

Section 5.13 Title: Using the Rotary Evaporator
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Revision Date: 11/01/19
P.I.: Prof. John F. Berry

Prior Approval: This procedure is NOT considered hazardous enough that prior approval is needed from the Principal Investigator.

Involves Use of Particularly Hazardous Substance (PHS)? No
 Carcinogen Reproductive Toxin High Acute Toxicity
Does this procedure require medical surveillance? No
Does this require use of a fit-tested respirator? No

Brief Description of Procedure:

Using a rotary evaporator to remove volatile solvents.

Location: *List the locations (buildings/rooms) where this procedure may be performed. For use of a PHS indicate a more precise location within the room, if appropriate, as a designated area.*

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Chemicals Involved:

Chemical	Physical or Health Hazard (e.g. carcinogen, corrosive)
Organic solvents	Consult relevant SDSs for more details

Other Hazards: *Include hazards, other than chemical, that may be present during operation of the procedure.*

Reduced pressure and low temperature (dry ice).

Exposure Controls: *(Check all that apply)*

PPE: Safety Glasses Face Shield Chemical Splash Goggles
 Chemical Apron Gloves (Nitrile) Lab Coat
 Respirator (type) Other:

Engineering Controls:

Fume Hood Biosafety Cabinet Glove box
 Vented gas cabinet Other: The exhaust from the vacuum pump is vented into a fume hood.

Administrative Controls: *List any specific work practices needed to perform this procedure (e.g., cannot be performed alone, must notify other staff members before beginning, etc.).*

N/A

Task Hazard Control Table: *For procedures involving numerous steps, it may be convenient to indicate specific requirements for individual tasks in the table below:*

N/A

Waste Disposal: *Describe any chemical waste generated and the disposal method used.*

Dispose of the solvents involved in appropriate carboy. Consult SDSs for more details.

Accidental Spills: *Describe the procedure for handling small chemical spills that may occur during this procedure. Note that for large spills it may be appropriate to call 911.*

Small spills may be cleaned with an absorbing material. The material should be placed in a fume hood to dry after the spill has been cleaned.

Decontamination Procedures (required for PHS use): *Describe the procedure for decontamination of personnel and equipment.*

N/A

Training: *Describe any training needed prior to performing this procedure. Include training performed in-lab and any required demonstrations of competency.*

Training is required. Training is performed by any experienced group member. The procedure will be demonstrated at least once and new members will be supervised their first time.

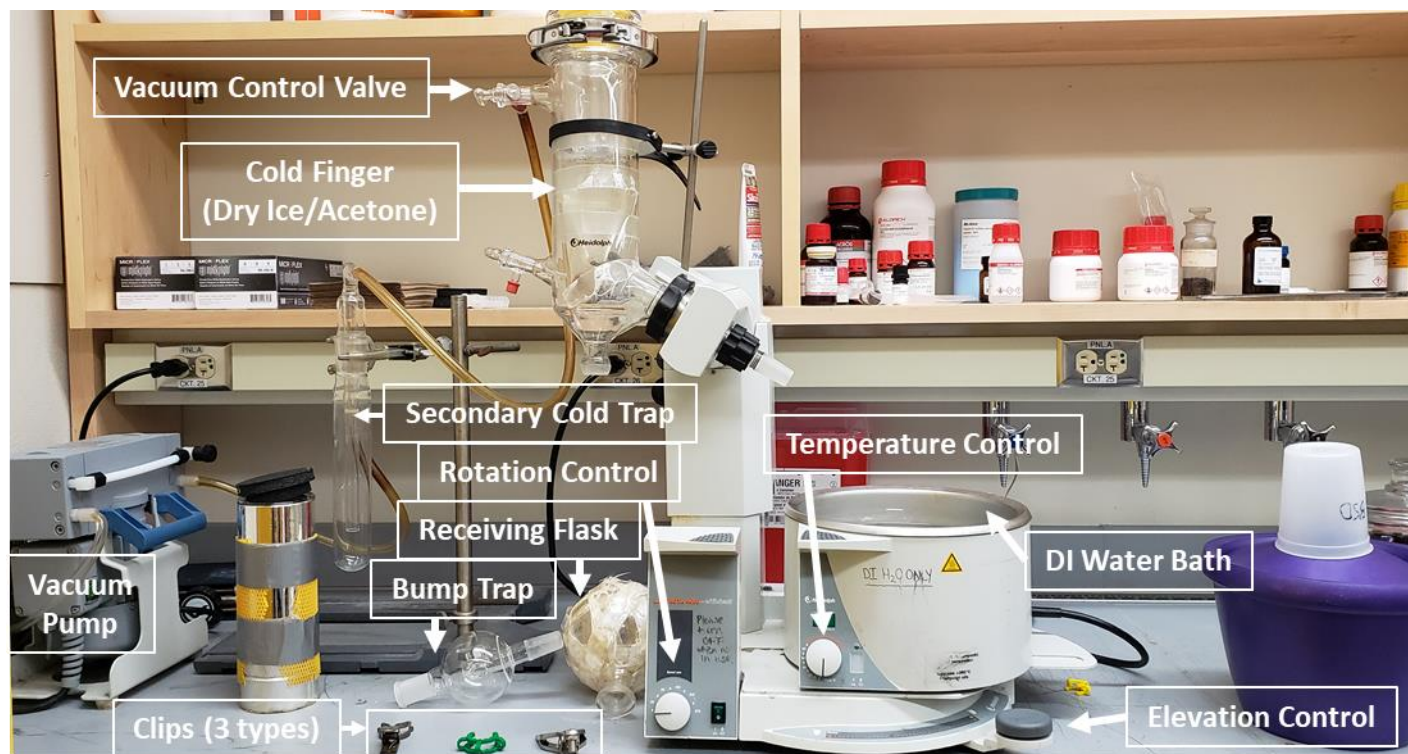
Principle Investigator Approval: I have reviewed this procedure and approved it for use. Note: Modifications to the procedure may require update to this form.

Name: John F. Berry

Signature: _____

Date: _____

Using the Rotary Evaporator (Rotovap)



Assembly of the Rotovap

When you begin, the rotovap will have been disassembled as shown in the picture above due to the cleaning procedure discussed below. It is possible the secondary cold trap will also be disassembled. The bump traps may be kept in alternative locations (i.e. drying rack or drawer) to avoid breakage.

1. To begin, choose a bump trap of appropriate joint size based on the flask you intend to use. Secure this trap to the rotovap using the spring-loaded metal clamp shown in the picture. Do not use any grease on these joints.
2. Attach your flask to the trap using the appropriately sized Keck clamp.

NOTE: This rotovap is equipped to handle round-bottomed flasks up to 1 L in size. To avoid potentially losing any sample to “bumping”, do not load your flask beyond 50% of its total capacity.

3. Lower the flask into the water bath to the point in which the solvent level in your flask is even with that of the water bath. This is to ensure there is sufficient support to the all-glass assembly. DO NOT begin rotating a free-hanging flask unsupported by the water bath.



4. To raise/lower the flask, push downward on the gray button/arm labeled “PUSH” and slide the lever to the left to lower and the right to raise. Once the flask is at your desired height, release the arm. If necessary, the water bath itself also



- slides from right to left on a track to accommodate larger/smaller flask assemblies. Fill/drain the water bath as necessary, using only DI water to refill.
5. Attach the receiving flask using the remaining metal screw-locking clamp.
6. Ensure the level of acetone is up to the top of the tapered section of the cold finger (~4”) and then slowly add dry ice until the cold finger is completely full.
7. If disassembled, put the secondary cold trap together and lower into a dewar with a dry ice/acetone or liquid nitrogen cold bath. This trap is necessary to avoid volatile solvents making their way through the diaphragm pump. This glass joint should be greased.

Removing Volatiles on the Rotovap

1. With the system completely assembled, cold baths filled, and your flask lowered into the water bath, power the system on by pushing the green power button beside the rotation control. This activates the rotation control knob and water bath temperature control.
2. Ensure the vacuum control valve is closed to the vacuum and power on the vacuum pump with the green power switch located on the front of the vacuum unit.
3. Adjust the rotation speed of your flask to a desired RPM and set the bath to your desired temperature. When rotating the temperature control knob, the number displayed will be the temperature in which you are setting the bath to. When left alone, after about 3 seconds, the display will change back and remain on the actual temperature of the bath.
4. Carefully open the vacuum control valve to vacuum and monitor your flask. Be ready to close the vacuum/open the system in case of vigorous boiling/bumping.
5. Keep an eye on the level of the dry ice and refill as required.

Cleaning the Rotovap

1. Once complete, open the system to atmosphere with the vacuum control valve.
2. Turn off the vacuum pump.
3. Stop rotation and lower the water bath setting back to 0°C. Power down the main unit.
4. Raise your flask from the water bath, remove, and set aside.
5. Remove and empty the receiving flask into the appropriate carboy located in the flammables cabinet, then place beside the rotovap to air dry.
6. Remove the bump trap, rinse with an appropriate solvent, then place back in storage location.
7. Remove the secondary cold trap from the cold bath, disassemble and empty. Leave unassembled to air dry.

8. Leave all clips/clamps in an obvious location for the next user.
9. The rotovap station should now look like it does in the top picture.

Mishaps

1. If your sample bumps up into the bump trap, perform the appropriate steps from the cleaning section and wash your sample back into your flask.
2. If your sample goes beyond the trap and into the cold finger section of the rotovap, disassembly of the rotovap will be necessary. Contact the person in charge of the instrument since this is a more involved procedure that requires working knowledge of the assembly of the entire unit.
3. Operations manual and part numbers of the Heidolph LABOROTA 4000 can be found at: <https://heidolph-instruments.com/en/service/downloads/operation-manuals>