Common Standard Operating Procedure

for work with

Chemical name/class:	**Quenching of pyrophoric substances and waste**
PI:	**Ken Henderson**
Date:	**01/07/2015**
Building:	**Stepan**
Room #:	**Laboratory 386**

1. **Circumstances of Use:**

Quenching and disposal of pyrophoric materials (i.e. organolithiums, alkali metal, metal hydrides, etc.)

2. **Potential Hazards:**

These reagents will ignite when exposed to moisture or air in an uncontrolled manner. Resulting fires may be smothered with sand or extinguished with an appropriate fire extinguisher (D-class).
Use appropriate personal protective equipment: safety glasses, standard laboratory gloves, and flame-resistant lab coat.

3. **Engineering Controls:**

Quenching process should be performed in a well-ventilated fumehood.

4. **Work Practice Controls:**

Remove flammable materials from the area prior to quenching.

Two methods are presented for the quenching and disposal of pyrophoric materials. The first method is recommended to quench unspent pyrophoric material. The second method concerns decontaminating glassware that has been used to handle pyrophoric materials.

Method 1)

The pyrophoric material to be quenched is initially transferred to a Schlenk flask, or round-bottom flask equipped with a vacuum adapter. Solids, such as Na metal, may be suspended in toluene or another high-boiling, non-reactive solvent to facilitate stirring during the quenching process. The flask is then placed under an inert atmosphere of N_2 (use Ar for Li metal) using a bubbler to vent those gasses evolved during the quenching process. The inert atmosphere prevents solvents from catching fire if the quenching mixture should become hot. The flask is cooled to 0 °C followed by slow addition of isopropanol. Isopropanol is used as an initial quenching agent because it reacts less vigorously than water does with reducing materials. After the addition of isopropanol to the quenching mixture no longer results in a vigorous reaction, a 1:1 mixture of isopropanol/water is added, and finally water is added. Once the pyrophoric material appears to have been entirely consumed, and the addition of water gives no sign of reaction, the quenching mixture should be warmed to 20 °C and stirred for at least 6 additional hours to ensure that the contents have completely reacted. The solution should be neutralized by the addition of citric or acetic acid while the mixture is still under an inert atmosphere. Upon neutralizing the mixture after the acid addition, the hydroxides formed should dissolve and the mixture should become homogenous. Extreme caution must be exercised if the mixture is not homogenous at the end of this process, as small amounts of pyrophoric material may still be present if they are coated in a layer of oxidized material. Because of this, it is best to stir the mixture until all solids dissolve. Once

the process is complete, the mixture may be disposed of as organic solvent waste. If toluene or other organic solvents have not been added to the flask, the mixture may be poured down the drain. (Note: If a mercury amalgam is quenched, the resulting waste must be treated as mercury-contaminated waste.) If you collect the liquid waste, do not tighten cap of waste container until positive it is no longer reacting. Recommend using a vented cap or leaving liquid waste in hood loosely caped for few days.

Method 2)

It may not be possible or practical to transfer the contents to a Schlenk flask for quenching of items that have become contaminated while handling pyrophoric materials. Some examples included methyllithium solution left in a syringe after use, sodium powder on a frit removed from the glovebox, or alkali metals that adhere to a metal spatula. As long as the amount of pyrophoric material is small, it may be quenched by the careful addition of ice. The material should be added to a large quantity of ice. Because the ice both quenches and cools the reaction, it is advisable to add all the ice at once. When using this method, organic solvents should never be added to the quenching-mixture, as they would provide a source of flammable material that could be ignited. Once the material appears quenched, the ice bath should be allowed to melt and the contents stirred until it warms to 20 °C. The solution should be neutralized by the addition of citric or acetic acid if it is to be disposed of in an appropriate aqueous waste container.

5. **Personal protective equipment (PPE):**

Minimum PPE would be safety glasses, lab coat (flame resistant), and standard laboratory gloves.

6. **Transportation and Storage:**

7. **Waste Disposal:**

If only isopropanol and water have been used (i.e., no toluene is not contained in the flask) the neutralized waste may be disposed of in an appropriate aqueous waste container

8. **Occupational injuries:**

As with any work-related injury, complete the work-related injury or illness report found at: http://chemistry.nd.edu/safety/supervisors-report-of-injury-to-the-health-center.pdf. If medical attention is needed, see http://chemistry.nd.edu/safety/procedures-for-injury-illness-or-incident.pdf.

9. **Emergency Procedures:**

In case of an emergency, contact the Police by calling 911 from a campus phone or 574-631-5555 from a cell phone.

10. **Training of personnel:**

All personnel are required to complete the General Lab Safety session thru RM&S. This session includes an introduction to general chemical safety. Furthermore, all personnel

shall read and adhere to this SOP.