ASAP 2020 PHYSISORPTION

LABORATORY PRACTICAL EXERCISE GUIDE

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DAY 1 - INTRODUCTION TO SAMPLE FILE USAGE AND SAMPLE PREP

LAB Session 1 - Intro to sample prep and sample files

Physi Sample Prep

Sample preparation is the foundation upon which any good, reliable, reproducible analysis is built. With the ASAP 2020 very few things can go wrong in sample preparation. In this section we will examine these critical aspects.

Sample Quantity

The ASAP 2020 measures pressure and then computes volume adsorbed as a result of changes in pressure. Very slight errors in measurement may be caused by minute, inherent transducer error.

There is a sacrifice of accuracy when the total amount of sample in the test tube is very low. For the highest accuracy possible, greater amounts of sample should be used. Total surface area amounts approaching 50 m2 are not uncommon for highly accurate analyses. Remember, however, that the greater the amount of total surface area present for analysis the more time must be allowed for the analysis. You should develop your own standards as you discover the best methods for your needs.

Of course, any weighing errors, no matter how small, are magnified when low sample masses are used. This is why a minimum of 0.1 g is specified. When analyzing a light or fluffy material, the entire area of the test tube within the heating mantle can be filled with sample in an attempt to attain sufficient sample mass.

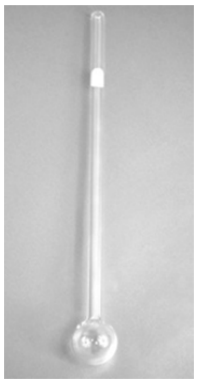
Remember that the sample mass initially placed in the sample tube will be reduced as a result of the removal of trapped moisture (degassing). It is usually a good idea to use 5 - 20% excess sample mass than required so a sufficient mass will remain after the sample has been cleaned.

The seal frit is a convenient method to insure sample material is not contaminated as it is transferred from the degas port to the analysis port. It is simpler to use than a rubber stopper and we recommend its use as part of standard sample preparation.

LAB – Initial Sample Preparation

Please use the following items , an analytical balance, and the sample data sheet (below)to perform the steps below.

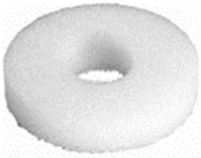
Clean Sample Tube



Filler Rod

Seal Frit

Weighing Support



Reference Material (Carbon)

1. Tare the balance with weighing support in place.
2. Weigh the assembled sample tube (including filler rod and seal frit) using the weighing support for stability.
3. Record this mass on the sample data sheet.
4. Tare balance with weighing dish.
5. Weigh approximately 0.60 grams of Carbon reference material.
6. Load the sample into the sample tube.
7. Insert the seal frit into the top of the sample tube.
8. Prepare a second sample tube, this time using about 0.25 grams of the Silica-Alumina reference material.

ASAP Series Sample Data Record

Sample tube:_____ Sample: __ Carbon_____

Before Degas:

1. Mass of empty sample tube assembly (sample tube, seal frit and filler rod) _____ g

2. Mass of sample tube assembly plus sample _____ g

3. Mass of sample (Step 2) – (Step 1) _____ g

After Degas:

4. Mass of sample tube assembly plus sample _____ g

5. Mass of sample (Step 4) – (Step 1) _____ g

After Analysis:

6. Mass of sample tube assembly plus sample _____ g

7. Mass of sample (Step 6) – (Step 1) _____ g

You may use the After Degas value (Step 5) or the After Analysis value (Step 7), provided they are close to the same value.

Compare the sample mass obtained after analysis (Step 7) with the sample mass after degas (Step 5).

These two values should be close in range. If a significant difference is noted, analysis problems may exist or the sample may have been improperly degassed.

ASAP Series Sample Data Record

Sample tube:_____ Sample: _____Silica Alumina_____

Before Degas:

1. Mass of empty sample tube assembly (sample tube, seal frit and filler rod) _____ g

2. Mass of sample tube assembly plus sample _____ g

3. Mass of sample (Step 2) – (Step 1) _____ g

After Degas:

4. Mass of sample tube assembly plus sample _____ g

5. Mass of sample (Step 4) – (Step 1) _____ g

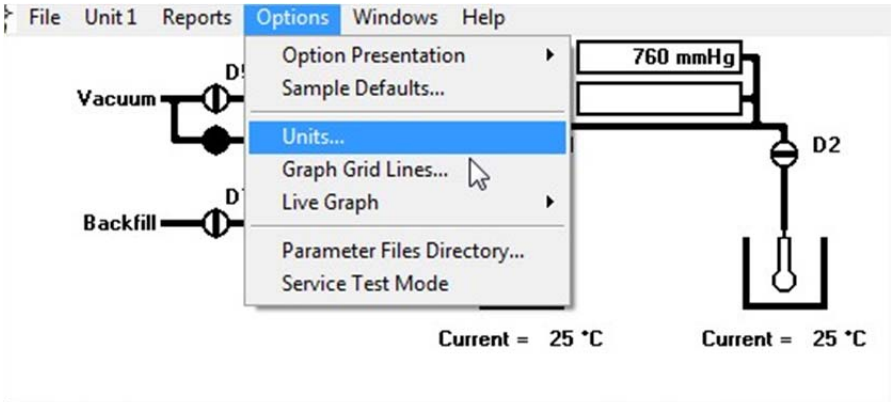
After Analysis:

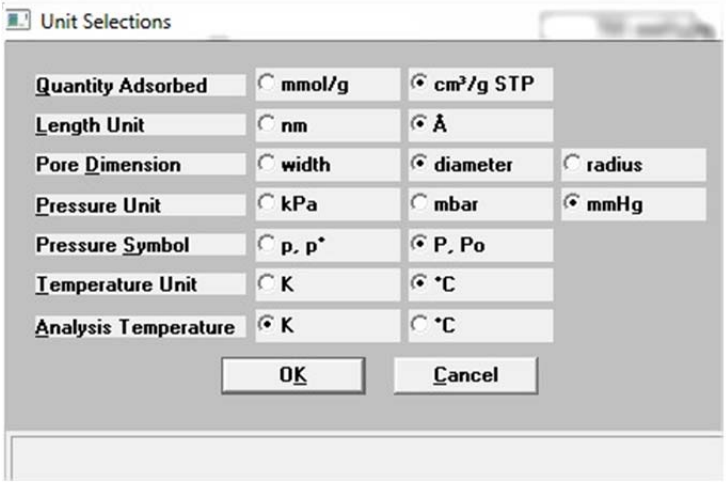
6. Mass of sample tube assembly plus sample _____ g

7. Mass of sample (Step 6) – (Step 1) _____ g

TRAINING ACTIVITY – UNITS

Some of the values displayed by the 2020 can be presented in a variety of units. The selection of units is found in the Options menu. Before you begin your other laboratory activities, you need to change a few of the default unit assignments. Please select Units on the Options menu and make the Unit Selections outlined below.





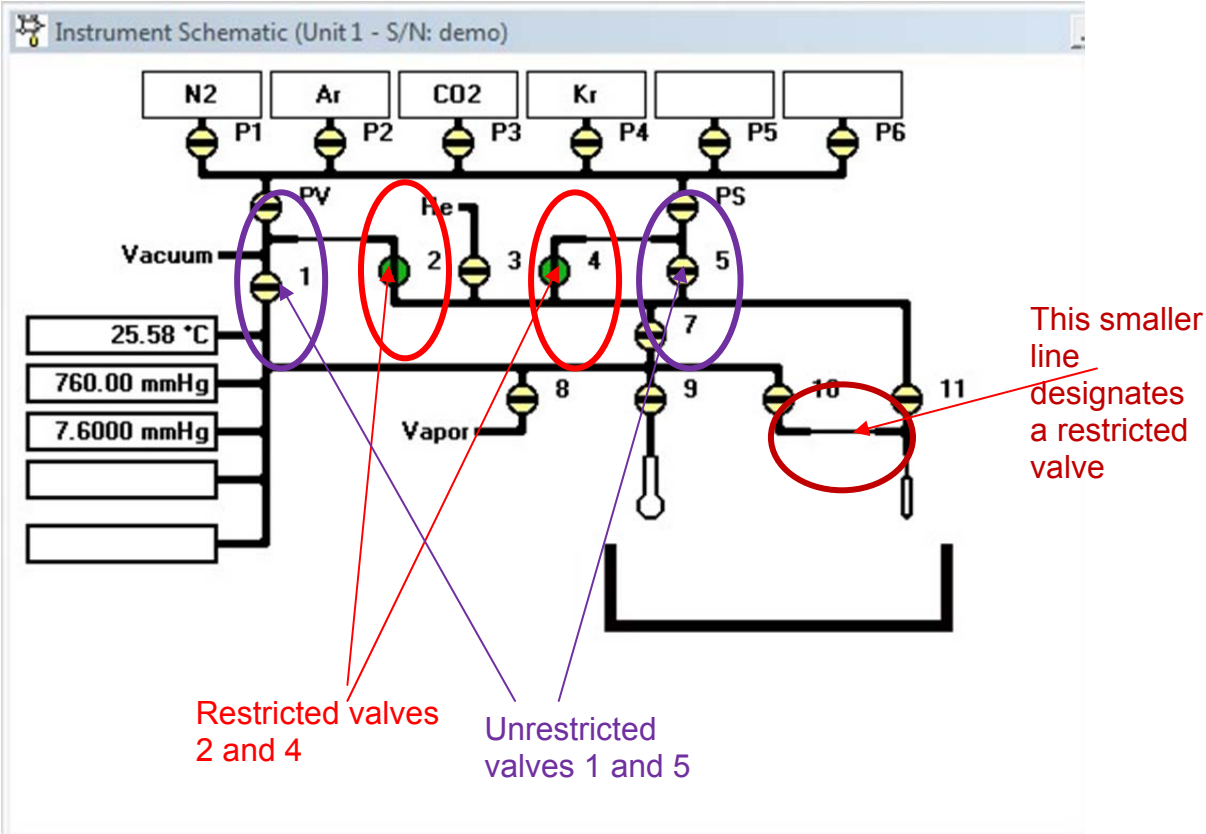
CLEANING THE SAMPLE

Before a sample can be analyzed, the sample surface must be cleaned. Any volatile materials adsorbed on the surface must be driven off. This process is known as degassing. If degassing is not performed or is incomplete, the total sample surface will not be available for adsorption and the collected data will not represent a true measure of the surface area and porosity of the sample.

With this instrument, the sample is exposed to heat while being placed under vacuum. The heat serves to drive off these contaminants while the vacuum system exhausts them away.

The instrument requires that the operator select a temperature for each degas port. This should be the maximum heat possible which will not cause a change in the surface and porosity characteristics of the sample. Using the highest possible temperature will shorten the degas time required to prepare a sample. If a safe temperature is not known and/or a low temperature must be used, the degas time will be lengthened. The instrument is designed so that the level of sample outgassing can be checked at any time during preparation.

When the system exposes the sample to a vacuum, it introduces the sample to this vacuum gradually. This is accomplished through the use of a restricted valve. The operator is required to determine a pressure crossover point at which the unrestricted vacuum valve opens. This exposes the system directly to the vacuum. Using a restricted valve initially prevents sample fluidization. If the sample is either a very fine powder or a very fluffy material, this crossover point must be carefully set, usually at a low level (typically less than 100 μmHg). In the case of Silica Alumina. This is not a problem. It is too heavy to fluidize and too large to get past the seal frit.

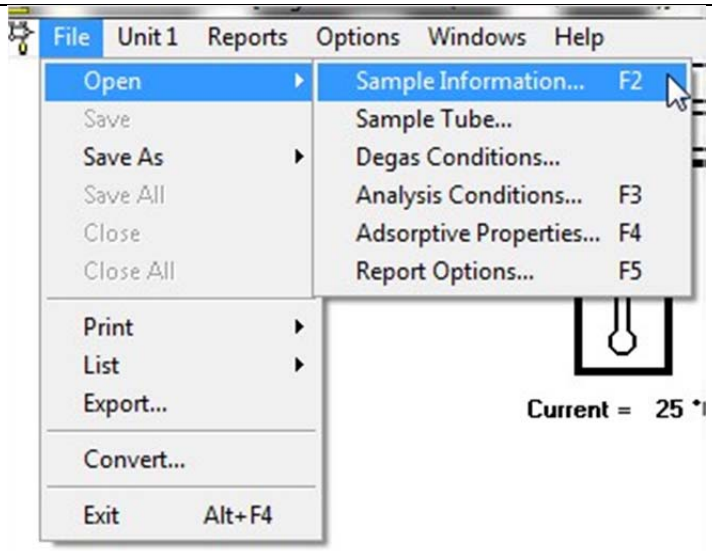
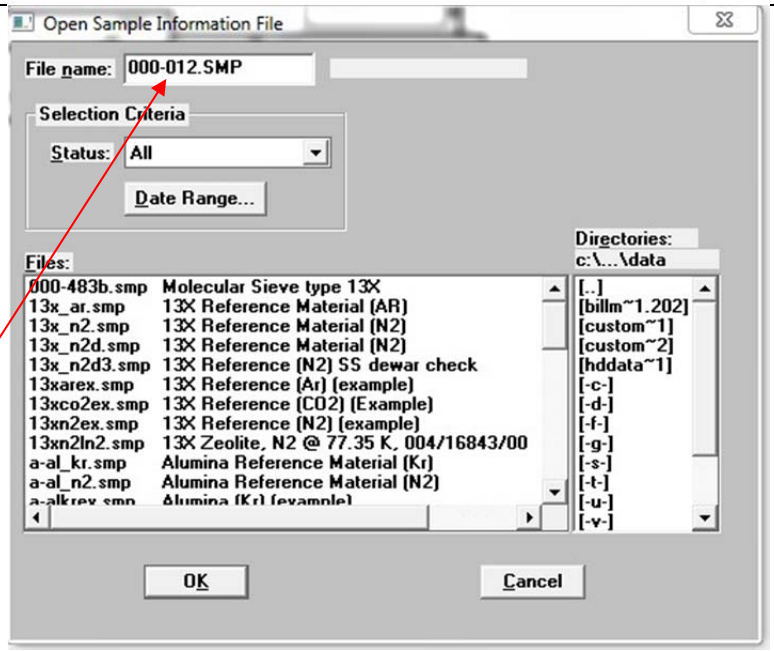


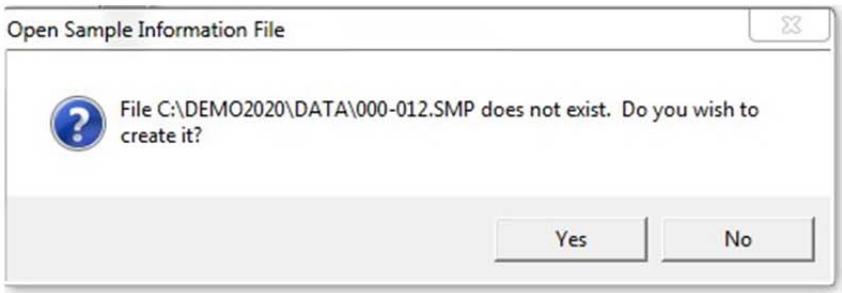
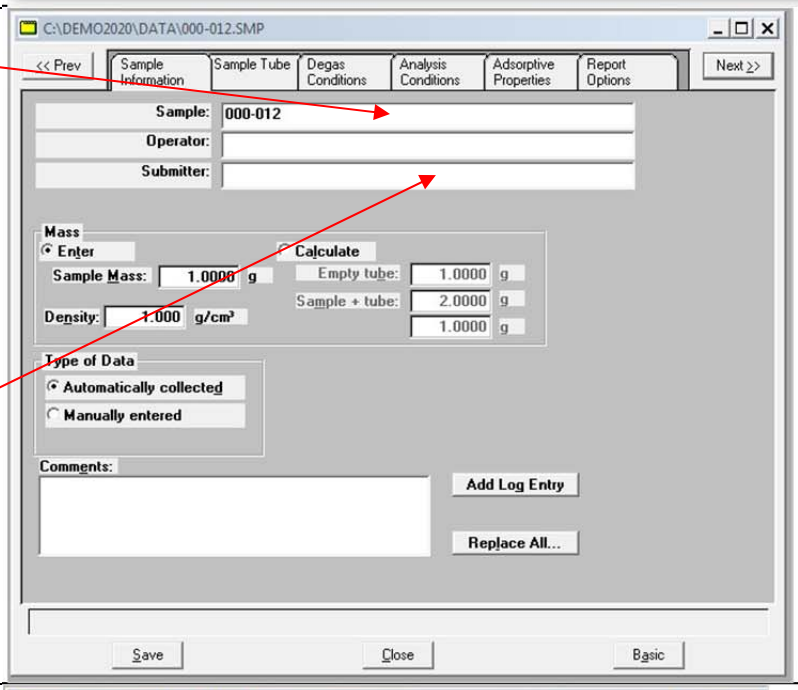
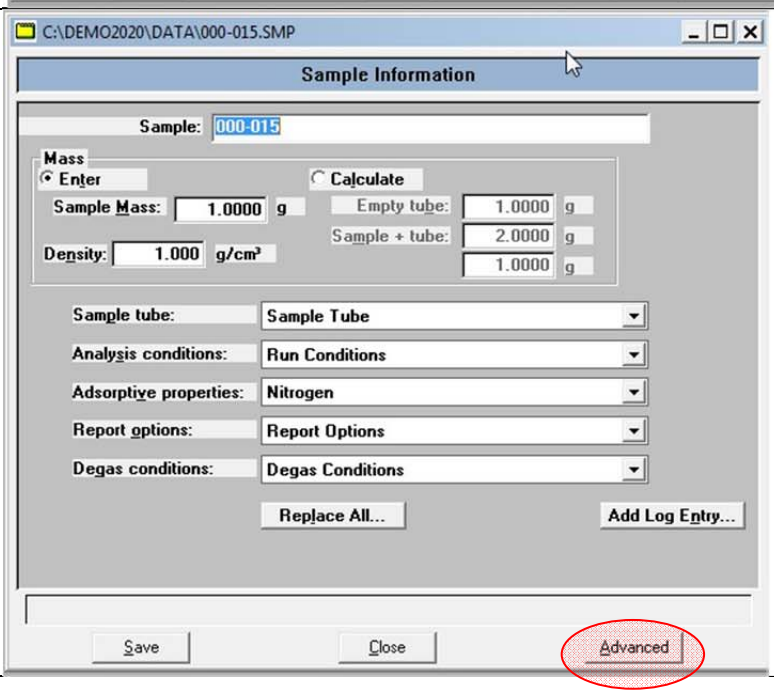
TRAINING ACTIVITY – CARBON SAMPLE FILE CREATION FROM PRE-EXISTING PARAMETERS

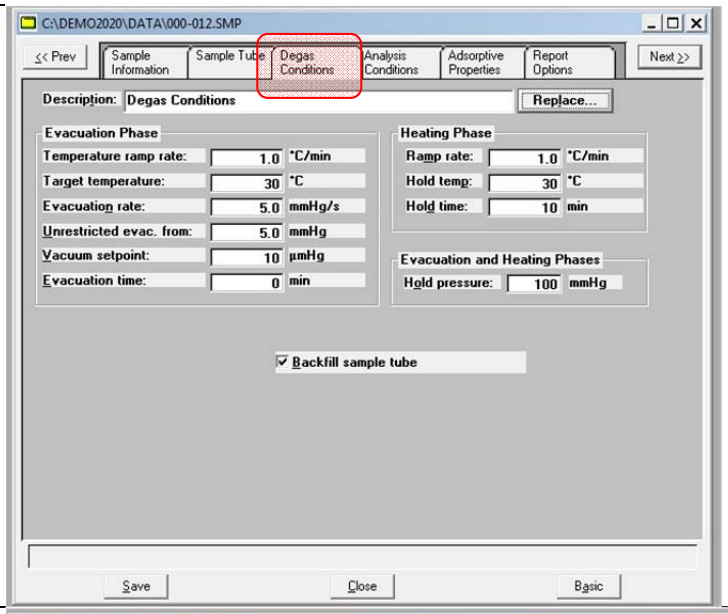
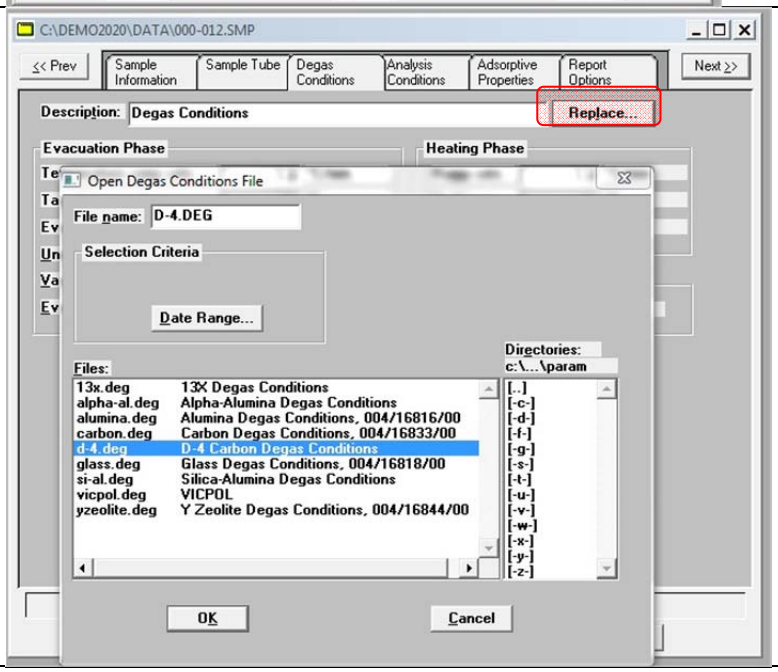
We will load each sample onto the degas system of the instrument shortly, but first we must create sample files for each.

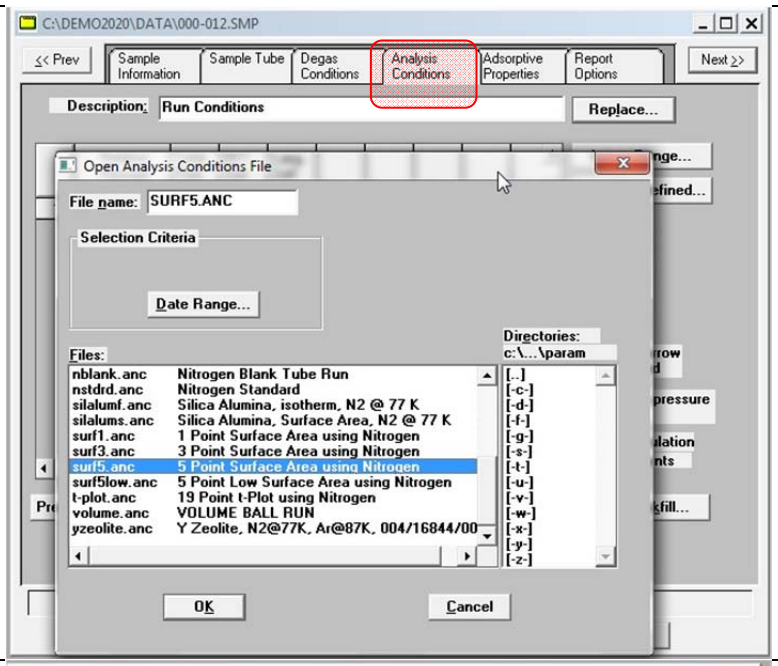
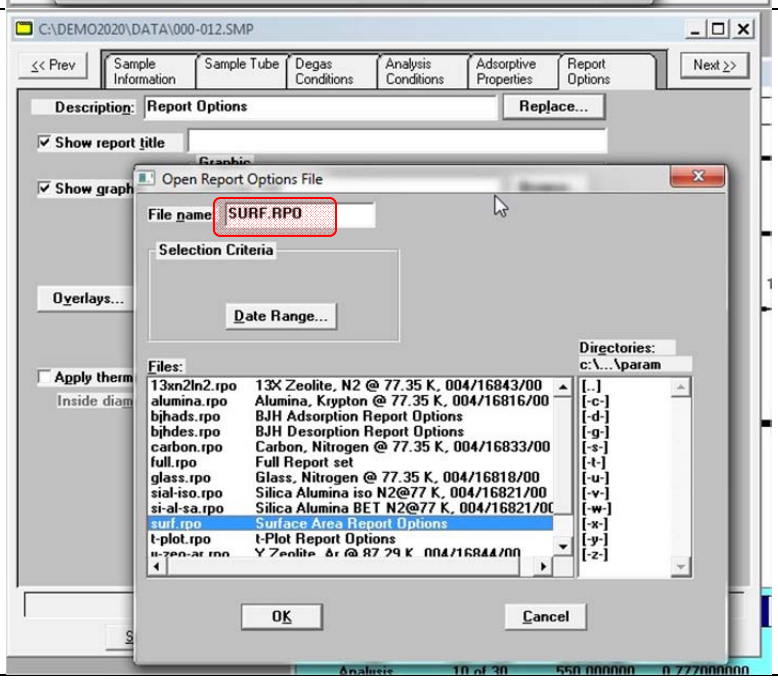
Sample degassing is controlled through the ASAP operating software. The degas conditions are kept in the sample information file which is also used to control analysis of the sample, reporting of the results and storage of the analysis data. We will create the sample file for the Carbon Reference Material first.

LAB (Creating Carbon Sample file)

Click on File, Open, Sample information.	
Degas parameter files with the conditions we need to degas and analyze the carbon sample are contained within 2020 software. You will use these parameter files to create a sample file. You would click **OK** to accept the default file name in this case it is 000.012.SMP (Your default name may differ.). For this test please type in "CARBON". It will make it easier to locate the file later.	

Then Click on **Yes** to create the new sample file.	
Type Carbon Reference Material for your sample identification. It is also a good practice to add your sample tube and/or stopper identification to this field. You may enter your initials as the Operator. Please leave "Submitter" blank. You would use this to track who asked for a data file to be run.	
NOTE: If your file does not contain the tabs shown above, please click the "Advanced" tab to change from the "Basic" file mode.	

Next, select the Degas Conditions tab.	
Click **Replace** and select the D-4 Carbon degas conditions file. We are not using D-4 carbon, but the degas conditions used will be the same.	

To finish the file, use the same procedure as above to select surf5.anc for the Analysis Conditions ….		
and surf.rpo for the Report Options. Finally, click **OK**, then **Save**, and **Close**.		

Training Activity – Automatic Si/Al Sample File Creation

The ASAP 2020 program is designed to allow you to easily create and analyze sample files. One way the software does this is to use Automatic Sample File creation. An operator may define several conditions like, for example, the Analysis Conditions or Report Options that were selected during the last exercise. These conditions can then be used to automatically generate a sample file. This procedure will be covered in detail now.

LAB – Si/Al Sample File Creation

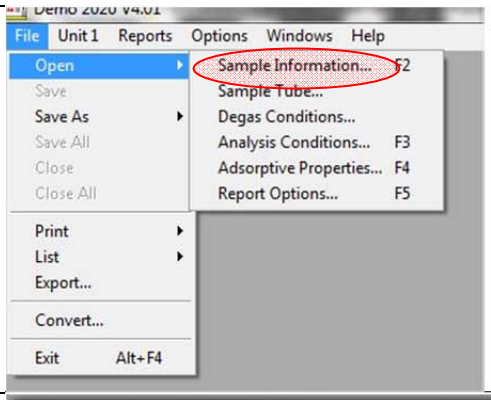
You will now create another file using both default and previously programmed conditions. The ASAP 2020 allows six types of files to be created.

1 Sample Information	a complete sample file
2 Sample Tube	sample tube information
3 Degas Conditions	parameters which control sample degassing
4 Analysis Conditions	parameters which define the analysis process
5 Adsorptive Properties	the analysis gas properties
6 Report Options	data reduction methods

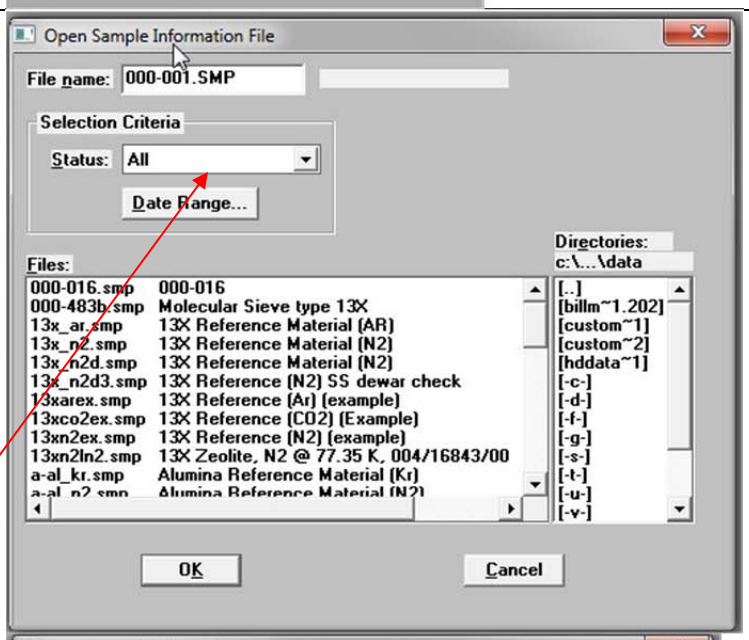
The last five file types above are used to insert into a Sample Information file to assist with automating the file creation process.

Please perform the following steps to initiate your sample file creation.

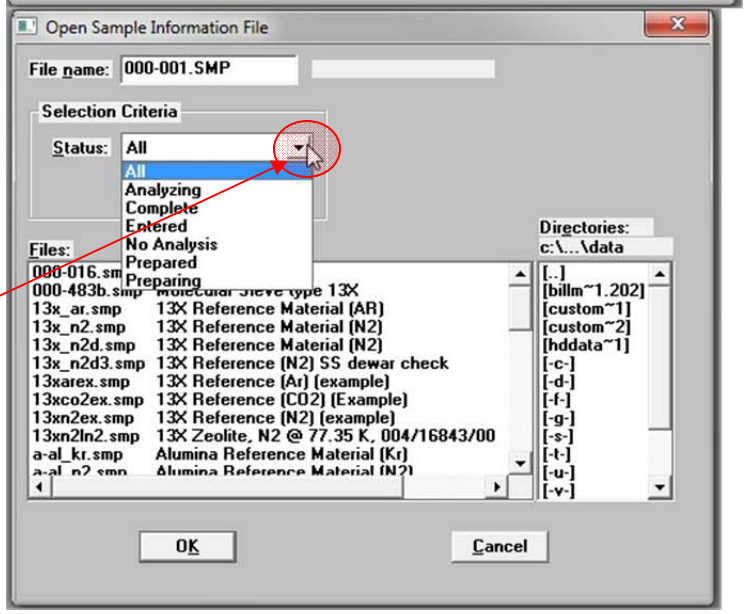
Click on File, Open, Sample Information



Once you have accomplished this you will see the Open Sample Information File dialog box. A File name is automatically displayed. This is the file name under which this sample will be stored on the computer and is chosen from the default values. You may overwrite the default file number provided with your own file name with up to 8 characters.
A box for <u>Selection Criteria</u> is displayed below. Here we can select the files from which we wish to choose in the event that we are opening an existing file.

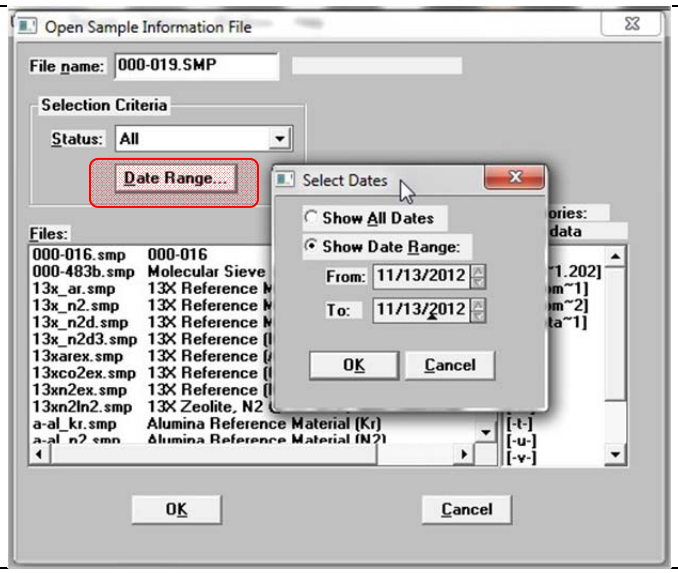


Click on the down arrow at the right of the Status box. We can choose to see files which: have not been analyzed; are currently analyzing; are complete; are manually entered; or all sample files.



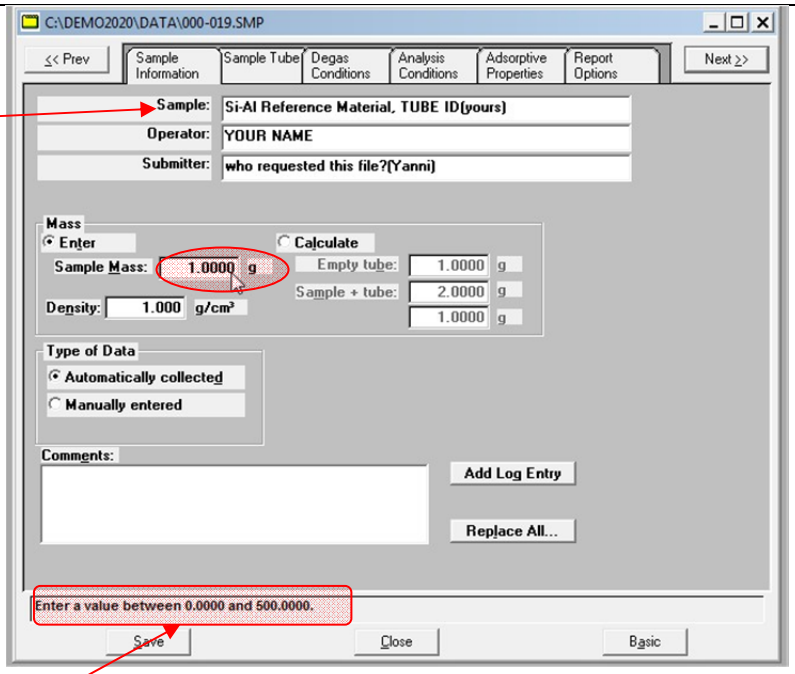
A further selection method allows the choice of date ranges. Click the

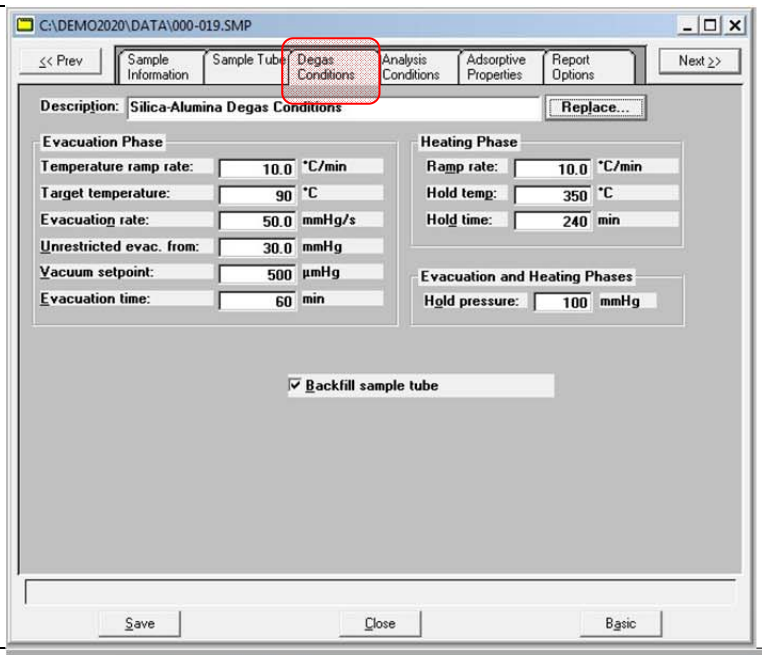
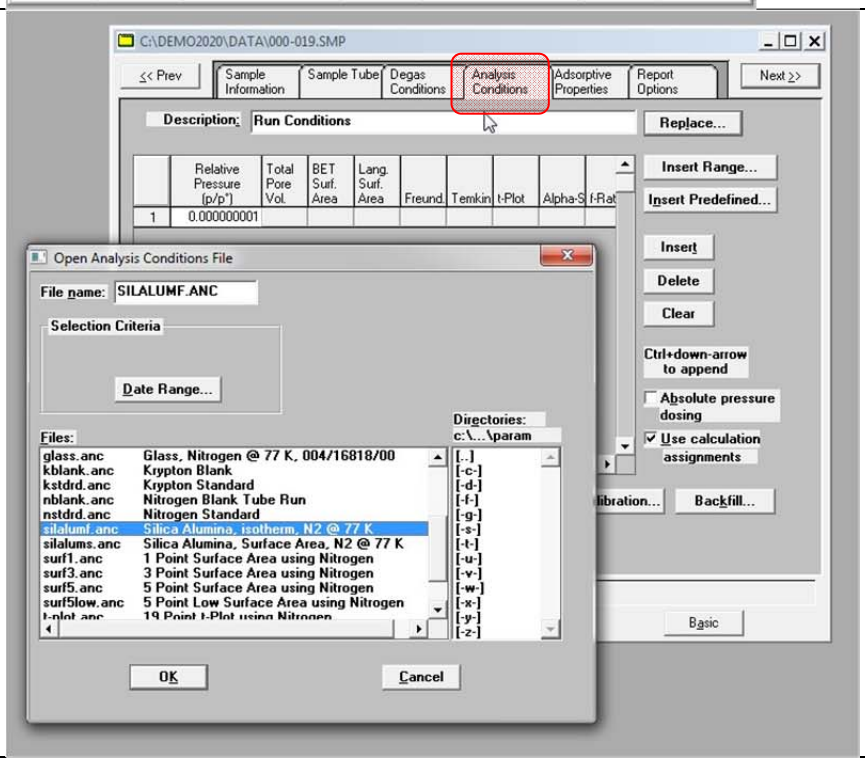
button. This allows the user to select files by all dates or a date range.
F2 clears the date
F3 inserts current date
F4 displays a calendar for a date selection.
selection by all dates or a date range.

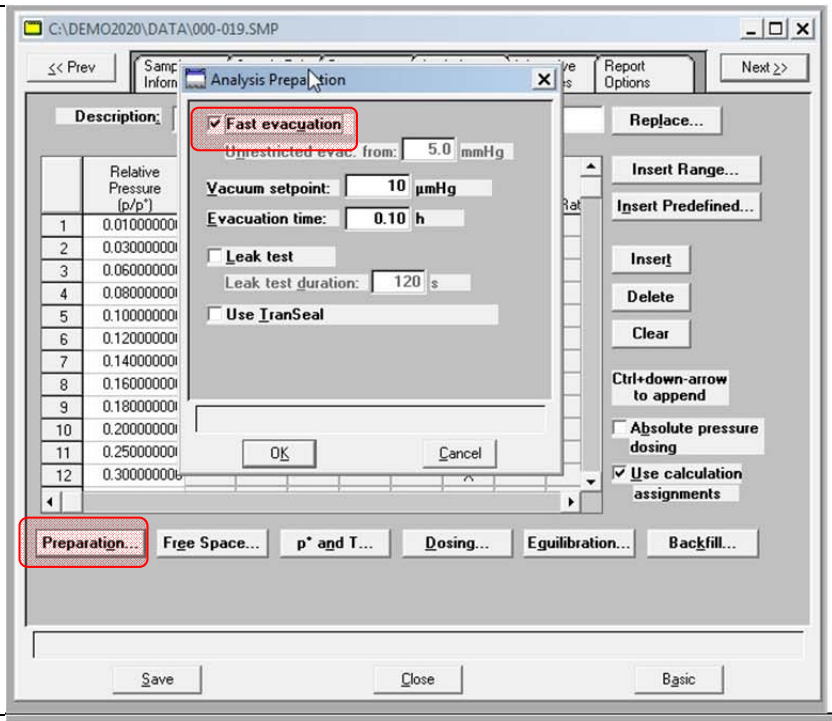
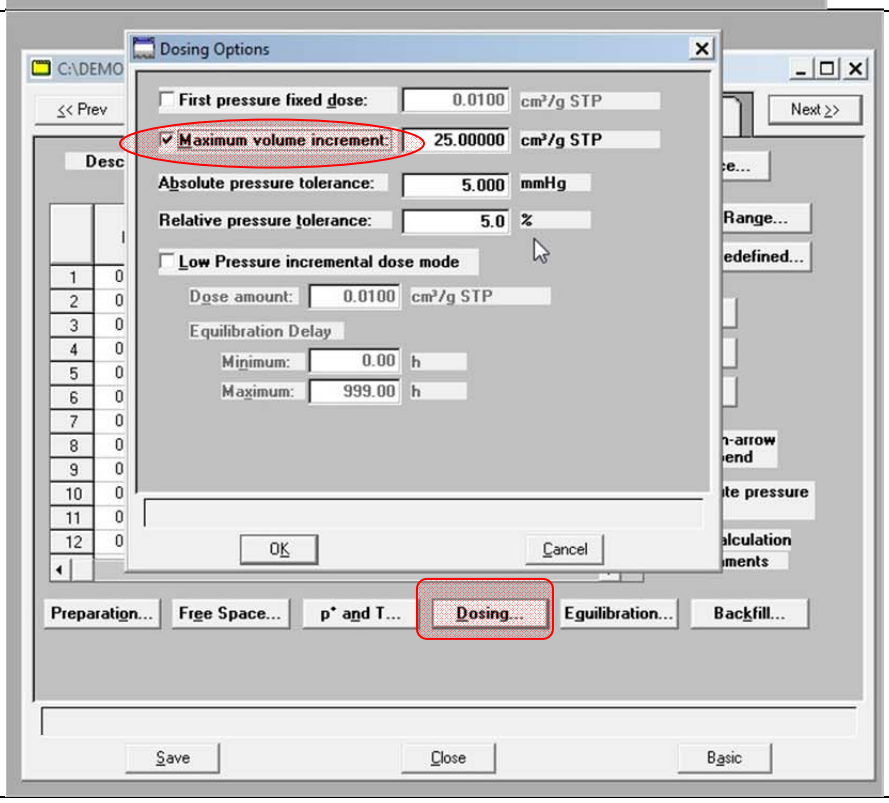


After looking at these options, click the **Cancel** button to return to the previous dialog box. This will allow you to *NOT* save your changes in this case. Click the **OK** push button to open this sample file and make the following changes.

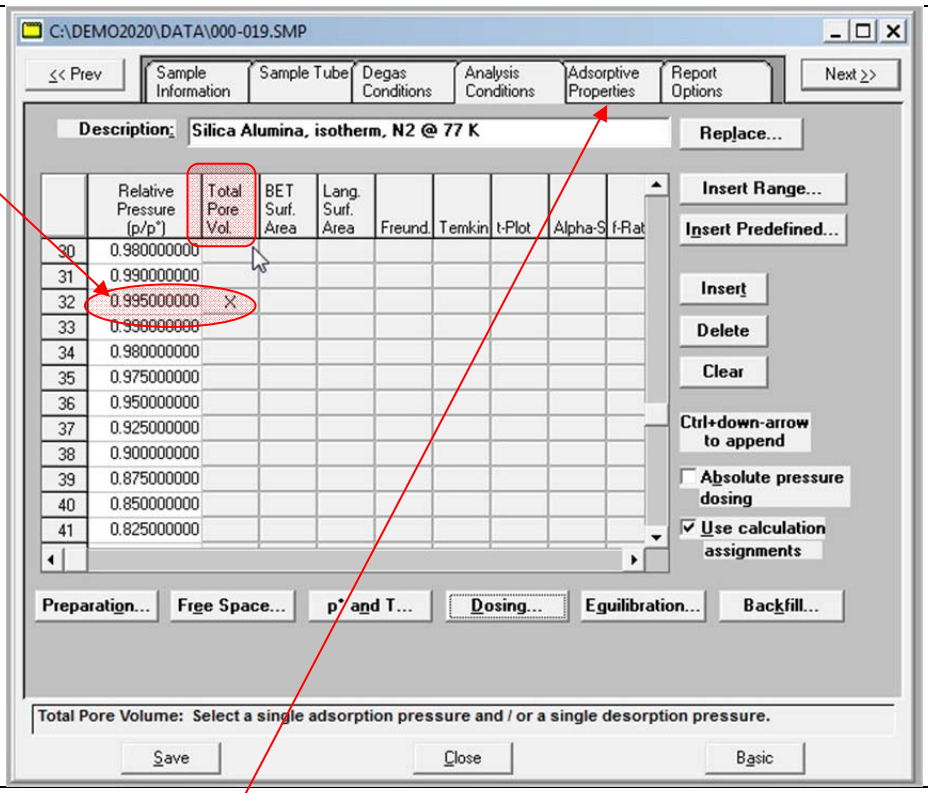
This time, type Si-Al Reference Material for your sample identification. It is also a good practice to add your sample tube and/or stopper identification to this field. You may enter your initials as the Operator. You may leave "Submitter" blank if you choose. Please leave the Mass as the default number. (1.0000) We will change this later. Also note that when you click in the Mass field, you get a message at the bottom of the screen describing the range for that entry.



Select the Degas Conditions menu. Enter the following information into the specified fields.	
Click on the Analysis Conditions tab. Select the Silica Alumina, isotherm, N2 @ 77 K analysis conditions set. This allows selection of preprogrammed analysis parameters.	

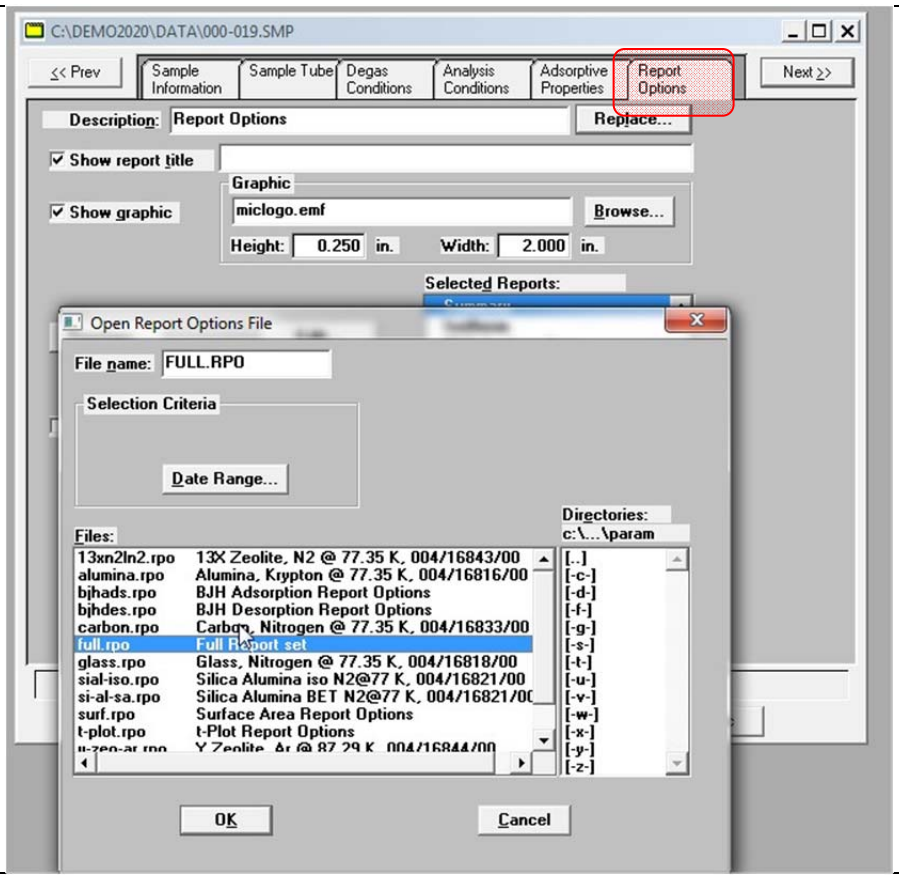
Before you start the analysis, please make the following changes to the default values. Under Preparation, select Fast Evacuation since this sample will not fluidize.	
Under Dosing, select Maximum Volume Increment and make sure the value is set to 25.0 cm3/gm STP.	

Finally, select the 0.995 P/Po data point for Total Pore Vol. Please note that you may have to click on the current value in order to deselect it first. All other Analysis Conditions can be left as their default values.



The next tab is for the Adsorptive Properties. Adsorptive properties are used to define adsorptive gas characteristics for the analysis gas. No change needs to be made here since we will use nitrogen as the analysis gas.

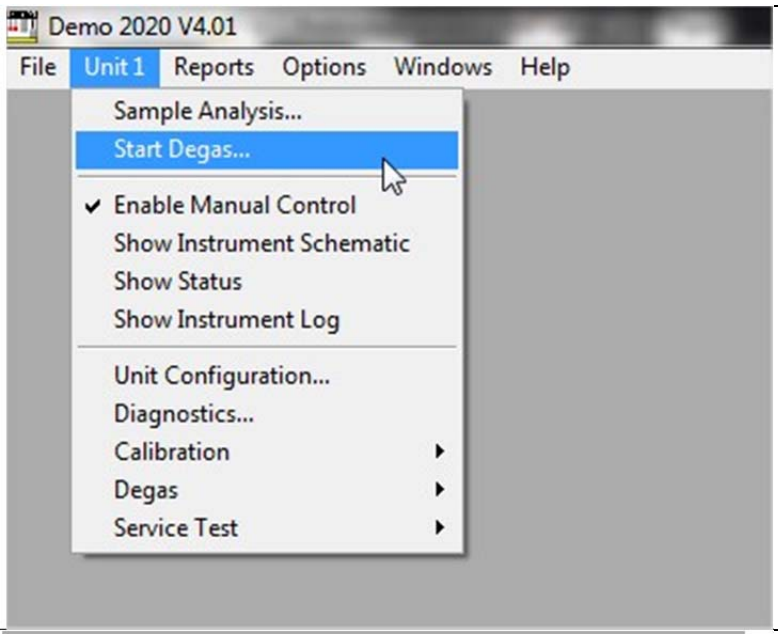
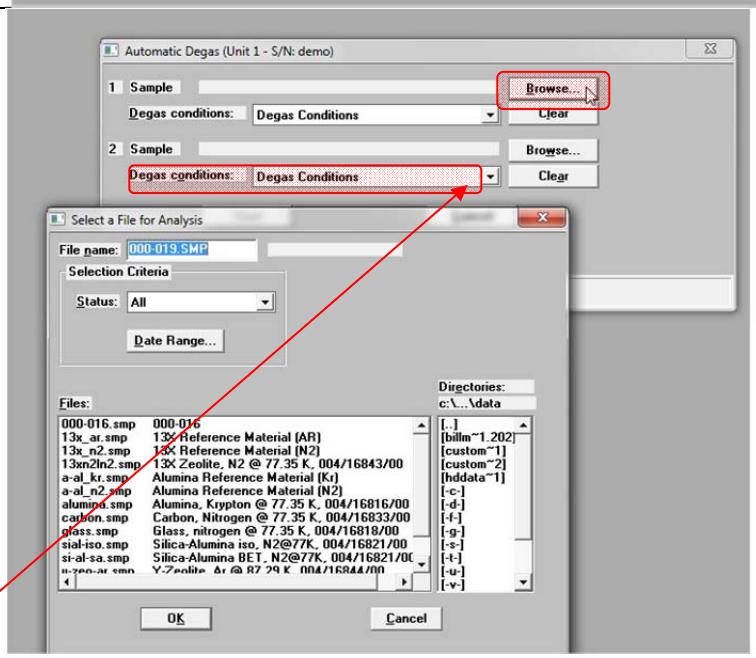
The last part of the sample file is used to select the Report Options. Click on the **Replace** and select the Full Report Set.



We are now finished with programming this sample file. Review the conditions you have enetered, and be sure to ask your instructor if you have any questions. If not, click the **Close** and **Yes** buttons to save the file.

TRAINING ACTIVITY – Start Degas

If both Carbon and Silica-Alumina samples are loaded onto their respective degas ports and both sample files have been created, you may initiate the degas process for these samples. Please perform the steps below to do so.

Select Start Degas… under the Unit 1 menu.	
For the appropriate ports, click Browse... and select the file you created for each sample. The instrument will use the degas conditions you programmed into these files. If you wanted to change these conditions or if you had not already programmed a sample file, you could select conditions using the Degas Conditions pull-down menu seen here. We will not be utilizing that feature in this case.	

Once you have selected the appropriate files, click Start and begin the degas process for both samples.

Training Activity – Filling Analysis Dewar

Please read the Precautions below and then follow the instructions for filling an analysis dewar. Your instructor will show you how to operate the nitrogen Pump provided.

Precautions

Always handle Dewars with care. Any product with a glass vacuum flask is a **potential safety hazard and should be treated with caution**. Micromeritics recommends that the following safety practices when handling Dewars containing liquefied gases:

Protect yourself by wearing goggles (or a face shield), an insulated or rubber apron, and insulated gloves.

When pouring liquefied gases from one container to another:

- cool the receiving container gradually to minimize thermal shock.
- pour the liquefied gas slowly to prevent splashing.
- vent the receiving container to the atmosphere.

Use a plastic stirring rod when stirring substances in a Dewar containing liquefied gases (or other materials of extremely low temperature). Do not use a glass or metal stirring rod unless it is coated with some form of protective coating.

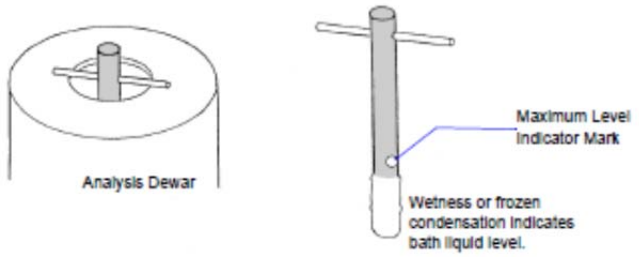
Do not handle heavy objects above the Dewar. If unavoidable, place a protective cover over the Dewar's opening. If an object of sufficient weight is accidentally dropped into the Dewar, shattering may occur.

Always install the Dewar cover before performing an analysis. The cover reduces the accumulation of ice. Accumulated ice could cause the Dewar to bond to the sample tube.

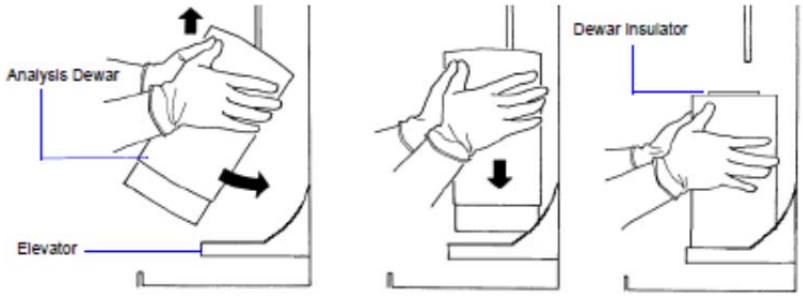
Using the Analysis Dewar

Fill the analysis Dewar with the analysis bath fluid to about 5 cm (2 inches) from the top. Incorrect fluid levels can lead to measurement errors. Do not overfill the Dewar.

Check the analysis bath fluid level with the dipstick as shown below.



Insert the analysis Dewar onto the elevator as shown in the following illustration.



4. Place a Dewar insulator over the open Dewar until you are ready to start your analysis; this helps to minimize ice accumulation.

5. When you are ready to start the analysis, remove the insulator and install a Dewar cover.

NOTE: A cold trap dewar is also part of the 2020 Physi instrument set up. For our reference materials during this class, they are not necessary. For unknown samples, and for general laboratory analyses, it is best to always use the cold traps which will prevent contaminants from the instrument's degas or analysis from damaging the system.

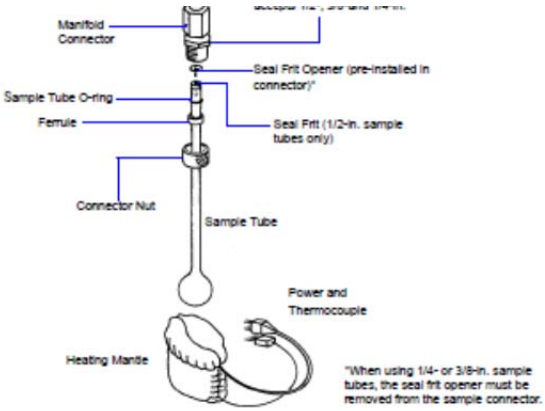
LAB Session 3 - Initiating carbon analysis

Training Activity – Unloading degassed sample

The next step in our analysis process is to unload the carbon sample we have degassed.

Transferring the Degassed Sample to the Analysis Port

Please take a moment to study the following drawing and instructions before your instructor demonstrates how to move a sample from the degas port to your sample port.



The sample tube assembly must be removed from the degas port, weighed and then installed onto the analysis port in order to perform an analysis. The following steps should be followed to accomplish this.

1. Allow the sample tube to cool. Please use caution when removing sample tubes and clips, if they are not allowed to return to room temperature, they can be very hot!

2. Carefully remove the heating mantle clip and the heating mantle from the sample tube and allow the sample tube to cool to room temperature (approximately fifteen minutes).

a. Please use a gloved hand to support the sample tube by the stem when removing heating mantles. The quartz tubes can/will break if not handled correctly.

3. While holding the sample tube, loosen the port connector nut and remove the sample tube from the degas port. If you are not using a seal frit, insert a stopper immediately. For this class all samples will use seal frits.

4. Remove the connector nut, ferrule, and O-ring from sample tube stem. See above drawing.

5. Weigh the sample tube assembly. Enter the weight on the Sample Data Worksheet as Mass of sample tube plus sample (After Degas).

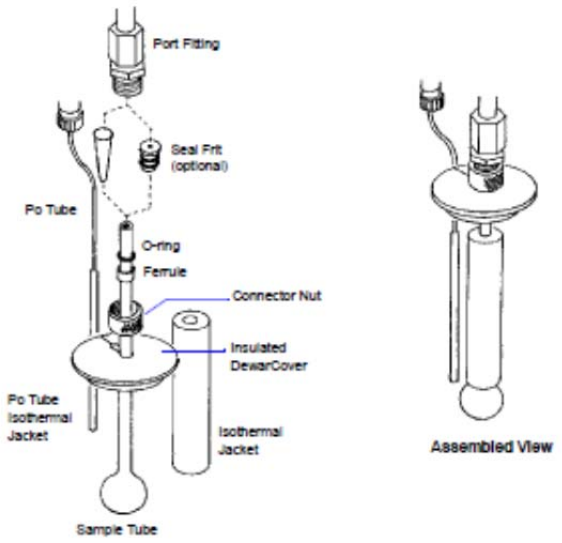
6. Subtract the Mass of empty sample tube (Before Degas) from the Mass of sample tube plus sample (After Degas) to determine the mass of the sample. Record this value as the Mass of sample (After Degas).

7. Slide an isothermal jacket down over the sample tube stem until it touches the sample tube bulb.

8. Place the connector nut, ferrule, and O-ring onto the sample tube stem.

9. Remove the stopper and immediately attach the sample tube to the analysis port, pushing it fully up into the port. Secure it in place by screwing the connector nut onto the analysis port connector and hand-tighten the connector nut.

10. Place the sample tube Dewar cover over the sample tube stem just above the isothermal jacket as shown in the following illustration.



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NOTE

If a sample is unloaded hot (assuming precautions to avoid burning were taken) and weighed, the mass will be lower than it should be. This is the result of the volume of air in the tube being less than when the tube was weighed at room temperature and the glass of the tube being dryer. Both of these would combine to reduce weighing accuracy. When helium is used as the degas backfill gas, care must be taken to either pre-fill the sample tube with helium before the initial weighing or use a buoyancy correction factor. Just think of a helium balloon.

We have now completed all the preparatory steps necessary to enable us to begin the carbon sample. One step remains, starting the analysis.

LAB Initiate Carbon Sample

Now we will begin the analysis. From the Main Menu Bar select Unit 1. Select Sample Analysis … and then file CARBON.smp. Select to generate a Report After Analysis. Make sure that Screen is selected for Destination:

Press **Start** to begin the analysis. Tomorrow morning we will briefly go over the reports.

LAB Session 5 - Preparing Y-Zeolite for analysis

LAB Weigh, create file and degas Y-Zeolite

Please measure out approximately 0.15 grams of Y-Zeolite.

Since we have one scale, please continue on to the next section and start the Y-Zeolite file creation if you are not using the scale.

Remember that you may use the After Degas value (Step 5) or the After Analysis value (Step 7). Please record both values and check to make sure that they are similar. This serves as an accuracy check for sample weighing.

Again, compare the sample mass obtained after analysis (Step 7) with the sample mass after degas (Step 5).

Sample tube:_____ Sample: _____Y-Zeolite_____

Before Degas:

Mass of empty sample tube assembly (sample tube, seal frit and filler rod) _____ g

2. Mass of sample tube assembly plus sample _____ g

3. Mass of sample (Step 2) – (Step 1) _____ g

After Degas:

4. Mass of sample tube assembly plus sample _____ g

5. Mass of sample (Step 4) – (Step 1) _____ g

After Analysis:

6. Mass of sample tube assembly plus sample _____ g

7. Mass of sample (Step 6) – (Step 1) _____ g

TRAINING ACTIVITY – Y-ZEOLITE DEGAS CONDITIONS

We have created a complete sample file and performed an analysis with the information entered. The ASAP has the capability of using many different analysis conditions. A frequently analyzed sample could use the same parameters such as fast evacuation, leak test and rate, equilibration interval, and analysis pressure points, etc. Once defined, they can be quickly recalled for use in an analysis.

One submenu of the File menu manages these for you. We will now perform an exercise that will give you experience entering and manipulating these analysis conditions.

It is helpful to build Degas Conditions, Analysis Conditions and Report Option Sets to apply to the majority of sample types analyzed in your lab. That way creating a sample file would be as simple as entering the information on the first page of the sample file and choosing the ANC and RPO sets to insert. This makes for simple file information management and frees you to perform other tasks.

LAB CREATING A DEGAS CONDITIONS FILE for Y-Zeolite

In order to simplify the operation where multiple analyses of the same sample material regularly take place the stored Degas Conditions can be used. We will now create a Degas Conditions file.

You should be more familiar with the software structure now so this section will not use as many screen shots. If you have difficulty finding a section, please ask your instructor.

From the File menu, select Open then Degas conditions.... Name the file yzeolite.deg.

Press **OK** followed by **Yes** to create this file.

Enter the following information into the specified fields. Enter Y Zeolite for the Description of this file.

For the Evacuation Phase

Temperature ramp rate: 10.0 °C/min

Target Temperature: 90 °C

Evacuation Rate: 10.0 mmHg/s

Unrestricted evacuation from: 10.0 mmHg

Vacuum setpoint: 500 µmHg

Evacuation time: 60 min

For the Heating Phase

Ramp rate: 10.0 °C/min

Hold temp: 350 °C

Hold time: 480 min

Press [Close] then [Yes] to store the file when finished. If you encounter any problems or have questions about this exercise, check with your instructor.

Place your Y-Zeolite sample tube onto your degas port and begin degassing when you are done.

LAB Session 6 - Initiating Silica Alumina analysis

Training Activity – Silica Alumina Sample Initiation

We have already created the sample file for the sample we will run overnight. Before we leave today we have to cool down, unload, weigh the sample and start the Silica Alumina file. Please refer back to the ASAP Series Sample Data Record for Silica Alumina on page 6 and enter the remaining weights into that sheet. Remember to top off the Dewar and select screen as the report destination.

DAY 2 - MICROPOROUS SAMPLES AND SAMPLE PREP

LAB Session 1 - Building Y-Zeolite Sample file

Training Activity – Analysis Conditions for Y-zeolite

The Y-Zeolite sample prepped overnight so it should be ready for analysis. This section will guide you through setting up the analysis file.

LAB – Creating a Y-Zeolite Analysis File

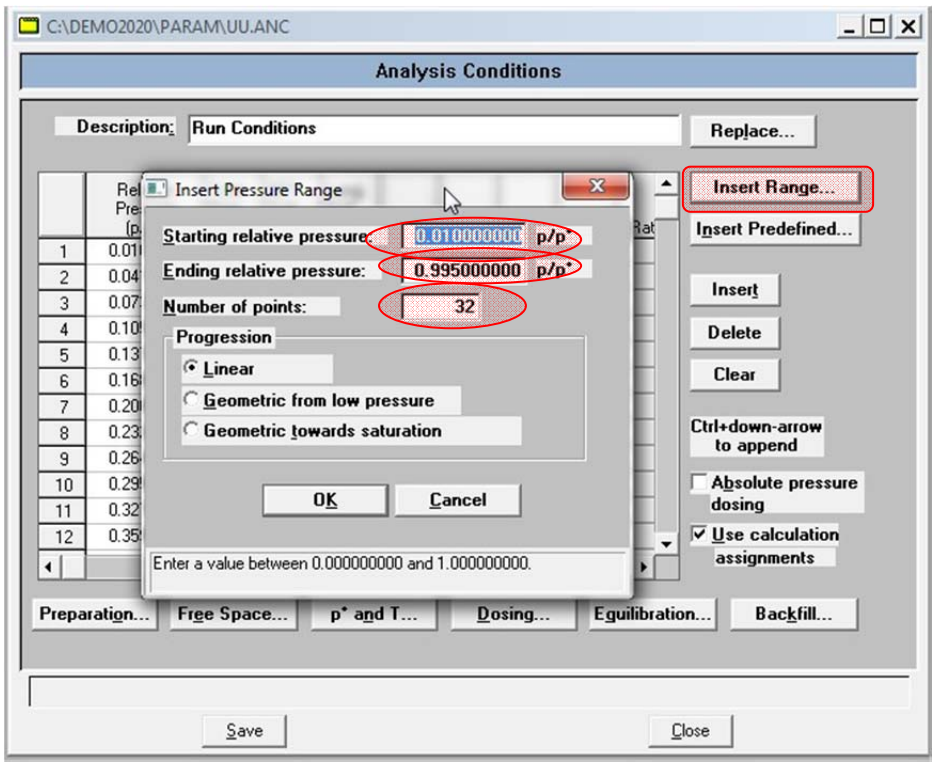
When multiple analyses of the same sample material regularly take place another useful parameter set is the Analysis Conditions file. We will now create an Analysis Conditions file.

From the File menu, select Open then Analysis conditions.... Name the file micropor.anc. Press [OK] followed by [Yes] to create this file.

Enter Y Zeolite Micropore Analysis for the Analysis conditions description:. Selecting the appropriate fields, enter the remaining information from the following table. For each section, click on the corresponding button in the analysis conditions dialog box. Clear the pressure table before entering the pressure range. If a field is not included in the table, we will use the default value.

Pressure table: Use 32 points from P/Po = 0.01 to P/Po = 0.995

Click on Insert Range and enter values



Preparation... button:

Fast evacuation Yes

Evacuation time 0.0 hours

Leak test No

Use TranSeal No

Free Space... button:

Free space Entered

Warm free space 28.0000*

Cold free space 88.0000*

Po and T... button:

Option1

Measurement Interval: 120 min

Dosing... button:

First Pressure Fixed Dose No

Maximum volume increment No

Absolute pressure tolerance 5 mmHg

Relative pressure tolerance 5%

Low pressure dosing Yes

Dose amount3.0 cm3/g

Equilibration... button:

Equilibration interval 30 sec

Minimum equilibration delay 600 sec

Backfill... button:

Backfill at start of analysis No

Backfill at end of analysis Yes

Backfill Gas N2

* The actual free space values will be taken from your free space analysis.

Press Close then Yes to store the file when finished. If you encounter any problems or have questions about this exercise, check with your instructor.

Training Activity – Report Options for Yzeolite

Another of the information files that may be inserted into the Sample Information file is the Report Options file. This file specifies the report name and the method of reducing and reporting data, including graphs, plots and reports.

As with other file types, Report options are entered from the File, Open menu. At this time we will create a report option.

LAB - Creating a Y-Zeolite Report Options File

Using the computer and the previously loaded software, create report options as described. Select File, Open, Report options... and enter yzeolite.rpo as the file name. Choose [Yes] to create the file. At this point the dialog box displayed is similar to other file management screens for sample information and analysis conditions.

Select Description: and enter Y Zeolite Micropore. Select Show Report Title: and enter ASAP 2020 OPERATOR TRAINING. The remainder of the screen shows the available report types. Once selected, the reports can be edited to configure the form of the report.

Select the Isotherm report, the BET Surface Area report, the Horvath-Kawazoe report, and the Summary report. Select the report by Double-clicking the report in the Selected Reports list. If a check appears beside the report, it is already selected. Selected reports can be deselected by double clicking again. Make sure that only the reports specified above are selected. Finally, check the box to Apply thermal transpiration correction.

Save the changes made and close the file. We will use them later.

Training Activity – Measuring Free Space of a Microporous Material

Due to the entrapment of helium by microporous materials, special care must be taken to obtain free space values before a micropore analysis. If free space is measured

directly before the analysis as usual, helium will be left behind in the micropores and lead to erroneous data during isotherm collection.

To properly obtain free space values for our Y-Zeolite material, we wil:

1. Measure free space independently BEFORE the micropore analysis.
2. Re-degas the material on the sample port. *
3. Proceed with the micropore analysis.

degassing materials on your instrument's sample port is NOT recommended. For a **second degas of a reference material, there is no change of instrument contamination. With all other samples, degassing should only be performed on the degas ports.*

Normally, an analyst would measure the free space of an empty sample tube prior to running an analysis on a microporous material. In order to fit as much content as possible into this course, we will use this procedure instead.

To independently measure free space, you will need to create a sample file that collects *just one* relative pressure point at 0.1 P/Po. The ASAP 2020 will then measure the free space of the sample material before measuring the single point. This quick sample file provides us with the free space values (warm and cold) that are necessary to enter into the subsequent micropore sample file.

LAB Session 3 -Y-Zeolite second degas

Training Activity –Degassing on the sample port (2nd degas)

Once the single point analysis used to obtain free space values is complete, we must re-degas the sample on the sample port. Follow the guidelines given by your instructor to re-degas your Y Zeolite sample.

Once this exercise is complete, you may create a sample file using micropor.anc and yzeolite.rpo to run the micropore analysis. Please flag your instructor when your sample is ready to begin for instructions on entering free space values into the sample file.

LAB Session 4 - Creating entire Alumina sample file

Training Activity –Creation of complete Alumina sample file and sample degas from scratch.

You will now use the previous day(s) activities to measure a sample, create a sample file and prep a low surface area Alumina sample.

Low surface area samples below $1m^2/g$ should use krypton gas.
Nitrogen has a saturation pressure of 760 torr whereas krypton has a saturation pressure of only 2.5 torr. Since pressure is proportional to the number of moles or molecules, there are ~ 300 molecules of nitrogen for every 1 molecule of krypton. Think of it like this: If you have an office party with 1200 people and 2 people sneak off, the boss will probably not notice. If you have a meeting with 4 people, and two people sneak off, the boss will *not* be happy.

A low surface area, BET Multipoint Surface Area file should be created and run.
All Micromeritics Reference materials come with a booklet that will tell the operator what degas conditions to use. This is what the Alumina Booklet looks like:

Alumina

Type of Analysis:
Specific Surface Area

Instruments:

ASAP Series	Gemini Series
FlowSorb	Pulse ChemiSorb

Test Amount:
2 to 3 grams

Preparation:

Heat to 400 °C at 10 °C/min; hold for one hour.

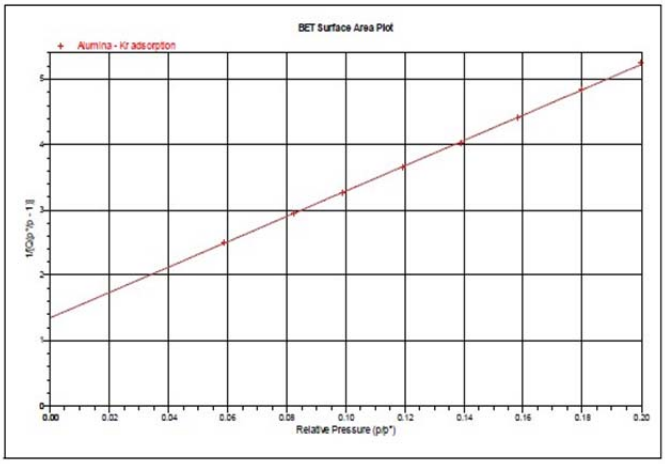
NOTE: *This material requires careful, precise sample preparation for consistent results. Use appropriate heater block inserts for FlowPrep and VacPrep.*

ASAP or SmartPrep: use 10 °C/min ramp rates.

Test Conditions:
–Use 10-sec equilibration interval.
–Collect at least 5 points between 0.06 and 0.20 relative pressure
Gemini Series only:

Evacuation time:	5 min
Evacuation rate:	500 mmHg/min
Equilibration time:	5 sec
Free space:	Measure

A typical BET equation using krypton gas is shown below.



Please use the “Test Amount”, the “Preparation” or degas conditions and the “Test Conditions” to create and degas your Alumina sample file. There are a few properties that are not mentioned in the reference material booklet because the complete files are included with the 2020 Physi software. Please enter as many properties as you can into your analysis, degas and report options files and then compare them with the Alumina files that are already saved in the software.

Again, compare the sample mass obtained after analysis (Step 7) with the sample mass after degas (Step 5).

Sample tube:_____ Sample: _____Alumina_____

Before Degas:

Mass of empty sample tube assembly (sample tube, seal frit and filler rod) _____ g

2. Mass of sample tube assembly plus sample _____ g

3. Mass of sample (Step 2) – (Step 1) _____ g

After Degas:

4. Mass of sample tube assembly plus sample _____ g

5. Mass of sample (Step 4) – (Step 1) _____ g

After Analysis:

6. Mass of sample tube assembly plus sample _____ g

7. Mass of sample (Step 6) – (Step 1) _____ g

DAY 3 - INDEPENDENT ALUMINA ANALYSIS

LAB Session 1 - Analyzing an Alumina sample

Training Activity – Analysis of a low surface area Alumina sample with krypton

Your Alumina sample should be ready now after an overnight prep. Please load the sample onto the analysis port, top off the analysis dewar, and alert your instructor *before* you initiate the analysis. The class will observe one analysis for an explanation of the analysis steps.