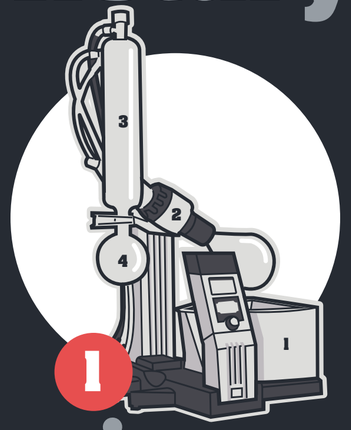
Rotary Evaporation Best Practices



Rotary Evaporators

Rotary evaporators are common laboratory instruments, found in virtually every organic laboratory, and are used to remove or isolate components of reaction mixtures based on differences in their boiling points. This is often done after a separation or extraction process. During rotary evaporation, the solvent is removed under vacuum and then trapped by the condenser and collected for reuse or disposal.

Most rotary evaporators have 4 major components:

1. Heating bath
2. Rotary Drive
3. Condenser
4. Solvent Collection Flask

Set-up

1. Select a flask that accommodates roughly twice the starting volume
2. Select a bump trap to reduce the likelihood of sample loss with bumping or violent boiling. Better yet, consider using a vacuum pump system. *(see "To Avoid Bumping" in Tips & Tricks)*
3. Select and attach a vacuum pump capable of reaching your desired vacuum level and that is compatible with your specific solvent vapors.
4. Select the proper temperature for your water bath–lower temperatures make for a slower process but reduce the likelihood of bumping or damage to your sample due to overheating.

Solvent	Boiling Point (760 torr)	Freezing Point (40 torr)
Acetonitrile	81.8 °C	7.7 °C
Diethyl Ether	34.6 °C	-27.7°C
Ethanol	78.4 °C	19 °C
Ethyl Acetate	77.1 °C	9.1 °C
Hexane	68.7 °C	-2.3 °C
Heptane	98.4 °C	22.3 °C
Methanol	64.7 °C	5.0 °C
Water	100 °C	0 °C

Commencing a Rotary Evaporation

1. Turn on the chiller and allow the temperature to reach set point.
2. Turn on the water bath. Allow bath to reach set point prior to beginning evaporation.
3. Secure your evaporation flask with Keck clamps. Unless you are very confident, don't rely on the vacuum to hold your flask.
4. Begin flask rotation. It should spin fast enough to create an even coating on the inner surface of the flask.
5. Turn on the vacuum pump, close the stopcock on the condenser, and allow the sample to spin under vacuum for approximately 1 minute. The sample will likely start to boil–if the sample begins to bump or boil violently, vent the system and adjust the vacuum set point.
6. Once boiling has ceased, and solvent is collecting in the solvent trap, lower the flask about halfway into the heating bath.
7. The process is best controlled by adjusting the vacuum, due to the pump's ability to respond quickly and accurately.

Halting a Rotary Evaporation

1. Raise the flask from the heating bath.
2. Open the stopcock on the condenser to vent the system to the atmosphere.
3. Turn off rotation.
4. Turn off the vacuum supply.
5. Remove the evaporation flask; remove and empty the solvent collection flask.
6. Turn off water bath and chiller (unless you are starting a new evaporation).

TIPS & TRICKS

To Avoid Bumping

- Don't overfill your flasks. Less than 50% full is best.
- Faster spin rates often help to prevent bumping .
- The difference in bath temperature and coolant temperature should be ~40 - 60°C.
- Use moderate bath temperature –too high will cause excessively fast evaporation and can damage some samples.
- Consider a vacuum system that allows for accurate adjustment of system pressures, thus reducing the likelihood of bumping and increasing solvent recovery. Better yet, use a vacuum system that automatically senses solvent vapor pressures, which eliminates bumping by not overshooting the optimum vacuum level.

To Thoroughly Dry a Sample

- Once you have removed the majority of a solvent, empty the collection flask. Reattach and continue with the evaporation.

To Prevent Implosion

- Inspect all your glassware prior to use.
- Don't use round-bottomed flasks with visible cracks or star-cracks.

To Manage Dangerous Solvents/ Reagents

- Acids and chlorinated solvents are dangerous when inhaled. Be aware that for highly volatile liquids not all of the solvent removed may be condensed in the traps. Try venting through a fume hood if possible or attach a suitable scrubber.
- Be aware of potentially reactive solvent combinations. Thionyl chloride is a good example. Rather than dealing with a stream of HCl and sulfur dioxide gas when it reacts with water–search for a better alternative for this extraction.
- Remote control of rotary evaporator and vacuum pump operation provides an additional level of safety when using a fume hood with closed sash.

To Save the Environment

- Avoid using a water aspirator as your vacuum source. Harmful solvent vapors can be pulled down the drain with the water. In addition, the continuous full-stream wastes a significant amount of water.
- Oil-using vacuum pumps, such as rotary vane, require the disposal of solvent-contaminated oil.
- Select an oil-free vacuum pump as the most environmentally-friendly option.

To Keep Your Lab-Mates Happy

- If your solution bumps into the bump trap or beyond –immediately clean all affected components–don't leave it for others to do. Also, if you continue to use the rotary evaporator after it has bumped, you risk fusing the glassware joints together with dried product.
- When you are done, empty the collection flask. Leaving unknown solvents for the next user to deal with is unkind and unsafe. Also, leaving organic solvents in the collection flask will degrade components in the rotary mechanism over time.
- Use only clean, deionized water in the water bath, and if the water bath is scummy–change it. Not only is it good practice to keep your equipment clean, you'll thank yourself the next time you drop your flask in the bath.