Pyrophoric Transfers: Syringe/Cannula Guidance

Preparation

1. Review the following documents:
   a. Sharps Safety Guidelines
   b. [Procedures for Safe Use of Pyrophoric/Water-Reactive Reagents] www.csus.edu/college/natural-sciences-mathematics/internal/safety/_internal/_documents/procedures-for-safe-use-of-pyrophoric-water-reactive-reagents.pdf
   c. Lab-specific pyrophoric or water-reactive SOP
   d. SDS of the pyrophoric material to help determine hazard level (SDSs can be found at Risk & Safety Solutions)
2. Determine an appropriate quenching scheme for emergency spill control and quenching of excess reagent BEFORE starting work.
3. Inform co-worker(s) in the laboratory about your handling of the pyrophoric reagent and indicate your work area with a sign (e.g., “DANGER – Pyrophorics in use!”).
4. Find a lab partner to be with you while handling the substance. (Undergraduates MUST have their graduate student supervisor present.) This person must be within visual range and be capable of providing immediate assistance, if needed.
5. Obtain Met-L-X or dry sand and set it near your workspace.
6. Ensure your workplace is free of clutter and unnecessary flammable and combustible materials (e.g., Kimwipes®, flammable solvents).
7. **Additional PPE Required:**
   a. Safety eyewear
   b. Fitted FR lab coat
   c. Fitted inner FR gloves and outer neoprene gloves (Figure 1).
8. Select an appropriate receiver flask based on the total volume of the reaction and quenching; never fill a flask more than ½ full.

Figure 1. Properly (top) and improperly (bottom) fitted inner FR Gloves (yellow) and outer neoprene gloves (green

9. Pyrophoric reagents are usually stored in septum-sealed (or Sure/Seal™/AcroSeal™) containers to protect the reagents from oxygen and moisture. Pyrophoric reagent bottles are usually stored at low temperature.
    a. Prior to use, inspect the bottle seal for any cracks or signs of a compromised cap.
    b. **The bottle MUST be placed in a secondary safety containment while transporting from the storage location (e.g., refrigerator to the fume hood)** (Figure 2).
10. Secure the bottle with clamps or a ring stand in a fume hood and allow the reagent bottle to warm to room temperature before dispensing any material out of the bottle. Failure to do so may cause water condensation on the needle surface during transfer, which may initiate ignition. Do not attempt to heat the bottle.
11. Once the bottle has reached room temperature, wipe off any moisture found on the bottle, septa, or Sure/Seal™.

Figure 2 Secondary containment for transport of pyrophorics.

Needle and syringe technique

Syringes and needles can be used to safely transfer **10 mL or less** of pyrophoric reagents. If a larger volume will be transferred, use a double-tipped needle (cannula) and consult the PI.

1. Decide the appropriate length, needle gauge, and syringe size you will need. **NEVER fill a syringe to more than one half volume capacity**.
    a. Luer-lok ™ disposable Teflon™ syringes should be used instead of glass syringes, when available, due to a better seal.
    b. All non-disposable syringes and needles should be cleaned, dried in an oven, and left to cool to room temperature in a desiccator to avoid the absorption of water. Pyrophoric reagents are often in solutions of volatile solvents that will evaporate when in contact with hot surfaces. This produces positive pressure that may push the plunger out of the barrel and expel reagent toward the user. Secure the reagent bottle with a clamp (Figure 3).
2. **Secure the reagent bottle with a clamp** (Figure 3).
3. Apply positive pressure to the reagent bottle by attaching a balloon or inert gas line. Ensure that the needle tip is above the liquid level (i.e., headspace of the bottle), to prevent bubbling (Figure 3).

Figure 3 Secured reagent bottle with positive pressure

4. Remove all atmospheric air from the needle and syringe by purging them at least three times with inert gas from a dry septum-sealed reaction flask. During the last flush, fill the syringe with dry inert gas then quickly move and insert the needle into the Sure/Seal™ bottle and expel the dry inert gas into the head space (Figure 4).

Figure 4: Prepared syringe demonstrating the bend in the needle and keeping the inserted side secure.

5. Tightly hold the syringe plunger to avoid the build-up of positive pressure which can blow out the plunger. Lower the needle tip into the liquid and then very slowly fill the syringe by either gently pulling the plunger up or allowing the positive pressure from the bottle to fill the syringe. Never fill a syringe to more than ½ volume capacity. Pulling too hard or too fast can cause air bubbles to enter between the plunger and syringe body, causing ignition of material.
6. Carefully hold the needle end in the bottle to prevent it from slipping out of the septum and to prevent tearing the septa. Once the syringe is filled (less than ½ volume capacity), tip the syringe upside down, bending the long needle. Slowly force any excess reagent and bubbles back into the bottle to get the desired volume (Figure 5).

Figure 5: Hold plunger and force excess reagent back into the bottle.

7. Raise the needle tip above the liquid level and draw a plug of inert gas from the headspace into the needle. This keeps you from spilling and protects the liquid from exposure to air during transfer (Figure 6).

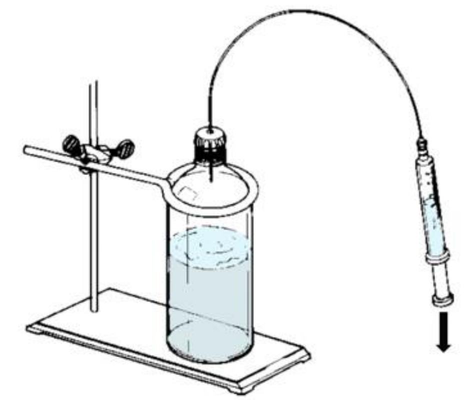

Figure 6: Drawing plug of inert gas for safe transfer.

8. Holding the syringe barrel carefully with one hand, slowly pull the needle out of the septum using your other hand. Then quickly insert the needle into a septum-sealed dry/inert gas purged receiver flask. The receiver flask should be selected based on the total volume of the reaction and quenching; never fill a flask more than ½ full. Don't delay – the transferring distance should be kept as short as possible to avoid prolonged exposure to atmosphere.
9. Slowly deliver the measured volume into the receiver flask (dropwise if necessary). The pressure inside the flask must be equalized by attaching an inert gas line or a balloon of inert gas (Figure 7).

Figure 7: Transfer of reagent into receiver flask.

10. When you are finished delivering the measured volume, immediately begin quenching and decontamination procedures for residual reagent in the syringe. Do not leave equipment contaminated, and clean-up/decontaminate the work area.
11. Remove gas lines from the reagent bottle and inspect th integrity of the septum bottle. If seal is deemed compromised, then dispose as hazardous waste. If the septum is in an acceptable condition, use a bottle carrier to return the pyrophoric reagent to storage.

Cannula technique

Before beginning a cannula transfer ensure you have picked an appropriate length and gauge cannula that can bend into a “U” shape. **Check for any pinhole leaks on the surface of the cannula**. Long cannula, over long-term use, can develop cracks or pinhole leaks on the surface. To check for leaks, elute acetone through the cannula and observe if any liquid is leaking out of the surface of the cannula.

1. Cannula should be cleaned, dried in an oven, and left to cool to room temperature in a desiccator to avoid the absorption of water upon cooling. Pyrophoric reagents are often in solution with volatile solvents which can ignite upon contact with hot surfaces.
2. Secure the reagent bottle with a clamp (Figure 3).
3. Apply positive pressure to the reagent bottle by attaching a balloon or inert gas line. Ensure that the needle tip is above the liquid level (in the headspace of the bottle), to prevent bubbling (Figure 3).
4. Insert one end of the cannula into the reagent bottle but keep the tip above the liquid level (in the headspace of the bottle). Inert gas will flush the cannula (Figure 8).

Figure 8: Insert the cannula into the reagent bottle.

5. Insert the other end of the cannula through the septum of an inert gas-flushed reaction vessel. The reaction vessel should be connected to an oil bubbler or balloon of inert gas to prevent over-pressurization. Keep the tip of the needle above the liquid level if there is any liquid in the reaction vessel. The receiving flask should be slightly lower than the dispensing bottle; this will help with transfer (Figure 9).

Figure 9: Arrangement of reagent bottle and receiving flask.

6. Push the needle into the liquid in the reagent bottle. Positive pressure from the inert gas source will push reagent through the cannula into the reaction vessel which is vented by an oil bubbler or balloon. If more positive pressure is needed:
    a. Use a gas line & bubbler to increase the gas flow rate.
    b. Use a balloon and gently squeeze the balloon.

    Make sure to control pressurization of the system, monitor bubbler and balloon size to avoid over- pressurization.

    Warning: over-pressurization can pop the septum and expose pyrophoric material to air. This can be mitigated by securing the septa with a copper wire.

7. Hold both ends of the cannula to make sure the tips remain inside the reagent bottle and reaction vessel during the transferring process. A stainless-steel cannula can easily slip out of the septa.
8. After transferring the desired quantity of the reagent, lift the tip of the needle inside the reagent bottle above the liquid level. Decrease (but don’t stop) the gas flow rate and allow inert gas to run through the system for a few minutes to flush out any remaining reagent.
9. Remove the needle from the reaction vessel first, then carefully and slowly remove the other tip from the reagent bottle. Before doing so, ensure that there is no more reagent dripping out of the cannula end.
10. When you are finished delivering the measured volume, immediately begin quenching and decontamination procedures. Do not leave equipment contaminated, and clean-up/decontaminate the work area.

Resources

Safe Handling of Cannulas and Needles in Chemistry Laboratories

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