Laboratory Safe Operating Procedure

Process: Setting up, filling, using, and quenching sodium stills

Prepared by: Christine Dunbar

Location: CHEM W5-54AA (toluene, benzene, ether), W5-50 (THF)

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Hazard Identification:

- The reaction between sodium and water produces hydrogen gas and is very exothermic, leading to fire and explosion hazards
- Sodium will react with atmospheric water and so must be kept in oil or under an inert atmosphere
- Sodium reacts vigourously with trace amounts of water in solvents; care must be taken while adding sodium to a still

Procedure:

Setting up a still:

1. All glassware must be dried in an oven (with Teflon stoppers removed)
2. The still set up should be put under argon immediately (see picture on next page); *note: if stills are set up in series, the argon should flow through the stills with the higher boiling solvents first to avoid contamination from other solvents*
3. Water hoses should be connected to the condenser; *note: if stills are set up in series, the water should go through the stills with the lower boiling solvents first*
4. The solvent should be pre-dried with molecular sieves prior to being added to the still
5. Adding sodium:
   a) Using long tweezers, transfer sodium cubes from storage container to beaker with hexanes
   b) Using a sodium knife, cut the sodium cubes into small pieces (each ~1/8th the size of the original cube)
   c) With tweezers, blot the small sodium pieces quickly on a piece of paper towel to remove the solvent and add to the still

d) Once finished adding sodium, add methanol to the beaker to quench any bits of sodium; put tweezers, knife, paper towel, and gloves in the beaker to quench any sodium left on these

6. Add a few scoops of benzophenone
7. Making sure the water to the condenser is on, set the still to reflux; *note: the low and reflux settings should be marked on the variac*
8. Once the solvent is a deep blue (see Appendix), the variac can be turned to the low setting; if after a few hours the solvent is still not deep blue, turn the variac off, wait for the solvent to cool, add more sodium or benzophenone, and reflux the solvent again

Filling a still:

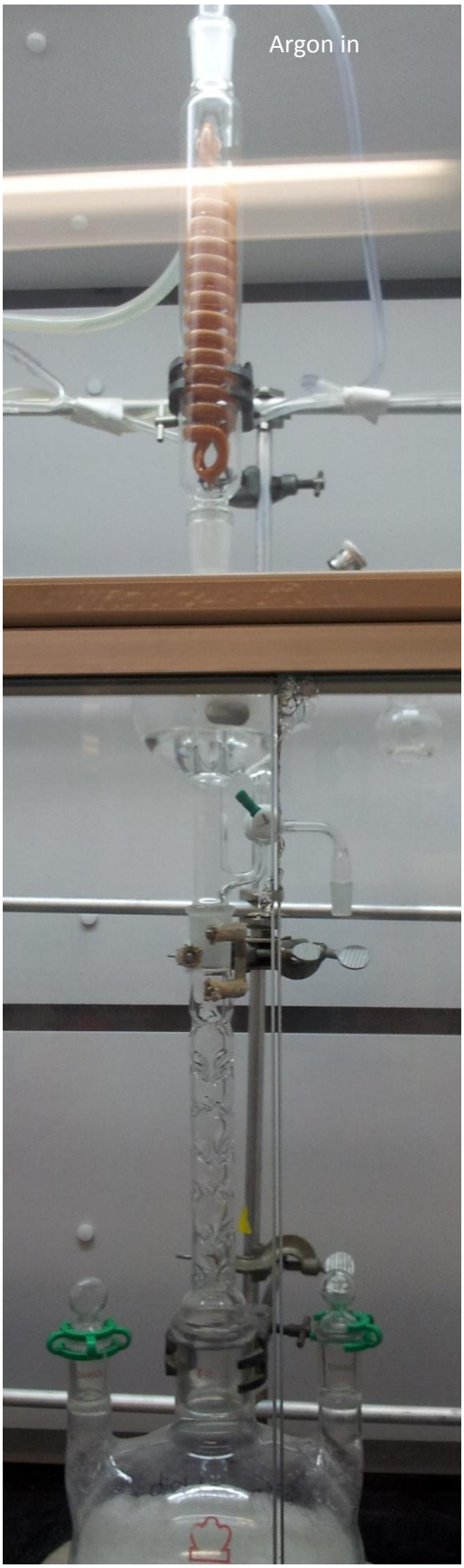
1. Turn variac off
2. Add solvent to an Erlenmeyer or beaker; add this to still; repeat until desired volume is met
3. Once solvent is added, set still to reflux; if solvent isn't blue after a few hours, more sodium or benzophenone should be added (see point 8 above)

Using a still:

1. Always make sure water is flowing through the condenser before setting the variac to the reflux position
2. Once the solvent is at reflux, set the stopcock on the bulb so that solvent collects
3. Once bulb is full or sufficient solvent has been distilled, turn the variac back to low
4. Let the still cool before collecting solvent
5. Flush syringe with argon before drawing up solvent
6. If air gets into the syringe while withdrawing solvent, remove the needle from the still and blow out the excess air; don't blow it into the still
7. Make sure stopcock is closed when finished; if you remove most of the solvent from the bulb, turn the still back to reflux and collect more in case someone else needs it

Quenching a still:

1. Turn the variac off and let the still cool



2. Set up a stir plate with a large ice bath in an appropriate fume hood
3. Separate the still pot from the Vigreux column, clamping a small round bottom flask to the bottom of the column (to maintain the rest of the system under argon)
4. Clamp the still pot in the ice bath over the stir plate and add a large stir bar and addition funnel
5. Once stirring successfully, begin adding *tert*-butanol dropwise, stopping if the reaction becomes too vigourous
6. Once there is little reaction with *tert*-butanol, *iso*-propanol can be added dropwise; methanol will be added similarly after the *iso*-propanol, followed by water
7. Once the sodium has been completely quenched, the solvent can be drained off into a waste container; wash out the still pot with soap and water, rinse with acetone, and put in the oven

Engineering Controls:

- Fume hood should be on and functioning
- Still should be under positive argon pressure; always have a full back up tank
- Variacs should be functioning and properly labelled

Administrative Controls:

- Still caretakers should review and sign this document, as well as get assistance from a senior member of the group if performing a procedure for the first time
- Never attempt this procedure when alone in the lab

Personal Protective Equipment:

- Safety glasses
- Gloves
- Lab coat

Emergency Response Procedures:

- Any person attempting this procedure should be aware of the location of the fire extinguisher, eye wash station, and safety shower. Additionally, this person should be trained in the use of the fire extinguisher or have someone nearby who is.

References:

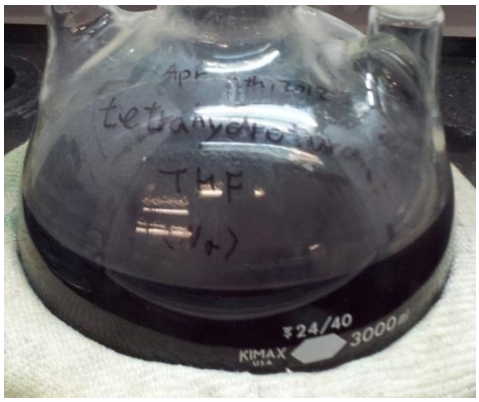
1. Sodium MSDS

Prepared by: Christine Dunbar **Date:** July 26, 2012

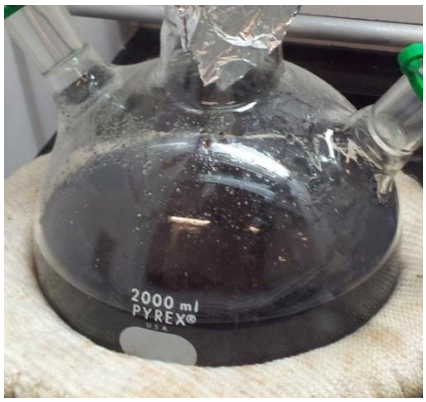
Approved by: Professor Frederick G. West **Date:** July 26, 2012

Appendix:

Good THF:

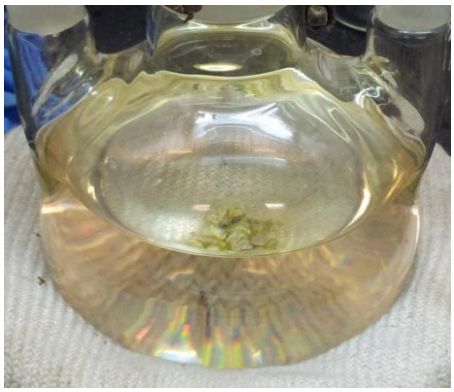


Good Toluene:



Good ether:

Good benzene:



Look for shiny bits of sodium