

# Rotary Evaporation (Rotovap): Setup and Operational SOP

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*CAUTION: Do procedure in Chemical Fume Hood!*

## Setup Notes:

1. Make sure the round bottom (RB) flask is twice the volume as your sample volume.
2. Elevate the vacuum pump above the traps to aid in keeping the solvents out of the pump.
3. Hook up tubing as indicated in Figure 1. Be sure to note the in and out labels on the dry ice trap.
4. Be sure to add an open/close valve between the last trap and the vacuum pump to aid in solvent removal from traps
5. Keep the inside of all tubing as large as possible so the vacuum is strong. Suggested vacuum tubing size is approximately 3/16" wall thickness x 5/16" ID.
6. Using dry ice is the intended method of keeping the atmosphere cold to condense the solvent. If you choose to use an ice, rock salt (used for making ice cream) and water slurry please test the system first to see how many traps you must use to keep solvent out of the vacuum pumps as that can damage the pumps over time. A handy way to check this would be a check of the last side-arm flask before the pump and see if any solvent has precipitated. Also place a piece of clear tygon tubing on the out vent of the vacuum pump and check for solvent droplets that may have formed.
7. If you are using glass as part of the connection in a side-arm flask remember to use glycerin to wet the glass. Wear gloves to protect your hands and hold the glass next to the point you are applying pressure when putting the vacuum tubing on.
8. The temperature of dry ice is  $-78.5\text{ }^{\circ}\text{C}$ .

## Rotary Evaporation Procedure:

1. Fill water bath with deionized water, set temperature and turn on.
2. Put solvent "catch pot" in place.
3. Fill ice bucket and place under catch pot to remove as many solvents as possible from condenser.
4. Hook up dry ice trap.
5. Fill trap with dry ice or flaked ice and rock salt (if known to work).
6. Add a second and even a third side-arm flask "trap" between dry ice trap and vacuum pump if you are seeing condensation in the clear tubing coming out of the vacuum pump.
7. Place secondary  $\pm$  tertiary "traps" into an ice bucket to catch any additional solvent fumes carried past the first trap. Please see **Test Procedure for rotoevaporation use of flaked ice.**
8. Make sure tubing from the condenser carrying the warmed water is in the drain.
9. Fill cooling coils inside condenser and leave cooling water flowing if you're ready to evaporate a sample. (Use chiller if you have one. In the future we will have to use a chiller for the cold condenser fluid.)
10. Make sure vacuum valve at top of the condenser is "open" so you're not pulling a vacuum yet when you put on your sample flask.
11. Attach the RB sample flask to the condenser and fasten into place with green clamp.
12. Start rotation of flask slowly and gradually increase speed up to a setting of 8 or 9.
13. Slowly close the vacuum valve at the top of the condenser – watch for bubbles in your sample! Adjust vacuum as needed.

14. Once the sample has stabilized, lower the flask into the water bath.
15. Continue to rotovap as long as necessary.
16. If you need to break the vacuum to the pump to discard solvent from the trap or add ice to the dry ice trap, open the valve and leave the vacuum pump running. Discard solvent/add ice, shut valve and continue with rotary evaporation of sample.
17. When completely done with evaporating all samples, open valve to break vacuum from pump; disconnect tubing from inlet of vacuum pump and allow pump to run at least 5 minutes to dry out any droplets of moisture that might be inside it.
18. Shut off vacuum pump after 5 minutes. It is possible to plug the vacuum pump into a timer so that the pump is turned if you forget to watch the time.

Note: Solvents should be poured into proper bottles and labeled for waste collection.