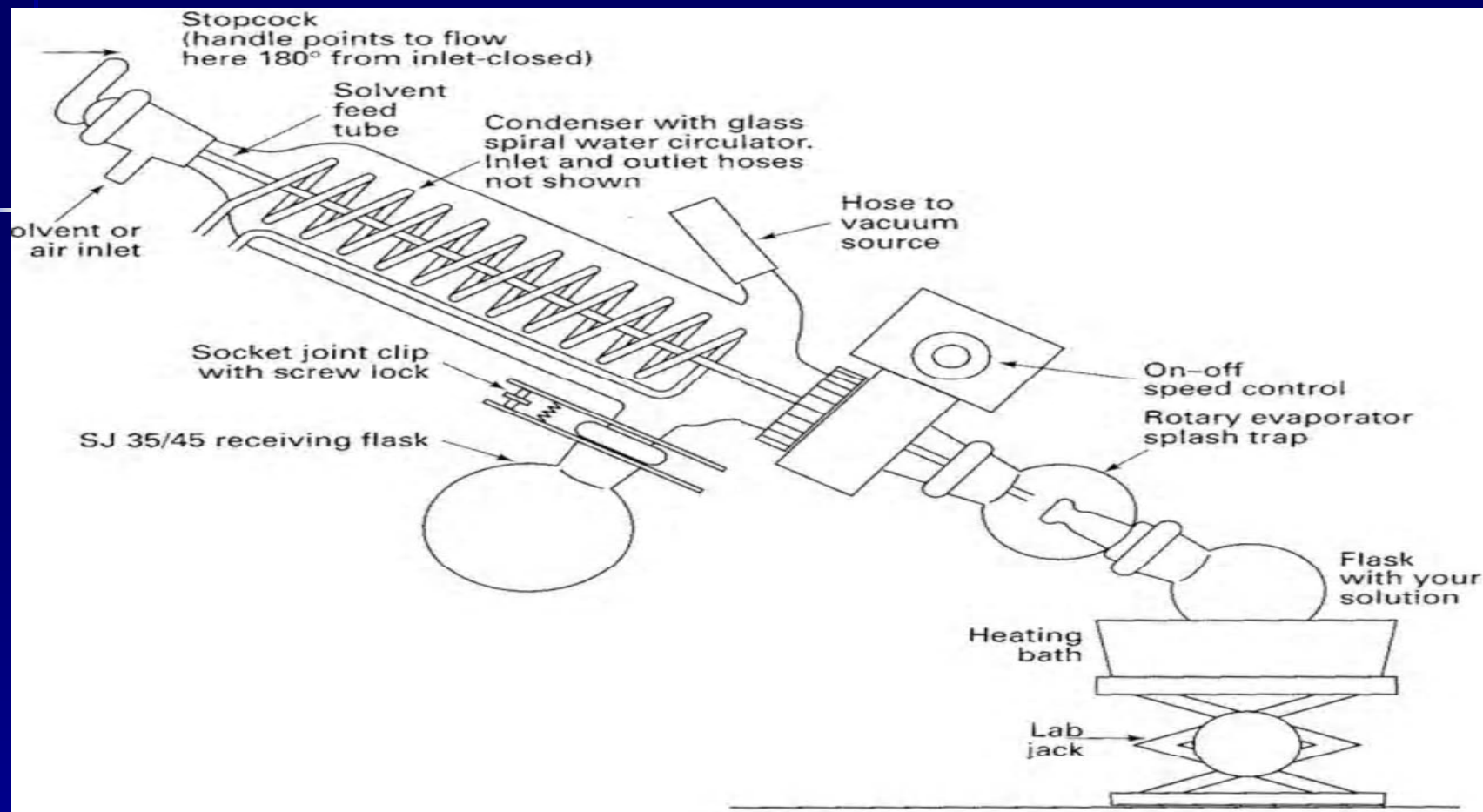


Standard Operating Procedure for Roto-evaporators



What is Rotoevaporator?



- Rotary evaporators (also called "rotavaps" in lab slang) are used to remove solvents from reaction mixtures and can accommodate volumes as large as several liters. A typical rotary evaporator has a water bath that can be heated in either a metal container or crystallization dish. This keeps the solvent from freezing during the evaporation process. The solvent is removed under vacuum, is trapped by a condenser and is collected for easy reuse or disposal

General rules for usage of a rotary evaporator

1. Empty and then replace the solvent collection flask on the unit. SAFETY FIRST! You don't want to accidentally mix incompatible chemicals.
2. Place the round-bottomed flask with the solution on the rotary evaporator. Most people use a bump trap to prevent their material from accidentally splashing into the condenser (and being contaminated). It is highly advisable to start with a clean bump bulb!
3. A metal or Keck clip is used to secure the round-bottomed flask and the bump trap. The green one shown fits 24/40 ground glass joints. Similar blue clips fit 19/22 joints and the yellow ones fit 14/20 joints, which you will most likely be using.
4. The dial on the motor is used for speed of the flask rotation. A typical roto-evap uses a variable speed sparkless induction motor that spins at 0- 220 rpm and provides high constant torque.
5. The aspirator vacuum is turned on. On most models, the vacuum on/off control is managed by turning a stopcock at the top of the condenser. This stopcock is later also used to vent the setup.



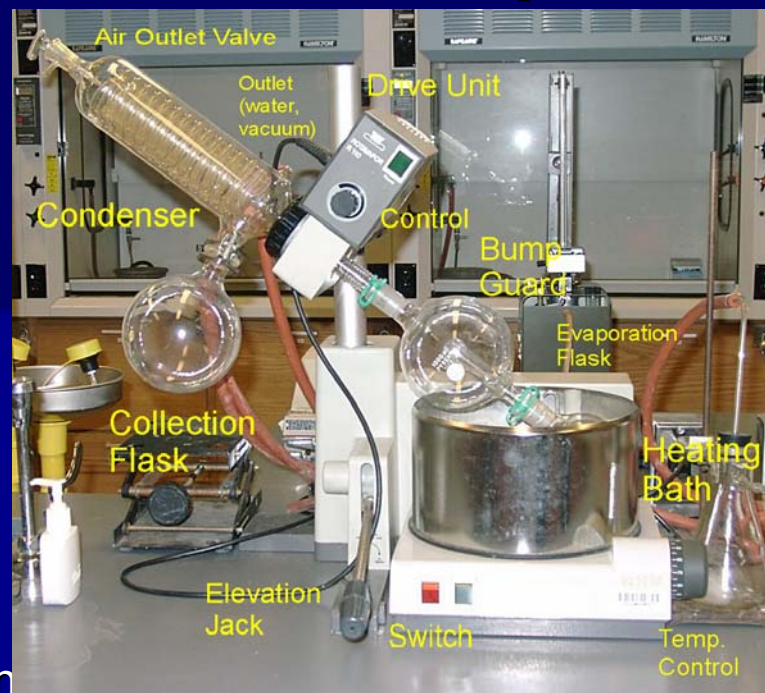
- 6 The flask is lowered into the water bath or the water bath raised to immerse the flask in the warm water. At this point, the flask should not be more than half filled. (On most models, a convenient handle (with height locking mechanism) moves the entire condenser/motor/flask assembly up and down. Often the tilt of the condenser assembly can also be adjusted. The water bath temperature should not exceed the boiling point of the solvent!! For small amounts of common solvents you do not need to turn on the bath heater.
- 7 Once all the solvent has evaporated (no solvent condenses in the trap anymore), the vacuum is released (by allowing air to bleed into the setup), the flask raised out of the water bath and the rotation turned off. The Keck clip is removed, and then can flask with the remaining material.
- 8 The bump trap has to be cleaned and the receiving flask is emptied upon completion of the evaporation.

Are There Dangers Associated with Rotovaps

Proper operating procedure should always be followed

- Rotate the flask
- Slowly apply a vacuum
- Use a lukewarm water bath to heat

There are dangers associated even with simple operations such as evaporation. These include implosions resulting from use of glassware that contains flaws, such as star-cracks. Explosions may occur from concentrating unstable impurities during evaporation, for example when rotavapping an ethereal solution containing peroxides. This can also occur when taking certain unstable compounds, such as organic azides and acetylides, nitro-containing compounds, molecules with strain energy, etc. to dryness



There are 4 areas of concern for safe rotovap operation

- Implosion
- Explosion
- Hazardous waste
- Hoses are tied down/good shape to preventing water flood

Implosion

When a vacuum exists within glassware there is a risk of implosion.

- Use only glassware that is free from cracks and imperfections
- Use heavy-walled vacuum flasks
- Use a shatter-proof collection bulb
- Use safety netting around the condenser



Explosion

You must investigate the properties of the compounds you are working with.

- Compounds such as azides and peroxides are thermally unstable and should not be isolated via rotovap
Ethereal solvents can form peroxides when exposed to air and light overtime. When you use and then evaporate these solvents, the residue is enriched in peroxide, a violent explosion can occur if taken to dryness
- Always date a freshly open can of ether
- Do not use ether that has been opened more than 3 months

Hazardous Waste

- Very large amounts of organic solvent vapor can be generated from a rotovap
- The condenser should always be cooled



General Safety Precautions

- Personal protective equipment, such as safety glasses or chemical goggles, face shields should be used to protect against the hazards of vacuum procedures.
- Use heavy-wall rubber tubing only. Thin-wall rubber tubing and plastic tubing are not suitable because they collapse leaving a partially evacuated closed system.
- Although glass vessels are frequently used in vacuum systems, they can explode or implode violently, either spontaneously from the strain or from an accidental blow. Therefore, pressure and vacuum operations in glass vessels should be conducted behind adequate shielding. Glass vessels should be designed for the operation intended, and should be visually checked for star cracks, scratches, or etching marks before each use.
- Flasks with volumes of 1 liter or larger and the cooling condenser must be enclosed in tape or plastic mesh to restrain fragments in case of implosion.
- Glass dewars should be fully wound in tape or enclosed in a metal container.
- The rotation speed and application of vacuum should be done gradually when using a rotary evaporator.
- Do not hold onto the sample whilst it is revolving: failure of the flask could result in serious lacerations to the hands.
- Slowly admit air back into the apparatus after evaporation of the solvent is complete.
- Clean the apparatus after use. Do not mix halogenated and non-halogenated solvents in the receiver.