ROTARY EVAPORATOR

Standard Operating Procedure

Lab: 3724 and 3710 Beckman Institute

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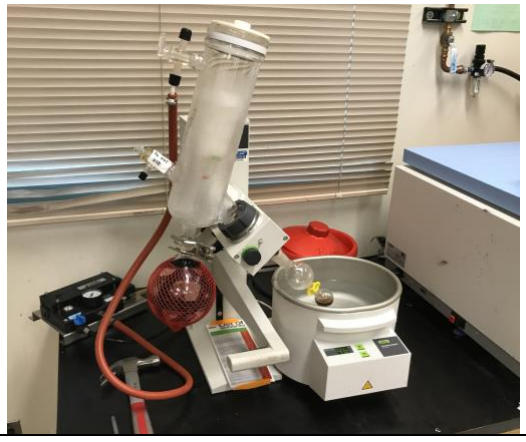
Section 1: Overview

Type of SOP: ☐Process ☐Hazardous Material ☐Hazardous Class of Materials ☒Equipment

Synopsis:

Rotary evaporators (also called "rotavaps" or "rotovaps") are used to remove solvents from reaction mixtures. Liquid solvents can be removed without excessive heating of the sample by lowering the pressure above a bulk liquid and thus lowering the boiling points of the component liquids in it. This method is most effective to remove common solvents from compounds that have significantly higher boiling points. However, concentrating mixtures that contain unstable or highly reactive components should be done with caution and is usually not an appropriate use of this equipment (see safety section for further details). The purpose of this SOP is to give a detailed procedure on how to safely operate this equipment.



Section 2: Risk Assessment Summary (Hazards and Control Measures)

Materials: Common Solvents

Solvent	CAS #	BP (°C)	FP (°C)	Class	HFR	Selected GHS Hazards
Acetone	67-64-1	56	-17	IB	2, 3, 0	highly flamm., serious eye irritation
Hexane	110-54-3	69	-26	IB	2, 3, 0	possible teratogen, aquatic tox.
Pentane	109-66-0	35	-49	IA	0, 4, 0	aquatic tox., extremely flammable
Heptane	142-82-5	98	-4	IB	2, 3, 0	very toxic to aquatic life
Acetonitrile	75-05-8	81	2	IB	2, 3, 0	highly flamm., serious eye irritation
DCM	75-09-2	40	none	none	2, 0, 0	possible carc., specific organ toxicity
Chloroform	67-66-3	61	none	none	3, 0, 0	possible carc./terat., toxic if inhaled
Diethyl Ether	60-29-7	35	-40	IA	2, 4, 0	extremely flamm. liquid and vapor
Ethanol	64-17-5	78	14	IB	2, 3, 0	highly flammable liquid and vapor
Methanol	67-56-1	65	10	IB	2, 3, 0	single exposure organ toxicity
IPA	67-63-0	82	12	IB	2, 3, 0	serious eye irritation
THF	109-99-9	65	-17	IB	2, 3, 0	possible carcinogen, serious eye irr.
Ethyl Acetate	141-78-6	77	-3	IB	2, 3, 0	serious eye irritation
Toluene	108-88-3	110	4	IB	2, 3, 0	teratogen, specific organ toxicity
Benzene	71-43-2	80	-11	IB	2, 3, 0	carcinogenic, aspiration hazard
DMF	68-12-2	153	58	II	2, 2, 0	teratogen, acute toxicity

Equipment Hazards:

Motor unit: rotates the evaporation flask or vial containing the sample
- Pinch point hazard

Vacuum system: reduces the pressure within the evaporator system
- Implosion hazard
- Ignition hazard if flammable liquid vapors escape the apparatus
- Inhalation hazard if toxic chemical vapors escape the apparatus
- Electrical outlet is a possible shock hazard

Water bath: to heat the sample
- Water bath temperature range is 25-95 ºC, possible burn hazard

Manual quick-action jack: raises and lowers glassware from heating bath
- Pinch point hazard
- Sharp glass hazard if flask is broken upon lowering into heating bath

Hazardous Conditions:

Moderate Temperature
- Water bath temperature range is 25-95 ºC, possible burn hazard

Reduced Pressure
- Implosion hazard
- Vapor formation creates an ignition and inhalation hazard

Cold finger: coolant mixtures are placed here to re-condense the solvent
- Burn hazard: liquid nitrogen (−195.79 °C)
- Burn hazard: dry ice (−78.5 °C)

Technique Hazards:

Chemical Concentration
- Ensure sample is not reactive or explosive when concentrated.
- Explosions may occur from concentrating unstable impurities during evaporation, for example when rotavapping an ethereal solution containing peroxides. This can also occur when taking certain unstable compounds, such as organic azides and acetylides, nitro-containing compounds, molecules with strain energy, etc. to dryness.

Chemical Transfer
- Flammable or toxic chemicals may be exposed to air while assembling/disassembling glassware
- Exposure may also occur if vapors don't re-condense and there is leakage in the vacuum line
- Extremely toxic or flammable/combustible materials are not appropriate for this technique

Warming up cryogen
- Dry ice/IPA mixtures should be cold enough in almost all cases, avoid using liquid nitrogen whenever possible
- Liquid nitrogen may condense oxygen from air (always operate under vacuum)
- Always pull vacuum immediately after filling cold finger with liquid nitrogen
- Empty cold finger directly after use (evaporation of liquid nitrogen by pouring on ground is fine)

Personal Protective Equipment

- Wear eye protection appropriate for the chemicals being used (either safety goggles or glasses)
- Wear lab coat and appropriate shoes/pants
- Wear appropriate gloves while transferring chemicals and touching equipment

Engineering Controls

- Electrical outlet
- Chemical fume hood for exhaust
- PIAB compressed air pump

Section 3: Procedures

Pre-operation

1. Read Material Safety Data Sheets to determine if reaction mixture and undesired solvent can be exposed to these conditions.
2. Ensure proper fire extinguisher is nearby.
3. Locate nearest eyewash and safety shower.
4. Place a note in front of the equipment to indicate what chemicals are being used.
5. Wear appropriate PPE.
6. Check water level of the rotovap. If it is below halfway, fill with a 50/50 volume mixture of DI/tap

7. water. Do NOT use pure DI to fill the bath, it will corrode the stainless.
8. Check trap and make sure it is empty. If the previous user did not empty the trap, inform the
9. superuser .
10. 3. Check top valve and make sure it turns easily. If it does not, re-grease with a SMALL amount of
11. Apiezon grease. Do not apply excessive grease; it will clog the valve. Do not use silicone grease, it
12. will corrode the glass joints.
13. 4. Fill the top trap with dry ice and isopropryl alcohol. Wait until the alcohol is thick and viscous.
14. Ensure that there is solid dry ice visible in the trap at all times.

Operation

1. Fit your sample to a bump trap of the appropriate size and attach with a keck clip
2. Attach sample-trap apparatus to the rotary unit, and secure to the rotary unit with the metal clip.
3. Turn on the power switch, on the right hand side of the water bath. Note that it is not possible to run the rotary unit without turning on the bath. The bath will automatically heat to its previous setting – if you want to use the rotovap at room temperature, adjust the water bath temperature to 23 degC .
4. If the unit is attached to a positive pressure vacuum unit, check that the main pressure valve (valve 1), blue valve on the pump (valve 2) and the top valve on the rotovap manifold (valve 3) are all OFF (perpendicular to the line). Turn ON the main pressure (valve 1) and check that the vacuum gauge reads ~-950 mTorr, then turn ON the blue pump valve (valve 2).
5. If the unit is attached to a mechanical vacuum pump, check that the pump is OFF and top valve on the rotovap manifold (valve 3) is OFF. Turn ON the vacuum pump and check that the vacuum can reach <50 mTorr.
6. Turn ON the rotary unit, so that your sample is spinning.
7. Turn ON the rotovap manifold valve (valve 3). Your sample is now distilling.

Shutdown

1. When your sample has finished distilling, turn OFF the manifold valve (valve 3)
2. If the unit is attached to a positive pressure vacuum unit, turn OFF the main pressure valve (valve 1), but not the pump valve (valve 2). If unit is attached to a mechanical vacuum pump, turn OFF the pump.
3. Rotate the manifold valve to open to atmosphere (pointing up). You should hear a whistling sound as the manifold re-pressurizes to atmosphere.
4. Turn OFF the rotary unit, so that your sample stops spinning.
5. Rotate the manifold valve to re-open to the vacuum line (pointing down). You might hear a slight whistling sound as the vacuum line re-pressurizes.
6. Rotate the manifold valve back to OFF. If attached to a positive pressure unit, close the pump valve (valve 2).
7. Empty the solvent trap.
8. **Turn the water bath OFF**
9. Remove your sample.

Section 4: Waste Disposal/Cleanup

Clean solvent trap and bump trap into an approved container and ensure that the chemical contents and your name are clearly labeled. Place the waste in the designated secondary containers for chemical pickup

and indicate if you would like it submitted for disposal at the next lab cleanup. If it is ready for disposal at that time, the lab waste manager will submit the waste to DRS.

Section 5: Emergency Response

- To quickly shut down rotovap: turn off pump, vent to airand turn off all power switches.
- Consult MSDS forsf for proper course of action in the case of chemical exposure.
- In the case of fire: evacuate lab, pull alarm, and call 911 (the rotovap is not contained in a fume hood so fire extinguisher use may be dangerous).
- If there are injuries from implosions/explosions call 911 immediately.

Section 6: Additional Information

Advice:

1. Select a flask that accommodates approximately twice the starting volume of solvent.
2. Do not use round bottom flasks with visible chips, cracks, or scratches. Vacuum glass will implode violenty if damaged and placed under vacuum.
3. If you don't allow the cold finger to cool long enough, uncondensed vapors will enter the pump.
4. To prevent evaporation from the collection flask, a cooling bath can be placed underneath.
5. As you lower the flask into the water bath, make sure you do not press the bump trap against the water bath or the collection flask against its cooling bath.
6. If liquid nitrogen is needed, it must be added directly after turning on the vacuum to prevent oxygen condensation and the pressure must be decreased at a slower rate so that the cold finger can cool under reduced pressure.
7. Faster spinning increases the surface area and prevents nucleation of bubbles. This lowers the chances of accidentally bumping.
8. Double check that the pressure valve on the condenser unit is closed when trying to pull vacuum.
9. Chose a temperature appropriate for the solvent being evaporated. Lower temperatures make for a slower evaporating process but reduce the likelihood of bumping.
10. For higher boiling solvents, check to see if your solvent can be combined with another to form an azeotrope that has a lower boiling point.
11. Boiling is not the same as bumping. As long as the bubbles don't reach the neck of the flask it's fine.
12. If the bubbles seem to be in danger of reaching the neck, reduce pressure immediately.
13. Once you have removed that majority of the solvent, empty the collection flask and continue rotovapping. This will ensure that the sample is thoroughly dry.

Checklist:

☐Read Material Safety Data Sheets.

☐Determine if reaction mixture and undesired solvent can be exposed to these conditions.

☐Proper fire extinguisher is nearby.
☐Nearest eyewash and safety shower are located.
☐Wearing appropriate PPE.
☐Cold finger cooled properly (procedure is different between IPA/dry ice and liquid nitrogen).
☐Note placed in front of the rotovap to indicate to others what chemicals are being used.
☐Solvent trap is emptied and bump trap is cleaned prior to use.
☐Bump trap and solvent trap are secured with metal or Keck clip.
☐Reaction flask is attached to the bump trap with a metal or keck clip.
☐Bath is filled with appropriate amount of DI water.
☐Follow procedure to evaporate solvent.
☐Follow procedure to shut down rotovap.

References:

(1) http://www.sigmaaldrich.com/labware/labware-products.html?TablePage=17191351
(2) http://www.chem.ucla.edu/~bacher/Specialtopics/rotavap.html
(3) http://www.chem.purdue.edu/chemsafety/chem/ln2.htm