

## Vacuum line Procedure

1. Trap (might be done by instructor)
  - a. Remove the trap from the hood
  - b. Hold top of trap against ground glass joint on main manifold valve
  - c. Attach trap to monkey bars with clamp
  - d. Secure top ground glass joint with clamp
  - e. Connect pump hose to side ground glass joint with clamp
2. Cool Trap (might be done by instructor)
  - a. Fill Dewar with liquid nitrogen
  - b. Move wooden platform aside
  - c. Insert trap into liquid nitrogen by raising Dewar
  - d. Move platform under Dewar for support
  - e. Alternatively, insert trap into empty Dewar and fill once in place
3. Start pump (might be done by instructor)
  - a. Check if main valve of manifold is closed
  - b. Start pump
4. Evacuate manifold (might be done by instructor)
  - a. Check that all stop cocks on manifold move
  - b. If not, warm frozen stopcocks with heat gun until moveable
  - c. Close all stopcocks. Note: stopcocks are not symmetric inside.
  - d. Plug in all three pressure meter read outs: there is one 1000 Torr and one 10 Torr MKS head, as well as a low pressure thermocouple sensor
  - e. Slowly open main valve on manifold
  - f. Listen to pump and watch pressure readout drop
5. Check pressure zero point (might be done by instructor)
  - a. Open stop cock on left connecting to thermocouple sensor
  - b. Watch pressure reading drop, wait until stabilized
  - c. Note this pressure and compare to remaining two pressure readings. The zero point of the MKS heads can drift, so note "Zero" pressure for both.
  - d. If the discrepancy is large, alert instructor
6. Connect gas supply (might be done by instructor)
  - a. Remove lecture bottle from under hood
  - b. Place bottle in holder
  - c. Connect to manifold with flexible tubing on right stop cock
  - d. Secure tubing with hose clamp
  - e. Open stop cock to evacuate tubing
  - f. Check that both valves on bottle are closed
  - g. Open outer valve briefly to evacuate short connector between valves
  - h. Close valve again
  - i. Check final pressure on readout. This should not deviate much from 5.c
7. Attach gas cell
  - a. Do not touch windows of cell
  - b. Push rubber tubing of connector piece onto glass tubing on cell
  - c. Push ground glass joint of connector against corresponding piece on center manifold attachment

- d. Slide cell onto platform
  - e. Remove and adjust height of connector if needed to rest cell on platform without strain
  - f. Secure joint with clamp
8. Evacuate gas cell
    - a. Close stop cock on cell which is not connected to manifold
    - b. Open stock cock on cell which is connected to manifold
    - c. Open center stop cock on manifold
    - d. Watch pressure drop
    - e. Read final pressure. Should not be much higher than 5.c
  9. Protect thermocouple sensor
    - a. Close left stop cock on manifold leading to thermocouple sensor
    - b. Keep open from now on
    - c. Sensor was only needed to check zero points
    - d. Aggressive gases could damage sensor
  10. Fill gas cell
    - a. Check that outer valve on lecture bottle is closed
    - b. Check that center stop cock (to cell) and right stop cock (to gas cylinder) on manifold are open
    - c. Check that left stopcock (to thermocouple sensor) is closed
    - d. Close main valve on manifold
    - e. Open main valve on lecture bottle 1-2 full turns
    - f. Slowly open outer valve on lecture bottle while watching pressure meter
    - g. Fill cell to desired pressure
    - h. Never exceed ambient pressure (760 Torr)
    - i. Close both valves on lecture bottle
    - j. Note final pressure
    - k. Close stop cock at cell
    - l. Open main valve of manifold to remove excess gas
  11. Remove gas cylinder (might be done by instructor)
    - a. Check that both valves on bottle are closed
    - b. Make sure that the tubing to gas cylinder is evacuated
    - c. Close stop cock on manifold
    - d. Loosen hose clamp
    - e. Remove tubing from bottle
    - f. Store bottle
  12. Remove cell
    - a. Check that cell stop cocks are closed
    - b. Make sure connection between cell and manifold is evacuated
    - c. Close center stop cock on manifold
    - d. Remove glass joint clamp
    - e. Slide cell toward you on platform
    - f. Break vacuum at joint by moving cell up
    - g. Remove connector piece
  13. Lower the pressure in the cell
    - a. Make sure that main manifold valve is open, all stopcocks closed

- b. Connect cell (see 7)
- c. Open center stop cock on manifold to evacuate connection to cell
- d. Close main valve on manifold
- e. Open stop cock on cell which connects cell to manifold
- f. Gas will expand into manifold, lowering the gas pressure in the cell about 2-3 fold
- g. Check final pressure
- h. Close stopcock on cell
- i. Open main manifold valve to remove excess gas
- j. Remove cell as in 12.

14. Evacuating cell

- a. Connect cell to manifold (see 7)
- b. Open center stop cock to evacuate connection to cell
- c. Open cell stopcock to evacuate cell
- d. Close cell and manifold stop cock
- e. Remove cell (see 12) and store in desiccator

15. Finishing up (might be done by instructor)

- a. Make sure all stopcocks and main valve are closed and neither cell nor gas cylinder are connected to manifold
- b. Open conical ground glass joint on lower valve which is connected to pump
- c. Turn lower valve 180° to vent pump
- d. Switch pump off
- e. Turn valve back 180°
- f. Open right stopcock on manifold to vent manifold
- g. Open main valve on manifold to vent trap
- h. Remove Dewar, discard liquid nitrogen
- i. Disconnect trap on top and side
- j. Loosen trap clamp
- k. Note: hold trap at top! Cold parts of trap are dangerous to touch
- l. Place trap in hood