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Rotary Evaporation to Remove Solvent

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Overview

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Rotary evaporation is a technique most commonly used in organic chemistry to remove a solvent from a higher-boiling point compound of interest. The rotary evaporator, or "rotovap", was invented in 1950 by the chemist Lyman C. Craig. The primary use of a rotovap is to dry and purify samples for downstream applications. Its speed and ability to handle large volumes of solvent make rotary evaporation a preferred method of solvent removal in many laboratories, especially in instances involving low boiling point solvents.

Principles

Roto-evaporation requires mechanical rotation of a flask under vacuum. The rotation of the flask increases the surface area of the solvent to be removed, increasing the rate of evaporation, and reducing the risk of "bumping": when a large pocket of solvent vapor forms rapidly and displaces the surrounding liquid. The vacuum reduces the boiling point of the solvent, as well as providing a means to separate the solvent from the compound of interest.

This video will explain the process of rotary evaporation, including the key components of a rotary evaporator, or "rotovap". Advice for the most common organic solvents and crucial safety considerations will be presented.

Procedure

1. Setup

1. Pour the mixture of solvent and desired compound in a round bottom flask. Best results are achieved when the flask is filled less than half full of the solution.
2. Fill the rotovap cold traps with dry ice.
3. Attach a glass "bump trap" which prevents any solution from entering the main part of the rotovap. Secure with a Keck clip.
4. With a Keck clip attach the flask and bump trap to the adapter portion of the roto-evaporator.
5. Lower the flask into the water bath. This helps to prevent the flask from disconnection.

2. Rotary Evaporator Operation

1. Start the rotation. Different speeds are preferable for different volumes.
2. Slowly start increasing the vacuum. The vacuum is at the proper strength when: 1) condensation of the solvent can be seen on the cold finger or in the receiving flask, or 2) the solvent begins to bubble.
3. Turn on the heat for the water bath. Recall from general chemistry that vacuum reduces the boiling point of the solvent, so significantly lower temperature is needed to evaporate the solvent using a rotovap than at STP.
4. Adjust the vacuum setting as needed.
5. When all solvent has been removed turn off the vacuum and return the flask to atmospheric pressure.
6. Stop the rotation.
7. Raise the flask from the bath.
8. Remove the flask from the adapter.
9. If there is more solvent to remove it can be added it to the same flask and the procedure is repeated. Remember to empty the receiving flask when the evaporation is complete.

Applications and Summary

Rotary evaporation can be used to separate solvent from many organic, inorganic, and polymeric materials. It is crucial that the desired compound has a lower boiling point than the solvent and that the compound does not form an azeotrope with the solvent. If these conditions are true, rotary evaporation may be a very efficient technique to separate solvent from the compound of interest. Lower boiling solvents work best, however, rotary evaporation is commonly used to remove water. Higher boiling solvents such as DMF and DMSO are more easily removed using other techniques such as lyophilization, however, with a very good vacuum pump, they may be removed using rotary evaporation.