

Working Safely with Solvent Purification Systems



Health, Safety

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Outline



- Emergency Equipment
- Emergency Situations-Fire, flood etc.
- Personal Protective Equipment (PPE)
- Hazards
- Column Purification Systems
- Refluxing Solvent Stills
- Conclusion

Acronyms



PPE- Personal Protective Equipment

PI- Principal Investigator

HSR- Health, Safety and Risk

THF- Tetrahydrofuran



Training



- Lab Safety Training
- Fire Safety Training
- Spill Response Training
- Are you ready? Website
- WHMIS-2015
- Worker Health & Safety Awareness Training

Emergency Equipment



Emergency Equipment (cont)



- Presence of appropriate fire extinguisher
- Knowledge to use it, if required
- Location of spill kit along with adequate training
- First aid kit
- Emergency eye wash station
- Emergency shower

Fire Extinguishers



Fire



- Notify personnel in the immediate area
- Vacate the lab but remain available
- Active the manual fire alarm
- Call Protection Services at 5411 if fire alarm was not pulled
- Inform your Principal Investigator
- Inform the Health, Safety and Risk Manager or the Assistant
- Fill the incident report (online)

Flood



- Notify personnel in the immediate area
- Stop the source if possible without putting yourself in danger
- Vacate the lab, if required
- Call Protection Services at 5411
- Inform your Principal Investigator
- Inform the Health, Safety and Risk Manager or her Assistant
- Fill the incident report (online)

Personal Protective Equipment



- Safety glasses or goggles or face shield
- Lab coat (button it please)
- Long pants or other full leg coverings
- Appropriate chemical resistant and heat resistant gloves – check the material's rating!
- Closed shoes (front and back)
- Long hair must be tied
- Avoid loose clothing and jewelry

Hazards



- Solvents can often be flammable, carcinogenic, toxic, and reactive
- Heating solvents produces even more hazards
 - Fires, static discharges, explosions
 - Pressure buildup
 - Production of byproducts (acids, gases, etc.)
 - Violent reactions with water-sensitive materials
- Column solvent purification systems reduce hazards
- Refluxing stills should **only** be used when there is **no lower risk alternative**

Column Purification Systems



Column Purification Systems (cont.)



- Sometimes called commercial stills, push stills, or solvent systems
- Uses vacuum and pressure to push solvent
- High purity solvent is passed through columns containing a drying agent using an inert gas
- Solvent from these systems has acceptably low levels of water and oxygen.

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- No heat used = less risk (fire, pressure, byproducts, dangerous reactions)
- Better water removal from organic solvents:
 - 5-10 ppm water in THF with column system
 - 20-40 ppm water in THF with refluxing still
- Easier and faster to use than refluxing stills

Benefits to Column Systems (cont.)



- Often equipped with multiple different solvents with multiple dispensers at once
- Can be integrated with gloveboxes
- Isolated systems are easily grounded
- Large exchangeable solvent reservoirs don't need to be changed as often

Hazards Associated with Column Systems



- Larger solvent volumes present greater risk in case of fire, leak, or malfunction
- Higher pressures used mean leaks can be especially problematic
- Vapor leaks can also be dangerous
- Refilling and degassing reservoirs can expose workers to solvent and fumes

Safety Precautions



- Wear all appropriate PPE
- Ensure you have completed mandatory in-lab training and instruction in the use of each still you will be using
- Check system and surrounding area for fumes or leaks before use
- Dispense only the volume of solvent required
- Report any leaks or malfunctions to PI immediately and cease use of still

Refluxing Solvent Stills



Refluxing Solvent Stills (part 2)



- Refluxes and distills solvent to produce a purified, water-free (<50 ppm) solvent
- Performed under inert atmosphere to remove unwanted gases and prevent side reactions
- Designed to remove water, unwanted solvents, and other contaminants
- Drying agents are integrated into still to further remove water

Refluxing Solvent Stills

(part 3)



- Can use simple distillation...
 - Solvent condensed and collected directly into collection flask

- ...or fractional distillation
 - Vigreux column separates similar solvents and eliminates contaminant micro-droplets that are carried up with vapor

Refluxing Solvent Stills (part 4)



- Temporary or necessary?
- uOttawa recommends that these stills are for temporary use only
- Some research may require a still to be in place for an extended period of time due to dangers associated with repeated quenching
 - Talk to your PI and Health and Safety about possible options
- Where possible, obtain or share use of the safer column solvent systems

Refluxing Solvent Stills (part 5)



- Work in a functional fume hood
- Store flammables in a flammable solvent cabinet
- Use minimal volumes
- Label clearly and use **full form of name**
Tetrahydrofuran instead of THF
- Further in-lab training must be completed before you start working
- Maintain updated training records

Equipment



Equipment



- Distillation flask
- Thermometer (optional: water bath)
- Adapter tubes
- Condenser
- Solvent still head – facilitates refluxing and solvent dispensing with safety valve to prevent still from running dry
- Glassware clamps and joint clamps
- Heating mantle or hot plate

Equipment Safety



- Maintain sash at the 'marked' height
- Equipment generating heat must be equipped with shut-off device
- Clamps must be secured properly
- Use plastic clamps instead of metal twisters
- Do not let the solvent stills run unattended
- **No running over-night set-ups are allowed**

Safety Measures



- Before you begin, check
 - mantles
 - fabric
 - cables
- Check glassware for defects
- Remove air and water sensitive agents
- Do not allow solid material to accumulate in the round bottom flasks
- Talk to your PI and HSR about safety and operations

Safety Measures (part 2)



- Ensure that any personnel using the still is trained in its use and associated hazards
- Never add fresh solvent, drying agent/indicator when the still is hot
- Do not leave the set-up unattended
- Overheating can result in an explosion
- Pay attention to water supply: water shut-off can result in overheating, variable pressure can result in broken glassware, sodium explosion, and flooding

Safety Measures (part 3)



- Before deactivating the still, ensure personnel are adequately trained in the proper deactivation procedure
 - Different drying agents and reflux set-ups will require different procedures and have their own associated hazards
- Deactivate still under an inert atmosphere
- Face shields must be used when deactivating still
- **Do not** dismantle while the still is hot

Storage and Use



- Keep in mind that potassium is much more reactive than sodium
- Take extra precaution
 - while quenching
 - if set-up was left for a prolonged period
- Ethers (tetrahydrofuran, ether) can form explosive peroxides when exposed to air or when stored for an extended period of time

Conclusion



- Completion of training is mandatory
- Discuss usage with PI, Department Chair, or Health, Safety and Risk Manager
- Safety is everyone's responsibility

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