

ASAP 2460 Standard Operating Procedure

Cleaning Sample Tubes

It is recommended to check to see if sample tubes are clean several hours before you plan to use them. The best cleaning method for the sample tube set depends on how dirty it looks; light, medium, or heavy.

For light the best method is;

1. File sample tube about half way with alcohol and use the sample tube brush to clean the tube. Use alcohol and kimwipes to clean the filler rod.
2. Use oven in N122 to dry tubes. I recommend 80-110 C for 2-3 hours.

For Medium the best method is:

1. Fill a beaker with water and mix in some Alconox (Use 5 grams of Alconox (or other suitable detergent) per 500 mL of warm water).
2. Use sample tube brush and Alconox solution to clean the tubes and use kimwipes and solution on the rods.
3. When tubes and rods are clean rinse them with alcohol a couple of times.
4. Use oven in N122 to dry tubes. I recommend 80-110 C for 2-3 hours.

For heavy the best method is;

1. Fill ultrasonic cleaning with water and mix in some Alconox (Use 5 grams of Alconox (or other suitable detergent) per 500 mL of warm water) and then run the ultrasonic clean for let's say 30 min.
2. Fill a beaker with water and mix in some Alconox (Use 5 grams of Alconox (or other suitable detergent) per 500 mL of warm water).
3. Use sample tube brush and Alconox solution to clean the tubes and use kimwipes and solution on the rods.
4. Repeat steps 1-3 until tubes and rod are clean.
5. When tubes and rods are clean rinse them with alcohol a couple of times.
6. Use oven in N122 to dry tubes. I recommend 80-110 C for 2-3 hours.

If neither of these methods works see the specialist.

Loading the sample and determining the Sample Mass

- 1) It is recommended to check to see if sample tubes are clean several hours before you plan to use them. If they are not clean than clean them by following the instructions in cleaning the samples tubes section and the start this document.
- 2) Label the sample tubes on the frosted part to keep track of the samples.
- 3) Create a data table. Example below

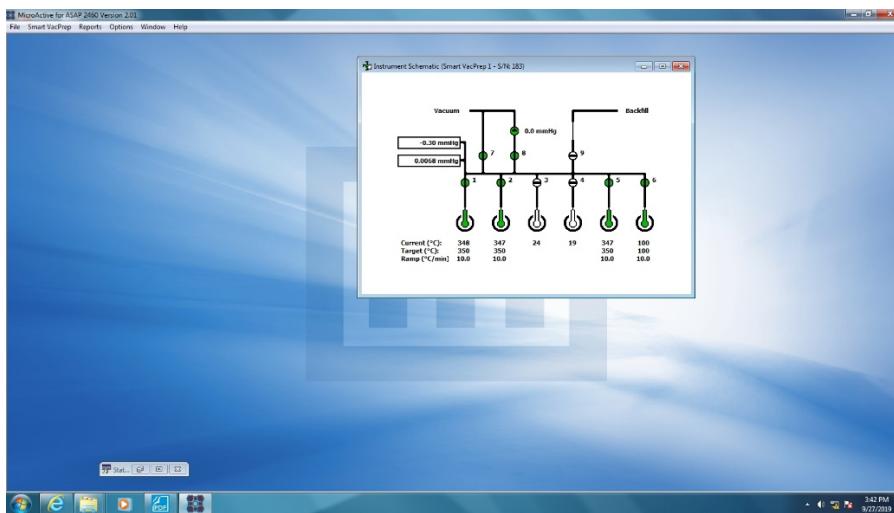
Tube label	Sample	Mass Empty sample tube set (g)	Mass filled sample tube set (g)	Mass after degas (g)
1 or A	FeCoPt 400	49.7144	50.0218	50.0083
2 or B	FeCoPt 500			
3 or C	FeCoPt 600			
4 or D	FeCoPt 700			

- 4) Weigh the empty Sample Tube Set and record in data table
- 5) Load the sample into sample tube.
- 6) Weigh the Sample Tube Set with sample and record in data table.
- 7) Load samples into Smart VacPrep

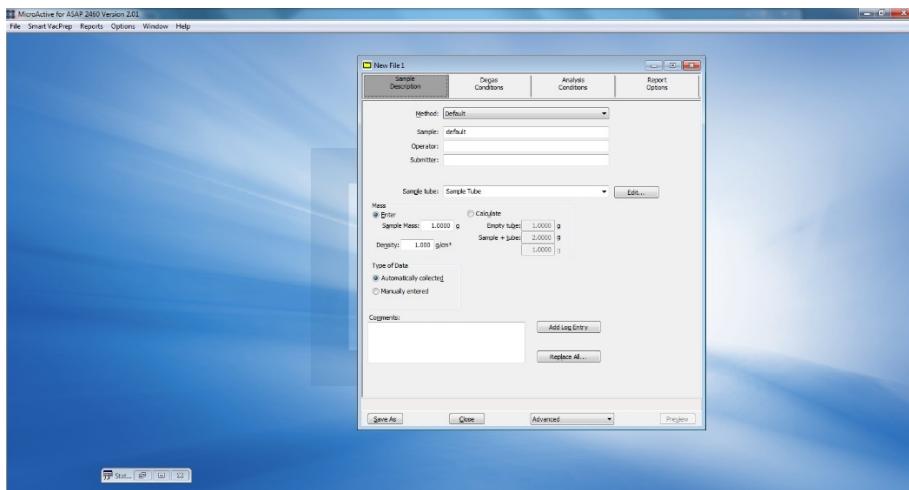


Setting up degas and measurement

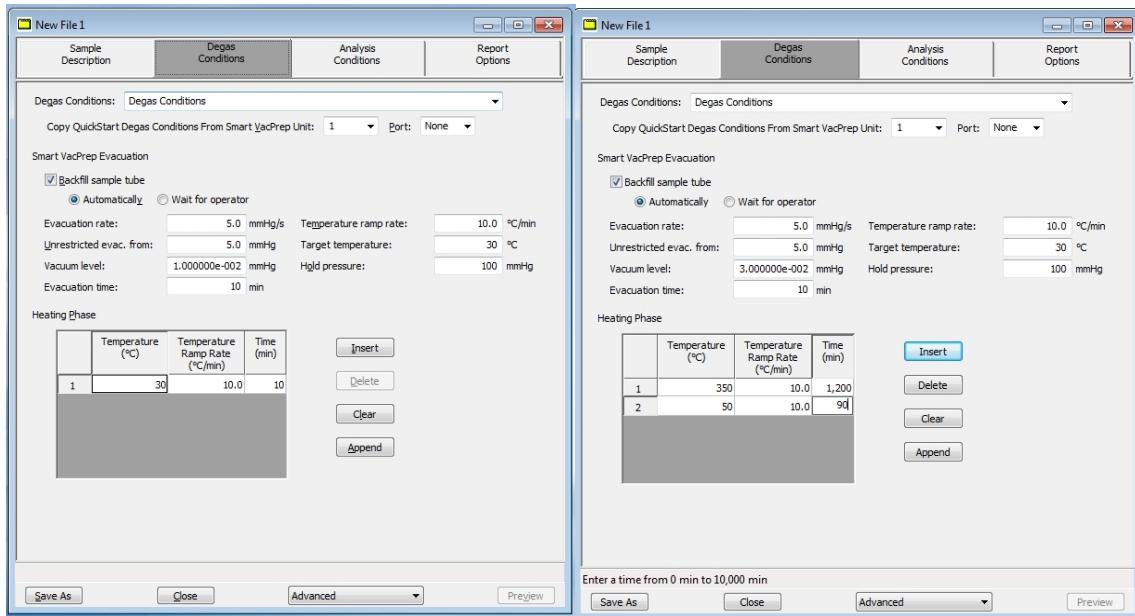
1. On computer use internet explorer to access FOM website, (Note, the computer is connected to intranet only and has access to the FOM server. It is not connected to internet,)
2. Log on to ASAP degas system. Logging will turn on the degas system.
3. Open the MicroActive for ASAP program. The program takes 2-3 min to boot up. It communicates with the degas system and tries to communicate with the measurement system but since only the degas system is on an error message will pop up just click OK.
4. The screen should look like this



5. Go to File/New sample and the screen should look like;

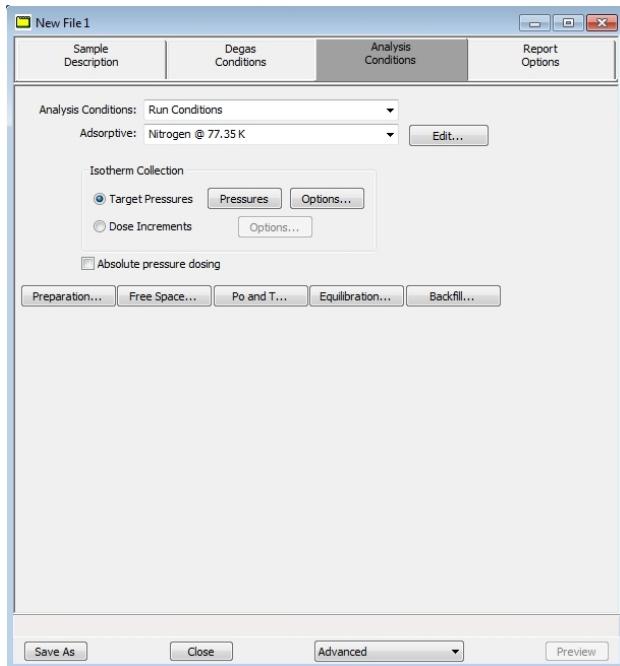


6. Enter sample name and tube ID. For sample mass click the calculate option and enter the mass of the empty sample tube set and the filled sample tube set.
7. Click the degas tab and the window will look like below left;



I recommend you change the Vacuum level to 3-4.00e-2 mmHg (= 30-40mTorr) and enter degas parameters you would like to use (example above right).

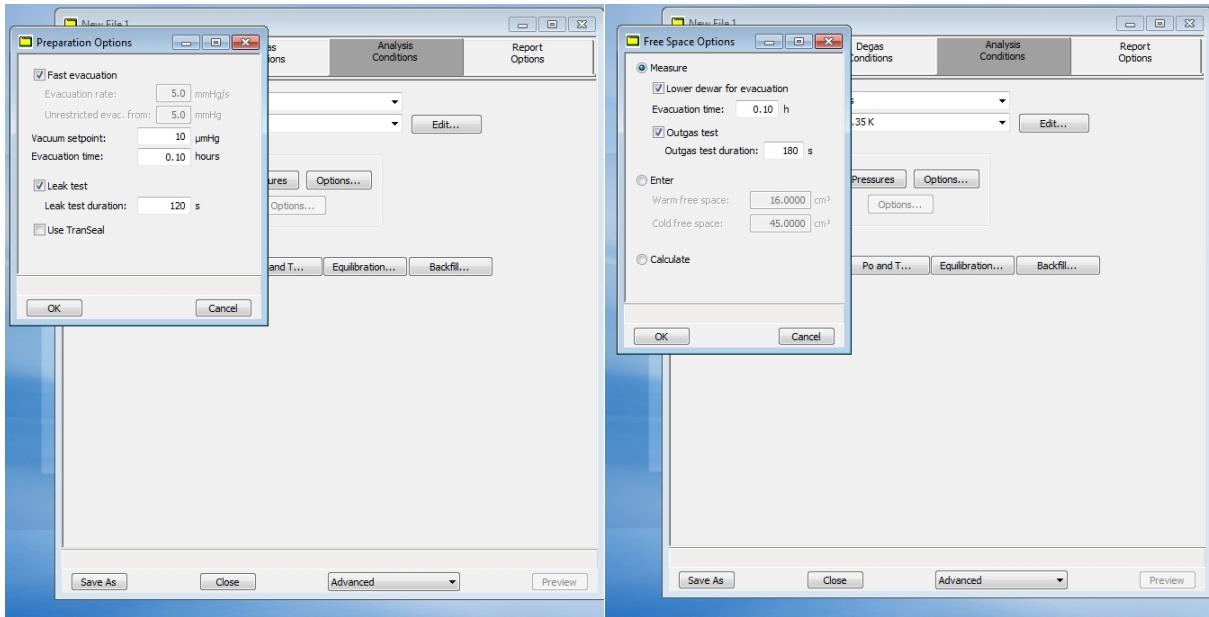
8. Next click the Analysis tap and the window should look like this;



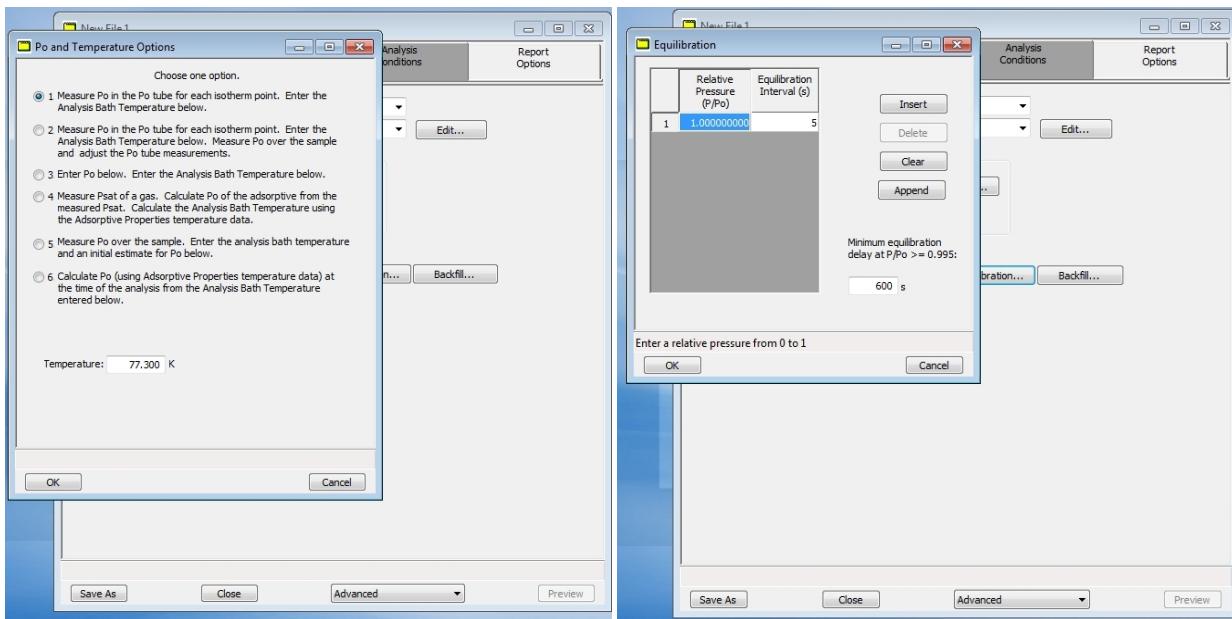
9. Now you need to change options on the tabs; Preparation, Free Space, Po and T, Equilibration, and Backfill.

10. For Preparation click the fast evacuation and the leak test. For Free space click measure option and make the option look like the right image below. I recommend the evacuation time

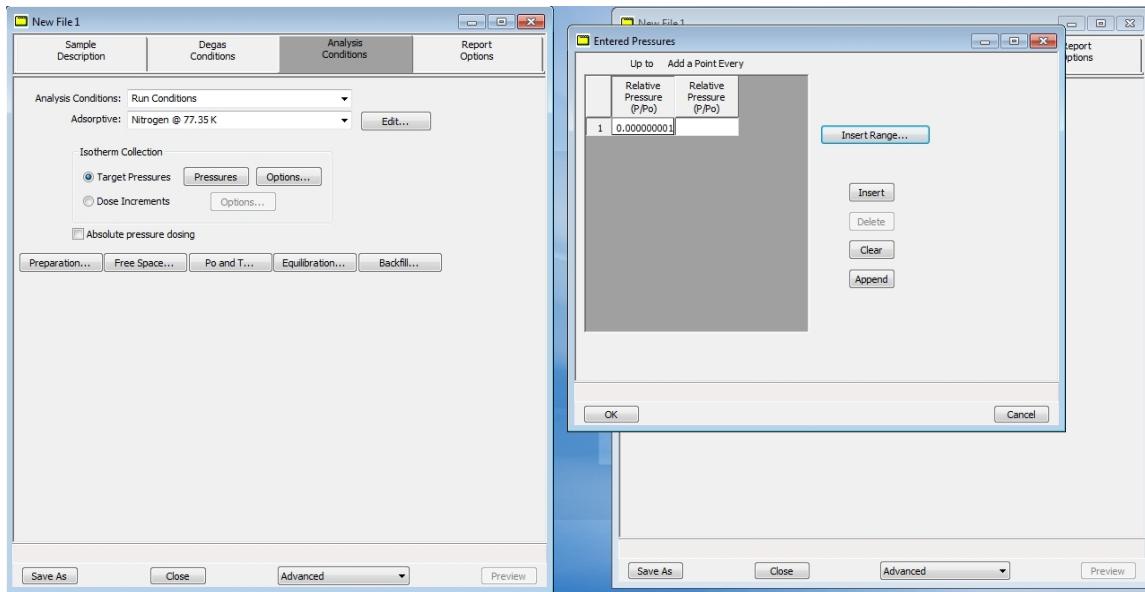
be set to 0.5 hours in both settings. Note it is recommended to run a leak test and degas test but not required.



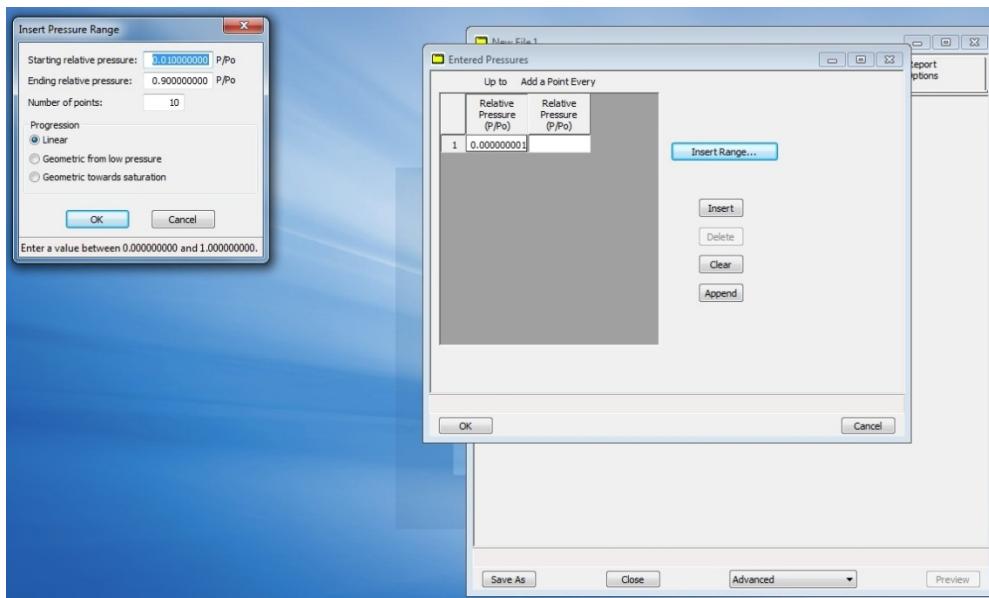
11. For Po and T use the measure option. For the equilibration it is recommended to use 10s. Note longer equilibrium times tend to give better data but at the expense of adding time to the measurement.



12. Press the pressure button near the middle of the window and a screen pops up that allows you to set P/Po values you want to take data at;



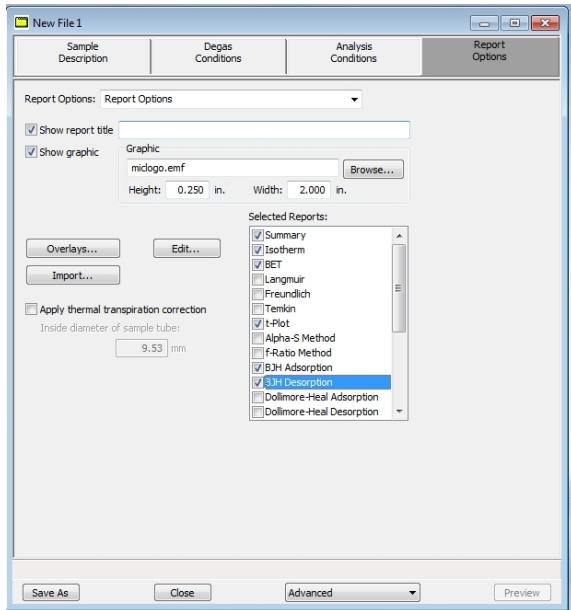
You can enter the values manually or use the insert range button to generate a range of P/Po values.



The standard measurement has three ranges;

- 1) 0.05-0.30, 11 points, progression = linear
- 2) 0.5-0.995, 14 points, progression = Geometric towards saturation
- 3) 0.995-0.10, 20 points, progression = Geometric towards saturation

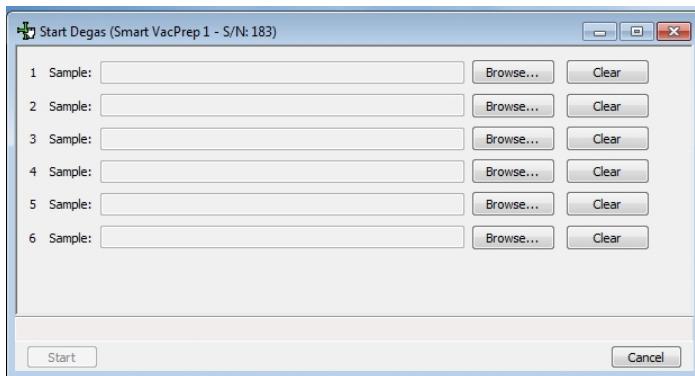
13. Next click the report tap and the window should look like this;



Click the reports that you want. I recommend Summery, Isotherm, BET, BJH absorption and desorption.

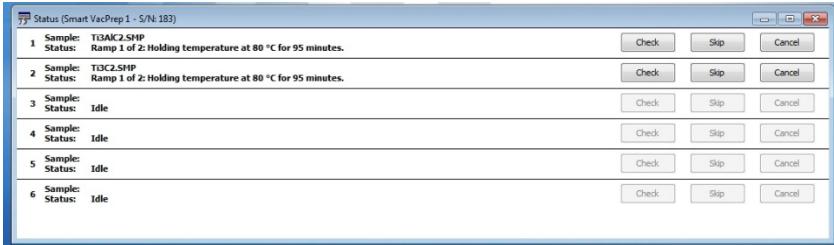
Running the degas and measurement

1. Go to File/smart VacPerp/ Start degas and a window pups up;



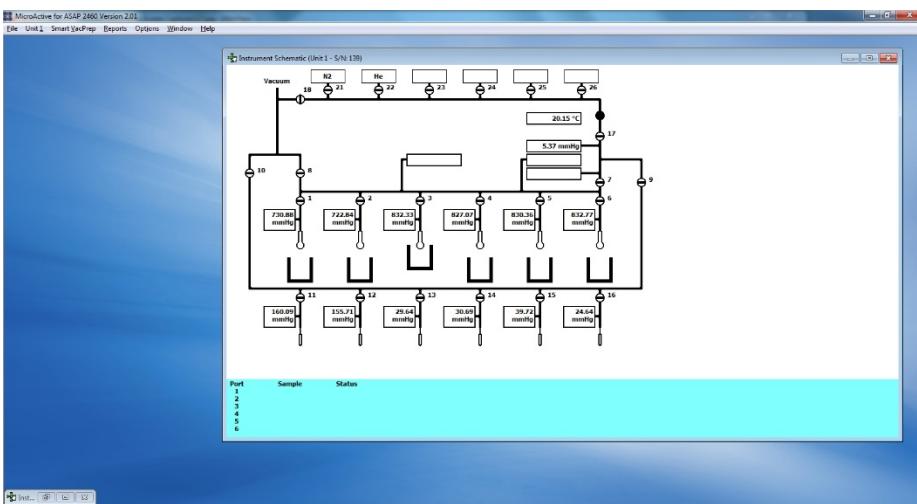
Use the Browse button on each port to load the correct samples and when finished push the start button.

2. Another window pups up;

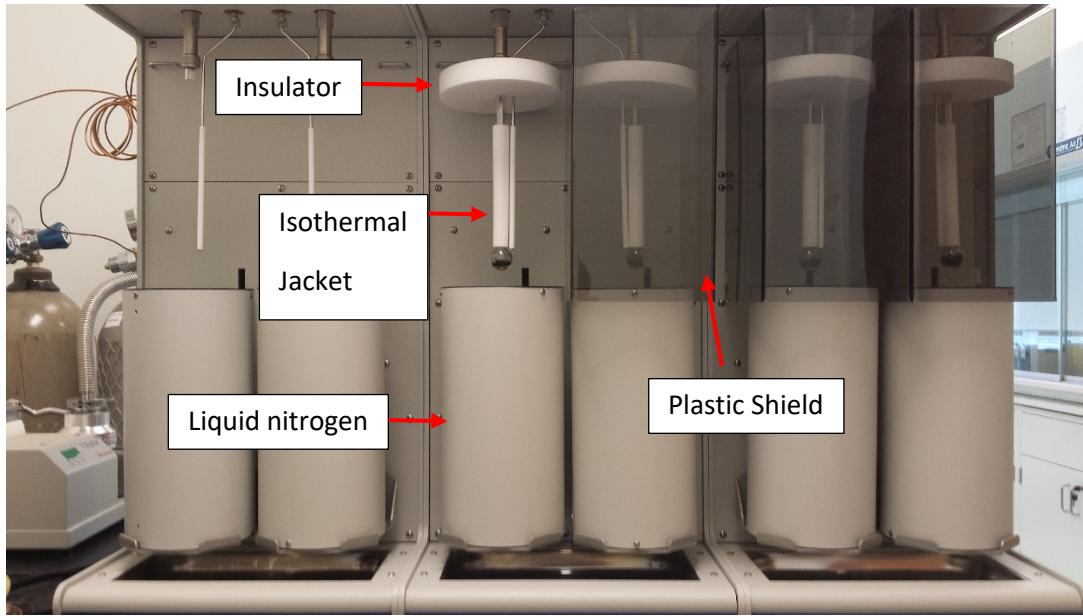


This window gives information on the degas process and allows you to check the degas rate of your sample. Note you want the degas rate $\leq 0.0025\text{mmHg/min}$ after 2-3 minutes of collecting data.

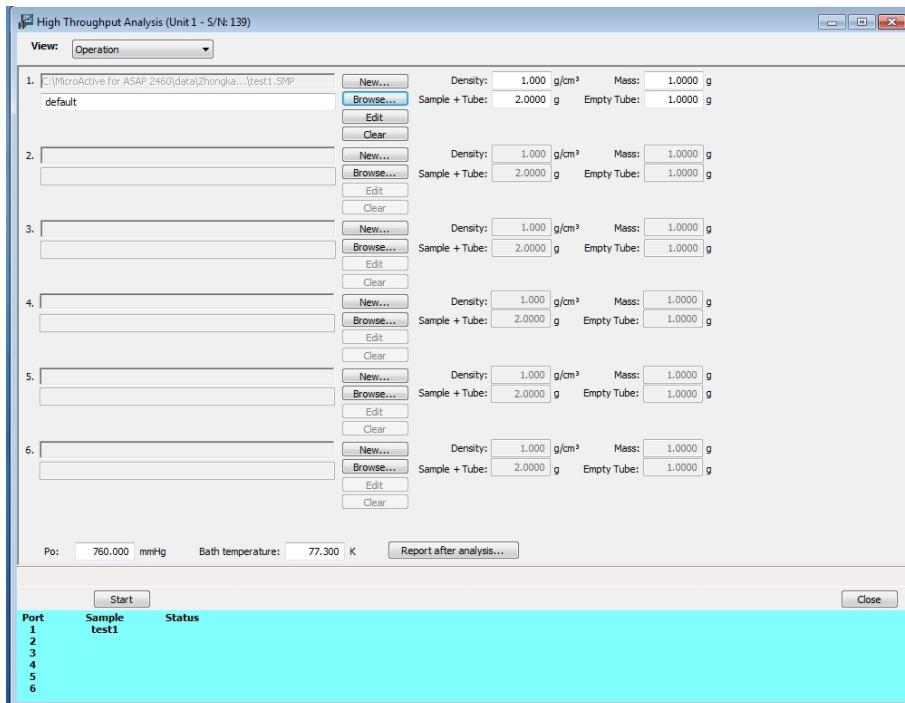
3. After the degas is finished log off to ASAP degas system on FOM and close the MicroActive for ASAP program.
4. On FOM Log on to ASAP measurement system.
5. Open the MicroActive for ASAP program. The program takes 2-3 min to boot up. It communicates with the measurement system and tries to communicate with the degas system but since only the measurement system is on an error message will pop up just click OK.
6. The screen should look like this;



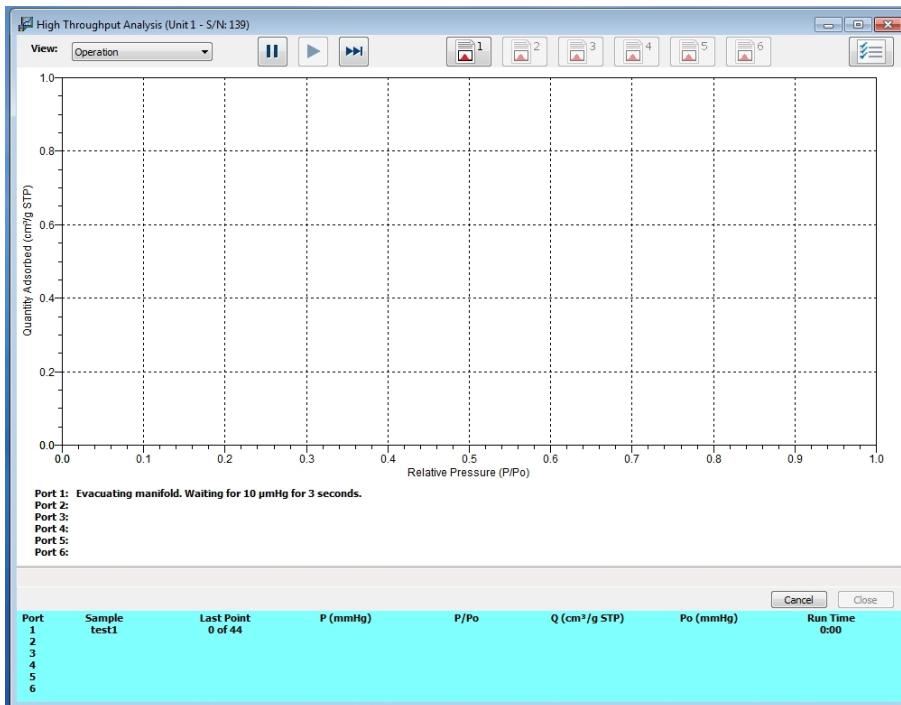
7. Get liquid nitrogen from JH 099A and fill the needed nitrogen dewars on the measurement system.
8. Remove your sample from the degas system and weigh it again. Record the new mass in the data table you created earlier. Note after the degas, you should expect a slight lower mass on the order of 1-5% but it is sample dependent.
9. Open the sample file and change the mass in the sample description tab
10. Mount the samples in the measurement system make sure to put on the isothermal jackets when finished they should look like the picture below;



11. Go to File/unit 1/Start analysis or high throughput analysis and a window pops up:



Use the Browse button on each port to load the correct samples and when finished push the start button. Then another window pops up which allows you to monitor measurement in process.



12. When the measurement is finished let sample warmup to room temperature and remove samples and clean the glassware.