

## Standard Operating Procedure

**Task:** Rotary Evaporation

**Date:** 5/13/2013

**Revision Date:** 08/24/2016

### Background:

- A rotary evaporator (“rotovap”) is used to remove solvent from a sample in a round bottom flask or vial by boiling off the solvent under reduced pressure. Heating the rotovap’s water bath above room temperature can hasten the removal of higher-boiling solvents, if necessary. This SOP is written for an IKA RV 10 Basic rotary evaporator and IKA HB 10 Basic heating bath. This rotovap uses a cold finger to facilitate solvent collection, and does not use a vacuum gauge.



### Potential Hazards:

- Solvent or other chemical exposure
- Implosion of glassware (do not use glassware with any cracks, especially star-cracks)
- Freeze burns
- Peroxides (ethereal solvents)

### Training Requirements:

- Rotary evaporation training required

### Materials Needed:

- Rotovap
- Bump trap
- Collection flask
- Sample in a round-bottom flask or 20 mL vial
- Appropriate glass adaptors (where applicable) (Rotovap 24/40 to threaded adaptor, Chemglass, CG-1318-10) and (Rotovap vial adaptor for 22-400, Chemglass, CG-1318-24)



CG-1318-10



CG-1318-24

**Procedure:**

1. Check that the cold finger of the rotovap is at least  $\frac{1}{4}$  full of acetone.
2. Check that the water level of the heating bath is at least  $\frac{1}{2}$  full. Refill the bath only with distilled water.
3. Check that the vent valve of the rotovap is open to air.
4. Add dry ice to the cold finger, a few chunks at a time. The acetone will bubble vigorously. Adding dry ice slowly avoids splashing. Continue to add dry ice until the mixture is bubbling slowly and the dry ice pellets are at level with the acetone.
5. Attach the collection flask to the condenser using a ball joint clamp.
6. Attach the bump trap to the 24/40 joint on the rotator of the rotovap using a green keck clip; then connect your reaction vessel to the bump trap using a green keck clip. **DO NOT USE GREASE ON THE ROTOVAP.** The clean ground glass joint connections will make an adequate seal, without getting grease in your samples.
7. Power on the rotovap using the button on the display screen.
8. Using the down-arrow key on the display screen, submerge your round bottom flask or vial into the water bath such that the water from the bath covers the flask at solvent level. *Be sure that the flask does not touch the bottom of the water bath.*
9. Power on the vacuum pump using the switch on the pump.
10. Start rotating the flask slowly by pressing the rotation dial on the rotator console. You can adjust the rate of rotation (rpm) by turning the dial.
11. Apply vacuum by slowly closing the vent valve. *Use caution and apply vacuum slowly to avoid bumping/splashing solvent.*

12. Once under vacuum, increase rotation speed to desired level (typically around 100-150 rpm).
13. Apply heat as necessary using the water bath (the heating bath is generally used only for high-boiling solvents). Make sure that the water bath is always  $\frac{1}{2}$  full, and remember to check its level periodically for long-term evaporation. Make sure that the cold finger is still cold (has plenty of dry ice) while removing solvent on the rotovap.
14. When all the solvent from the round-bottom flask has evaporated and collected in the receiving flask, stop rotation, turn off the heating bath (if used), and vent the system to air using the ground glass joint. Turn off the vacuum pump using the switch on the pump, and turn off the rotovap by pressing the power button on the screen.

**15. EMPTY THE COLLECTION FLASK AND CLEAN THE BUMP TRAP AFTER EVERY USE!!**

- Notes:
  - Be cognizant of the volume of solvent you are evaporating. Round bottom flasks and vials should not be more than half full for successful solvent evaporation devoid of bumping. Make sure the receiving flask is large enough to accommodate all of the solvent removed.
  - Is the chemical in your flask **toxic**? Does it **smell bad** (thiols, phosphines)? *Keep your round bottom flask capped with a glass joint or septum at all times between your hood and the rotovap cabinet! Keep the rotovap cabinet closed when not in use to minimize chemical exposure.*

**Related SOPs:**

- Waste SOP
- Cleaning Glassware SOP