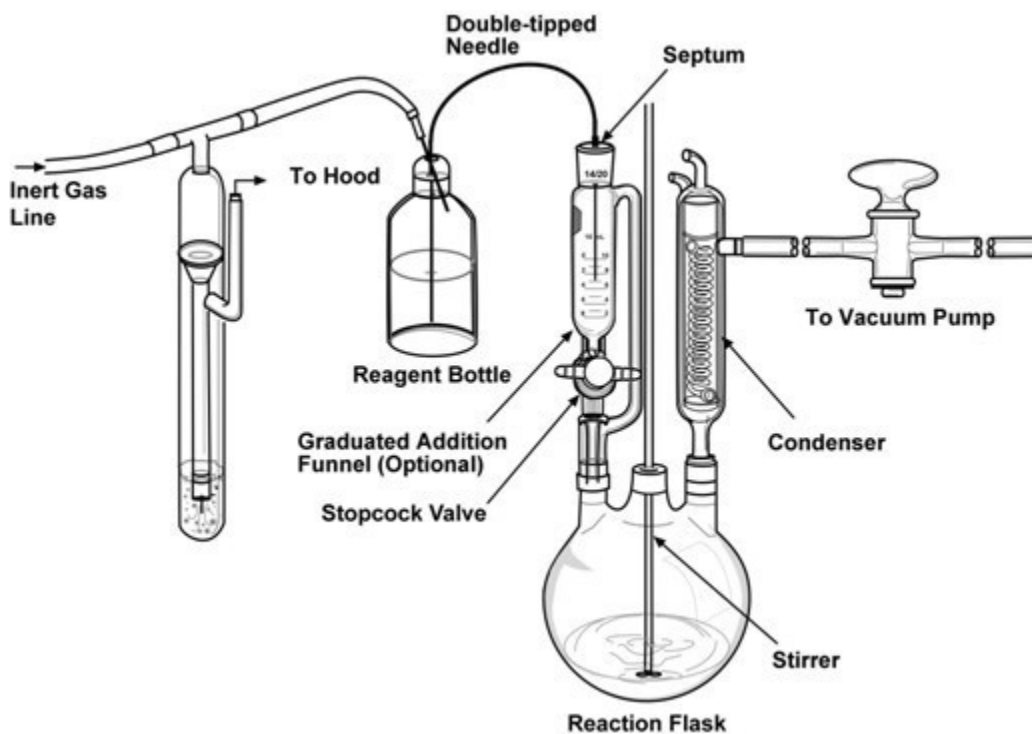


Figure 3.1. Double-Tipped Needle Assembly Equipped with a Vacuum Pump and Kept under an Inert Atmosphere during Reagent Transfer

Cleanup/Decontamination Procedure:

Needles and syringes used with pyrophoric reagents must be cleaned immediately to avoid clogging the needles and seizing the syringes. Draw hexanes into the syringe containing trace amounts of pyrophoric reagent several times and then discharge the diluted solution into isopropyl alcohol.

Storage Procedures:

Pyrophoric liquids must be stored under an inert atmosphere, with rigorous exclusion of air and moisture. Carefully ensure the integrity of storage containers before and after use. All storage containers must be clearly marked with the correct name and hazard warning.

Pyrophoric reagents can be handled and stored safely provided exposure to atmospheric oxygen

and moisture is avoided.

- Use and store minimal amounts of pyrophoric chemicals.
- Do not store pyrophoric chemicals with flammable materials or in a flammable liquid storage cabinet.
- A nitrogen-filled desiccator or glove box is a suitable storage location.
- If pyrophoric reagents are received in a specially designed shipping, storage or dispensing container, (such as the Aldrich Sure/Seal packaging system) ensure that the integrity of that container is maintained.
- Ensure that sufficient protective solvent, oil, kerosene, or inert gas remains in the container while the material is stored.
- NEVER return excess chemical to the original container. Small amounts of impurities introduced into the container may cause a fire or explosion.
- For storage of excess chemical, prepare a storage vessel in the following manner:
 - Dry a new empty container thoroughly
 - Select a septum that fits snugly into the neck of the vessel
 - Insert septum into neck in a way that prevents atmosphere from entering the clean, dry (or reagent filled) flask.
 - Insert a needle to inject inert gas and to maintain a blanket of dry, inert gas above the reactive reagent and quickly insert a second needle to vent the flask.
 - Once the vessel is fully purged with inert gas, remove the vent needle and add the reagent carefully, then remove the gas line.
 - For long-term storage, the septum should be secured with a copper wire.