



# UNIVERSITY of LOUISVILLE

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## Burns Group Vac Oven Standard Operating Procedure (SOP)



1. Grease vacuum trap using high vacuum silicone grease (white). Attach side arm of trap to back of vacuum oven. Attach top of trap to belt driven vacuum.



2. Check vacuum oven to ensure both the vent valve and the vacuum valve are closed (righty tighty).



3. Make sure the vacuum pump is plugged in. Turn on the vacuum pump using the flip switch near the back of the vacuum.



4. Fill trap dewar with liquid nitrogen and cover it with a towel to discourage evaporation.

5. Place material in the oven and close the door. Tighten both valves on the door.



6. Turn on the vacuum oven.

7. Slowly open the vacuum valve and ensure the vacuum pumps down to 30 inches vacuum mercury.

a. If the vacuum does not pump down properly:

i. Close the vacuum valve on the vacuum oven.

ii. Open the vent valve on the vacuum oven.

iii. Open and reclose the door, repositioning it to get a better seal.

8. Turn the heating dial to the appropriate number depending on the desired temperature. (See below)

9. Be sure to keep the trap filled with liquid nitrogen during the entire time using the vacuum oven.

10. To remove material from the vacuum oven:

a. Close the vacuum valve on the vacuum oven.

b. Open the vent valve on the vacuum oven.

c. Carefully remove the material (will be hot) with heat blocking gloves.

11. Turn off the vacuum.

a. Vent the vacuum by opening the vacuum valve and the vent valve on the vacuum oven.

12. Remove the trap.

a. Properly dispose of the liquid collected.

b. Clean the trap and leave it for the next person.

## Vacuum Heating Dial Settings and Corresponding Temperatures

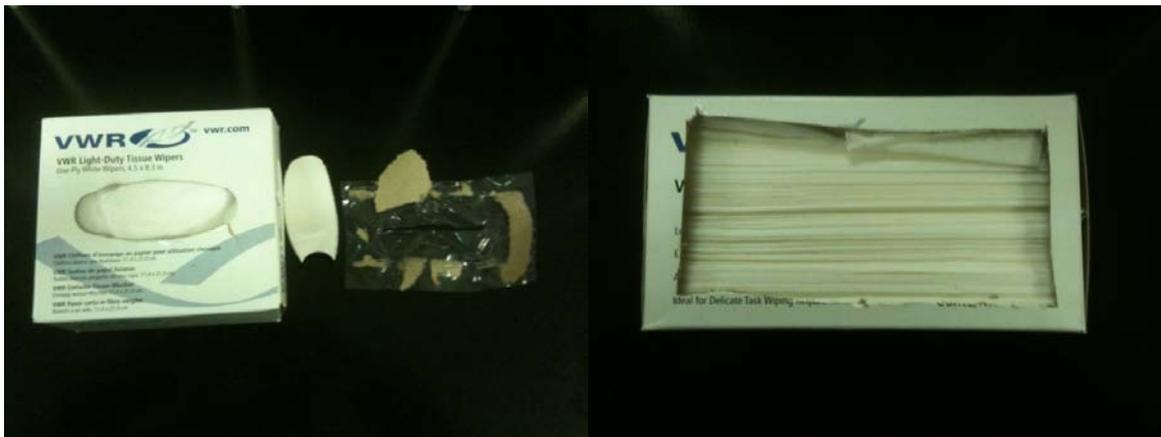
Heating dial setting	Temperature (°C)
Just before 1 (0.8)	40
At 1	50
Just past 1 (1.2)	60
At 1.5	80
At 2	100
Just before 6 (5.8)	190
Just after 6	Over 200

**Note:** Oven thermometer only reads to 200 °C

### Using the Vacuum Oven to Dry KimWipes

Paper products (paper towels, Kimwipes, etc) are filled with water and should not be brought into the box unless properly treated.

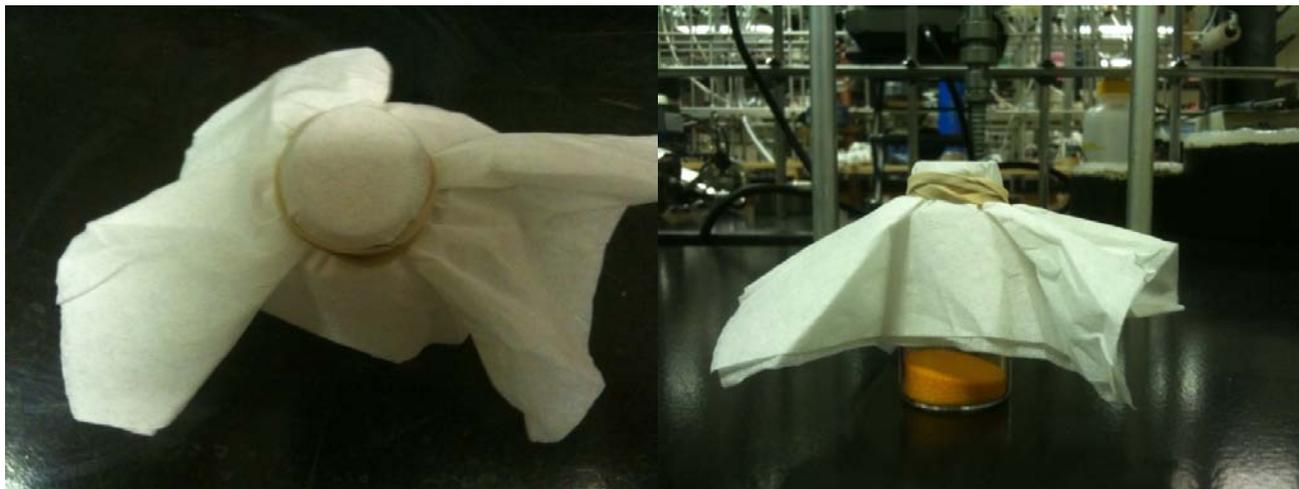
1. Open the top of the KimWipe container by removing the perforated cardboard piece.
2. Remove the piece of plastic from inside the container (this will melt in the oven).



3. Cut holes in the two sides of the KimWipe container that are not glued together.
4. Place the KimWipes in the vacuum oven.
  - a. Temperature of 80-90 °C – temperature dial setting of ~1.5
  - b. Allow to heat and remove water for 10 hours – overnight
5. When removing the KimWipes, place them directly in the antechamber while still hot and leave them under vacuum for 12 hours.

## Using the Vacuum Oven to Dry a Solid Compound

1. Make sure your compound is in a glass container.
2. Take off the top of the container and any other plastic material.
3. Place a folded KimWipe over the top of the container (will have two layer of KimWipe).
4. Secure the KimWipe with a rubber band.



5. Place the compound in the vacuum oven at the appropriate temperature.
  - a. Make sure the temperature is above the boiling point of the solvents present but below the melting point of the compound.
6. Leave the material until it is dry and free of solvents.
  - a. 12-24 hours to start

## Using the Vacuum Oven to Activate (Dry) Molecular Sieves

Activated molecular sieves are stored in the mBraun (4 Å) and the vacuum atmospheres glove box (3 Å). They must be heated in the vacuum oven at high temperature and over several days to activate them properly. The procedure is the same for both types of molecular sieves used

1. Find the large ceramic bowl (usually kept in the ceramics drawer at hood end of bench 1 in 340).



2. Weigh out 400 grams of molecular sieves in the bowl. If you use “recycled” molecular sieves make sure they have been rinsed/filtered of any residual solvent residue (acetone for 3 Å sieves, dichloromethane for 4 Å sieves) and dried at room temperature.
3. Place the ceramic bowl w/400 g of mol. sieves in vacuum oven and evacuate (see above).



4. Once vacuum has been established and the liq. N<sub>2</sub> trap filled, heat the oven to 190 °C. Leave the molecular sieves under vacuum and at 190 °C for 3 days (72 hours).
5. After 3 days under vacuum at 190 °C cool the oven to 130 – 150 °C. While the oven is at this temperature close the vacuum valve and open the vent valve on the vacuum oven. Immediately open the oven door and (with proper gloves) transfer the hot ceramic bowl/mol. sieves to one of the open glove box antechambers depending on sieve type (mBraun - 4 Å; vacuum atmospheres - 3 Å) and evacuate the antechamber.
6. The hot sieves are then allowed to cool to 25 °C under vacuum in the glove box antechamber before being transferred into the glove box and stored in the appropriately labeled storage bottle.