Rotary Evaporator: Solvent Removal Cheat Sheet

by [deleted] via cheatography.com/2754/cs/14223/

Introduction

A rotary evaporator is a specially designed instrument for the evapora-
tion of solvent (single-stage or straight distillation) under vacuum. The
evaporator consists of a heating bath with a rotating flask, in which the
liquid is distributed as a thin film over the hot wall surfaces and can evaporate easily. The evaporation rate is regulated by the heating bath
temperature, the size of flask, the pressure of distillation and the speed of rotation

Source: https://erowid.org/archive/rhodium/pdf/rotary.evaporator.pdf

Solvent removal

Solvent removal remains an unavoidable process that scientists and engineers must perform on scales ranging from a few milliliters to thousands of liters. The use of a rotary evaporator in this process has become ubiquitous for single batch and continuous processes. Most scientists become familiar with the technique in a university experimental laboratory course. The technique can become trivial quickly – much like other daily tasks. Unfortunately many rotary evaporator users forget the scientific principles of solvent evaporation. This is evidenced by pulling ultimate vacuums without control or knowledge of the pressure, the use of secondary condensers, problematic bumping and the use of bump traps. To achieve optimal distillation conditions, the distillation energy supplied by the heating bath must be removed by the condenser. An easy concept to remember for solvent removal via rotary evaporation is the 20/40/60 Rule.. These numbers refer to the D20ºC principle.

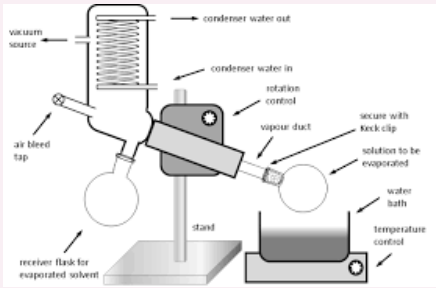
20/40/60 Rule

Use the 20/40/60 rule when utilizing your Rotary Evaporator for Solvent Removal: A Bath Temp of 60°C yields a 40°C Vapor Temp which will Condense at 20°C

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.

Rotary Evaporator



Procedure

1. Setup

☐ 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.

☐ Fill the rotovap cold traps with dry ice.

☐ Attach a glass "bump trap" which prevents any solution from entering the main part of the rotovap. Secure with a Keck clip.

☐ With a Keck clip attach the flask and bump trap to the adapter portion of the roto-evaporator.

☐ Lower the flask into the water bath. This helps to prevent the flask from disconnection.

2. Rotary Evaporator Operation

☐ Start the rotation. Different speeds are preferable for different volumes.

☐ 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.

☐ 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.

☐ Adjust the vacuum setting as needed.

☐ When all solvent has been removed turn off the vacuum and return the flask to atmospheric pressure.

☐ Stop the rotation.

☐ Raise the flask from the bath.

☐ Remove the flask from the adapter.

☐ 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.

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