

# Standard Operating Procedure for Distillation of Organic Solvents

## I. General Statement of Coverage

Purification and recovery of organic solvents by distillation is a common laboratory operation. Instructions and descriptions for the distillations of hexanes, tetrahydrofuran (THF), diethyl ether, dimethoxyethane, benzene, toluene and dichloromethane are included in this SOP.

## II. Hazard Assessment

A Job Hazard Assessment should be performed for work involving distillations of organic liquids and should thoroughly address the potential combination of water and electricity (shock hazards), the temperature and temperature control of the heating mantle and contents (burn and fire hazards), chemical toxicity and reactivity of the solvents and sodium reducing agent, cleanup and disposal of still pot residue, solvent storage, and required Personal Protective Equipment (PPE).

## III. Resources

### A. Available Training

See the Laboratory Supervisor for specific training references.

### B. Literature References

1. Department of Chemistry Safety Handbook
2. *Vogel's Textbook of Practical Organic Chemistry*, 5th Ed.; B.S. Furnis, et.al. Eds.; Longman: Singapore, 1989.
3. *Purification of Laboratory Chemicals*, 2nd Ed.; D.D. Perrin, W.L.F. Amarego, D.R. Perrin; Pergamon: Oxford, 1980.
4. *Organic Solvents, Physical Properties and Methods of Purification*, 3rd Ed.; J.A. Riddick, W.B. Bunger; Wiley-Interscience: New York, 1970.
5. *Prudent Practices for Handling Hazardous Chemicals in the Laboratory*; National Research Council, National Academy Press: Washington, D.C., 1981.

## IV. Chemical Storage

### A. Special Storage

1. The storage of flammable and combustible liquids in a laboratory, shop or building area must be kept to the minimum needed for research and/or operations. Not more than 10 gallons of flammable liquids can be stored outside of an approved storage cabinet. Flammable-liquids storage cabinets are not designed for the storage of acids, bases, or compressed gases. Approved storage cabinets are NOT required to be vented. Ventilation is recommended for the storage of large quantities of Class 1A flammable liquids (such as Diethyl Ether or Pentane) or malodorous compounds such as mercaptans.

2. Some flammable liquids, such as low molecular weight ethers and vinyl compounds, THF, and Dioxane, slowly form peroxides upon exposure to air and sunlight. This may necessitate periodic testing for peroxides.

## **B. Gas Cylinders**

Gas cylinders must be secured while in use or in storage (empty or full). They should be stored with the valve cap secured.

## **V. Personal Protective and Emergency Equipment**

### **A. Eye and Face Protection**

At a minimum, safety glasses with permanently attached top and side shields must be worn in the laboratory. These glasses, however, do NOT protect against splash hazards. When performing a hazardous activity such as adding sodium to or neutralizing sodium in the distillation pot, a face shield must be worn in addition to the safety glasses OR switch to chemical splash goggles (with shielded ventilation ports).

### **B. Gloves**

Appropriate gloves should be worn when handling organic solvents.

### **C. Protective Clothing**

Lab coats, closed toed shoes, and long sleeved clothing should be worn when performing distillations. Additional protective clothing, such as aprons or full-length arm protection, should be worn if the possibility of skin contact is likely.

### **D. Hearing Protection**

The use of hearing protection requires monitoring and training. See the Safety Coordinator for details.

### **E. Respirators**

The use of respirators requires medical certification, fit testing, and training. See the Safety Coordinator for details.

### **F. Eye Wash**

Where the eyes or body of any person may be exposed to organic solvents or vapors, suitable facilities for quick drenching or flushing of the eyes and body shall be provided within, or near, the work area for immediate emergency use. Bottle type eyewash stations are not acceptable.

## **G. Safety Showers**

A safety shower should be available and functioning as specified by ANSI Z358.1.

## **H. Fire Extinguishers**

All laboratories must contain at least one Carbon Dioxide (Type B-C) or Dry Chemical (Type A-B-C) fire extinguisher. A dry-powder fire extinguisher should be readily available when working with sodium in charging or cleaning the still pot.

## **VI. Controls**

### **A. Designated Areas**

Some flammable liquids such as Benzene, Methyl Mercaptan, and Carbon Disulfide, require Designated Areas.

### **B. Chemical Fume Hoods**

When possible, distillations involving greater than 500 ml of organic solvents should be carried out in a fume hood.

### **C. Safety Shielding**

Safety shielding is required any time there is a risk of explosion, splash hazard or a highly exothermic or unstable reaction.

### **D. Special Ventilation**

Fume hoods provide the best protection against exposure to flammable liquids in the laboratory and are the preferred ventilation control device. Always attempt to handle large quantities of flammable liquids in a fume hood. If your research does not permit the handling of large quantities of flammable liquids in your fume hood, contact the Chemical Hygiene Officer or the Division of Environmental Health and Safety to review the adequacy of all special ventilation.

### **E. Vacuum Protection**

Mechanical vacuum pumps and the "House Vacuum System" must be protected using cold traps and, where appropriate, filtered to prevent particulate release. The exhaust for the pumps must be vented into an exhaust hood. Vacuum pumps should be rated for use with flammable liquids.

## VII. Specific Procedures

Solvents are distilled under a nitrogen atmosphere from a distillation flask containing either metallic sodium and benzophenone or calcium hydride. The refluxing solvent travels up the vacuum-insulated distillation column and condenses on the water-cooled condenser. The distillate collects at the top of the column in the distillation trap. The purified solvent is removed via syringe through the septum at the top or through the stopcock at the bottom of the distillation trap. Each of the stills is connected in series by water lines for the condensers and nitrogen lines for the inert atmosphere.

### A. Setting-Up Solvent Stills

1. Set-Up Diagram (See Figure 1)
2. Charging and Using Stills
  - a. THF should be pre-dried for several days over potassium hydroxide (KOH).
  - b. The still is cooled down to room temperature. Ensure the variac is turned off.
  - c. The solvent is poured into the round bottom flask which serves as the still pot to no more than 2/3 of its capacity.
  - d. Add reducing and drying agents to the solvent.

### Hexanes, Tetrahydrofuran, Dimethyl Ether, Dimethoxyethane, Benzene

i. In a fume hood, a small amount of metallic sodium is cut into small pieces; the sodium quickly rinsed with a minimum amount of hexanes to remove the oil, the sodium is added to the solvent in the distillation flask. (**Note:** Sodium metal and hexanes is a potential fire hazard. Ensure that a dry-powder fire extinguisher is near by and accessible before you begin working with sodium.)

ii. Add 250 mg of benzophenone to the distillation flask, establish the inert atmosphere, and bring the solution to reflux for several hours to allow the sodium metal a chance to dry the solvent. When dry, the solution will have a deep blue to blue-green color.

iii. If, after refluxing for several hours, the deep blue color does not develop, repeat steps i and ii.

iv. Tetrahydrofuran and diethyl ether form peroxides. Do not distill without adding a reducing agent such as sodium.

### Toluene and Dichloromethane

A small amount of calcium hydride is added and the solution is refluxed under a nitrogen atmosphere to dry the solvent.

## **B. Maintenance and Inspections**

### 1. Daily:

#### Nitrogen:

- Check tank pressure gauge and replace tank if below 100 psig.
- Check flow rate at the flow meter and adjust to 2 on the gauge.
- Check for flow at the bubbler and check bubbler level.

#### Water:

- Check flow rate.
- Check water lines and connections for weak spots and leaks.

#### Still Pot:

- Check solvent level is at least 1/3 full.
- Check solvent color.

#### Night or Finished:

- Turn down variac to 10.
- Empty distillation trap.

### 2. Monthly:

- Check nitrogen lines.
- Change rubber septa on distillation trap.

### 3. Quarterly:

- Check electrical wiring and variacs.
- Check glassware integrity.

### 4. Cleaning:

- Do as needed.

## **C. Regenerating/Cleaning**

### **Hexanes, Tetrahydrofuran, Dimethyl Ether, Dimethoxyethane, Benzene**

i. After the solvent has cooled to room temperature, the distillation flask is moved to a fume hood.

ii. The residual solvent is carefully quenched with small amounts of 2-propanol added under a blanket of nitrogen over several hours and allowed to stand overnight.

iii. The residual solvent is then carefully quenched with small amounts of absolute methanol over several hours until all the sodium metal is destroyed. Now carefully add excess water under nitrogen and properly dispose of the contents.

### **Toluene and Dichloromethane**

i. After the solvent still has cooled to room temperature, move the distillation flask into a fume hood.

ii. The residual solvent is then carefully quenched with small amounts of absolute methanol added under a blanket of nitrogen over several hours and allowed to stand overnight.

iii. The residual solvent is then carefully quenched with small amounts of water under nitrogen and properly disposed of.

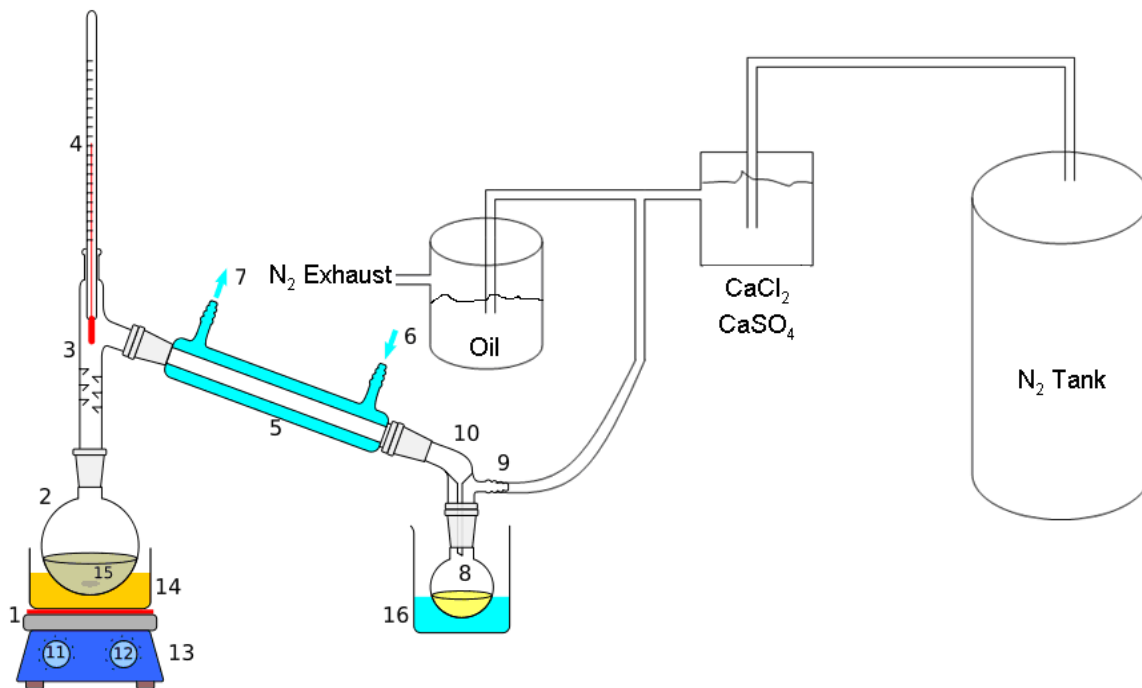


Figure 1: Distillation Setup

**1:** Heat source

**2:** Still pot

**3:** Still head

**4:** Thermometer

**5:** Condenser

**6:** Cooling water in

**7:** Cooling water out

**8:** Distillate/receiving flask

**9:** Vacuum/gas inlet

**10:** Still receiver

**11:** Heat control

**12:** Stirrer speed control

**13:** Stirrer/heat plate

**14:** Heating (Oil/sand) bath

**15:** Stirrer bar/anti-bumping granules

**16:** Cooling bath.