WAKE FOREST UNIVERSITY	**Standard Operating Procedure (SOP)**	

DISTILLATION AT ATMOSPHERIC PRESSURE

Effective Date:	**8/23/2013**	**Revised Date:**	**8/23/2013**

INTRODUCTION

- This SOP applies to distillation. Distillation is the traditional method of purifying a chemical liquid. It is also used to separate one component in a liquid mixture from another. Distillation in most laboratories involves refluxing volatile liquids at atmospheric or normal air pressure from a distilling flask through a "simple" or short path still head, or a longer "fractional" vertically held column, into a slightly downward angled condenser with a water-cooled jacket into receiving flasks.

DISTILLATION PROCEDURES

- Many different sizes and shapes of distillation heads and columns exist in chemical laboratories, but all adhere to the same basic principles of safe use. Trouble can arise mainly from excess pressure build-up due to too rapid heating and unsafe use of flammable solvents, resulting in fires.
- In general, common high-boiling or nontoxic solvents can be distilled on lab benches, with efficient condenser jacket water-cooling. Very low-boiling or more toxic compounds should be distilled only in a fume hood.
- Begin by attaching the water inlet hose on the lower water jacket inlet on the condensing column. The thermometer bulb should be placed just below the level of the roughly horizontal side arm of the distillation head. Just enough heat should be added to the distillation flask to raise the level of reflux only as high as the side arm. Additional heat is not needed.
- Do not completely fill the flask with liquid. A half-full or, at most, two-thirds full level is safer.
- Be sure all joints are tight, with grease if needed, and that the entire apparatus is well clamped and supported by ring stands. Fumes leaking through loose joints could come into contact with the heat source and cause a fire.
- Add boiling stones for atmospheric pressure distillations. More even boiling can be achieved with use of magnetic stir-bars. You should certainly use stirring for high boiling or very toxic compounds. Add boiling stones and stir bars to cool solutions, before you begin heating. Dropping cold boiling chips through a condenser into hot solutions will result in very rapid boiling and has been known to cause boil-over of liquid through the top of the condenser.
- Ordinarily, you should raise the heating mantel on a platform, or "lab jack" so that you may quickly remove the source of heat if the liquid "bumps" uncontrollably or loss of vapor occurs through the top of the condenser. Heat sources ordinarily used in undergraduate organic labs include bare corning stirrer/hotplates, on low thermostat settings of about "3", with distilling flasks just touching or just above the surface and surrounded in a funnel of aluminum foil. Research labs make use of various types of heat sources, including heating mantels attached to variable transformers and oil baths on

hotplates. When using oil baths, do not overheat the oil.

- The receiving flask should be of such design as to efficiently receive the condensed liquid through the receiving adapter. Vacuum adapters can be used for water-aspirator vacuum distillations or inert atmosphere applications. Gas cylinders of nitrogen or argon are commonly attached via hoses to reaction stills with appropriate regulators and fittings.
- Never heat a closed vessel. Always have some means of venting heated gasses through distillation setups. One could also attach a hose to the vacuum adapter and direct it into a hood for more effective removal of any uncondensed vapors which may escape from normal atmospheric pressure distillation. Purging of distillation apparatus with inert gasses while distilling is sometimes employed in research laboratories. Make sure to include some sort of "safety valve".
- Surround the receiving flask in an ice bath to further condense very volatile organic compounds.
- Make sure coolant is running through the condenser before you start heating the liquid. The rate of distillation, as determined by the number of condensed drops falling into the receiving flasks, should be relatively low, a few drops per second.
- Potentially reactive or explosive solvents should be distilled behind transparent explosion shields
- Refill liquid in the receiving flask or disassemble the entire setup only when the glassware has cooled down from the previous distillation.

POTENTIAL HAZARDS

- Excess pressure build-up due to too rapid heating and unsafe use of flammable solvents, may result in fires.
- Very low-boiling or more toxic compounds should be distilled only in a fume hood.
- Fumes leaking through loose joints could come into contact with the heat source and cause a fire.
- Dropping cold boiling chips through a condenser into hot solutions will result in very rapid boiling and has been known to cause boil-over of liquid through the top of the condenser.
- When using oil baths, do not overheat the oil.
- Never heat a closed vessel.
- Do not distill to dryness or "superheating" of the flask will occur, either cracking the glass or leaving a "tarry" residue which may be very flammable or even explosive.
- Potentially reactive or explosive solvents should be distilled behind transparent explosion shields

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HEALTH HAZARDS

Check SDS for each chemical to be distilled prior to procedure for information on specific health hazards.

PERSONAL PROTECTIVE EQUIPMENT

EYE PROTECTION

- Safety glasses, goggles or face shields shall be worn during DISTILLATION operations.
- Ordinary (street) prescription glasses do not provide adequate protection. Adequate safety glasses must meet the requirements of the Practice for Occupational Education Eye and Face Protection (ANSI Z87.1-1989) and must be equipped with side shields.

HAND PROTECTION

- Use disposable nitrile gloves when working with chemicals. Check chemical compatibility chart for breakthrough time when using
- Laboratory personnel should thoroughly wash hands with soap and water before and immediately upon removal of gloves.

LAB COATS, ETC.

- Button lab coats, closed toed shoes, long pants and long sleeved clothing shall be worn when PERFORMING DISTILLATIONS. Protective clothing shall be worn to prevent any possibility of skin contact with CHEMICALS DURING DISTILLATION.

EMERGENCY PROCEDURES

Emergency Numbers:

Fire and Medical Emergencies	x5911 (911 on cell phone)
Environmental Health and Safety	x3427
Hillcrest Urgent Care (employees)	336-760-8999
Student Health (students only)	x5218
Poison Control	800-222-1222

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FIRST AID

1. If inhaled: If breathed in, move person into fresh air. If not breathing, give artificial respiration. Call x5911 for medical assistance.
2. In case of skin contact: Take off contaminated clothing and shoes immediately. Wash off in safety shower for at least 15 minutes. Call x5911 for medical assistance.
3. In case of eye contact: Rinse thoroughly with plenty of water at eyewash for at least 15 minutes and call x5911 for medical assistance.
4. If swallowed: Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Call x5911 for medical assistance.
5. Call x5911 and describe the extent of injuries.
6. Report all accidental exposures to EHS and Human Resources (employees) or Student Health (students).
7. Complete an online injury/illness report if there is an over-exposure to the chemical or if there is an accident involving the chemical.

SPILL AND ACCIDENT PROCEDURES

SPILL QUANTITY	PROPER SPILL RESPONSE
Spill less than 500 mL	**Contact Environmental Health and Safety (x3427) and clean up spill using spill kit. Avoid breathing vapors.**
Spill greater than 500 mL	**Do not attempt to clean up spill. Leave the area and immediately report to WFU Police (x5911) and EHS (x3427).**