Turning on Physical-Absorption experiment:

1. Check that valves #1, 2, 3, 4, 7, 9, 10, 11, 12 and 13 are all closed.

2. After about five minutes (when the pump should no longer be gurgling), close valve #4 and open valves 3, 5, 6, 7, 8, and 9. Valves # 10 and 11 should **NEVER** be open during **ANY** pump-down phase. During this second pump-down period, fill liquid nitrogen (LN2) into the glass dewar surrounding the pump-trap (located between valves #2 and 4).

3. When the thermocouple vacuum gauge reads about 50 milli-Torr (or 50 microns of Hg), open valve #12 and pump down the sample cell until the gauge reading is at or below 100 milli-Torr. Then, close valve #12; by now, you should be able to close valve #3 and open both valves # 2 and 4. The thermocouple vacuum gauge should read below 10 microns in a few minutes if everything is proceeding normally.

4. Now, finish pumping down the absorption cell by opening valve #12 again.
    a. The ultimate pressure on the thermocouple vacuum gauge should be about 6 milli-Torr.
    b. Pressure on the accurate Baratron capacitance vacuum gauge is measured on the Keithley 2000 DMM as a voltage with a calibration of 10 mV/Torr. Your DMM should read between 3 and 4 mV. Is it correct?
    c. It is not unusual for this capacitance gauge to have a voltage offset. We could tune it out, but it will change over long periods of time, so the best thing to do is to push the "REL" button to eliminate the offset.
    d. Push the "FILTER" button and increase the number of readings to about 50. This should make the reading look less noisy.

5. Close valves # 5, 6, 7, 8, 9 and 12. Put LN2 into the dewar that surrounds the absorption cell. You need only wait a minimum of about 5-minutes at this cooling stage before proceeding.

6. You will note that valves # 10 and 11 have masking tape on them. They should be closed, by convention, when the tape is on these valves. When you start the experiment with N_2 gas, remove one end of the masking tape from valve #11.

7. At this point, push the "REL" button again on the DMM. Open valve #11 and then close valve #11.

8. There are multiple combinations of valve openings that are possible at this point, but to illustrate, open valve #7 and then close it.

9. Open valve #6 and wait a few minutes for the Baratron pressure to stabilize. This pressure represents your initial pressure for the first gas shot, $P_0(1)$.

10. Now, open valve #12 for your first absorption. It will take more than 10 minutes for the Baratron pressure to stop changing appreciable. We suggest that you wait a minimum of 15 minutes for this first shot to fully absorb into the 2D gas phase on the absorber. *[At this point, gas phase conduction is cooling off the sample.]* The second pressure measurement represents your final pressure for the first gas shot, $P_f(1)$. Subsequent shots will probably take less time for the pressure to stabilize, but you will have to experiment a bit here to see how long is necessary. Also, the gas absorbed in this example-shot will probably be only about 1% of the total you will end up with on the sample.

11. **CLOSE** valve #12 before preparing the next shot.

12. Open valve #11 and then close valve #11. **Do not push the "REL" button again on the DMM since your pressure is expected to be accumulative.**

13. Repeat steps 9, 10, and 11 to determine $P_0(2)$ for the pressure before opening valve #12 and $P_f(2)$ for the pressure after coming to equilibrium again with valve #12 open.

14. Repeat this sequence as many times as necessary according to the writeup, but if you make a major mistake (not recording a pressure or forgetting to close valve #12 before preparing the next shot), you will have to start over again, from the beginning.

15. If you need to start over, or you have finished taking your isotherm, go to the five steps listed on page 7 of the writeup to remove the gas from the absorption cell. It is important to follow these rules explicitly, because of the potential hazard of damage to the apparatus.

Some tips on dosing:

1. First and most important: Be sure the ENTIRE SYSTEM is well pumped out before admitting any gas from the storage bulbs. ***Failure to follow this may invalidate all of your data and possibly contaminate the storage bulbs***.
2. When starting with the N2, Close valves #7, 9 -- Then admit a dose of gas from the storage bulb into the 19.0 cm^3 space above the storage bulbs. Note that ~ 10 cc STP is roughly what is adsorbed at the phase transition. A single dose from the storage bulb to the 19.0 cm^3 space may well be adequate for the entire nitrogen dosing procedure if you are careful.
3. Dose from the 19cm^3 space to the 3.8 cm^3 space, then carefully into Vc (Be sure the sample space Vs is valved out at #12 -- This is the most common data taking error and once made will destroy your data run)
4. When approaching the phase transition at ~7 torr, dose in small pressure increments, perhaps a torr or even less over the Pfinal from your last dosing. This will ensure a well defined phase transition curve.
5. If you accidentally overshoot and get a larger dose in Vc than you intended, but you have NOT yet dosed the sample (Vs), you can remove some of the dose using the vacuum pump via valve #5. (Once you have dosed the sample, it is too late as you cannot determine what fraction of the gas was adsorbed)
6. The adsorption process is not instantaneous. Don't rush this experiment -- After dosing, be sure to give the sample time to adsorb the gas dose. The pressure should stabilize to within the accuracy of the pressure meter before proceeding to the next dosing.