

Free-Space Gas Adsorption Analyses

Many porous materials, such as zeolites and activated carbons, trap and hold helium in their complex pore structures for many hours after being exposed to helium. Helium trapped in pores can interfere with the analysis at low pressures, causing an “S-shaped” curve at the lower end of the isotherm. For this reason, it is recommended that you *enter* the warm and cold free-space volumes when performing gas adsorption analyses, thereby avoiding exposure of the sample to helium.

The *entered* method is to perform a short analysis on the sample after partial degassing (one pressure point with no incremental dosing), but prior to final sample preparation. Measure the free space during this analysis. The measured free-space values will be printed on the report and may then be entered into the sample file after more thorough sample preparation.

- Create a new sample file and under the Analysis conditions tab select Free Space and change the selection from measured to entered. (Images 1 and 2)
- Degass the samples and run the analysis again
- Create a second sample file and under analysis conditions change the analysis conditions to a single point analysis and make sure the Free Space is set to measured. (Image 3)
- Start the analysis with the sample as is in the sample tube without degassing or removing the sample.
- When the second analysis is complete, report the results and open the first sample file and in the Free Space options of the first sample file enter the Warm and cold free space from the results of the second analysis. Save the file and generate the report.
- If the slope and results are still negative, use the calibrated method, this is described in the operators manual

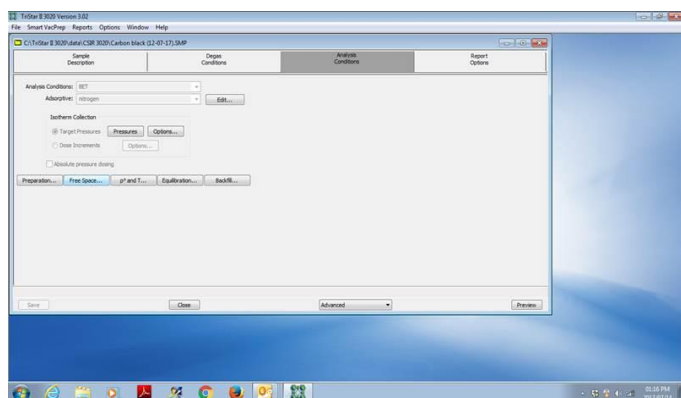


Image 1

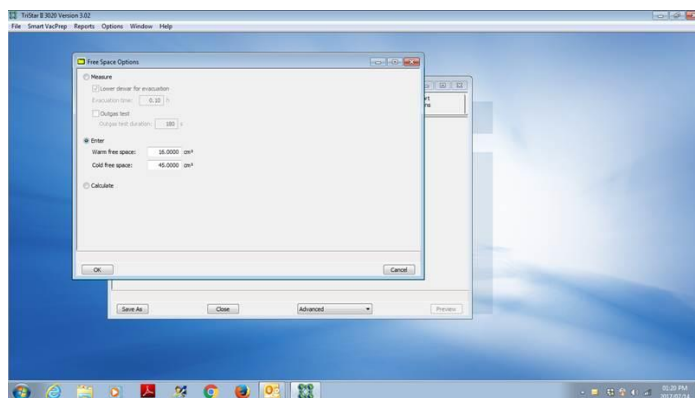


Image 2

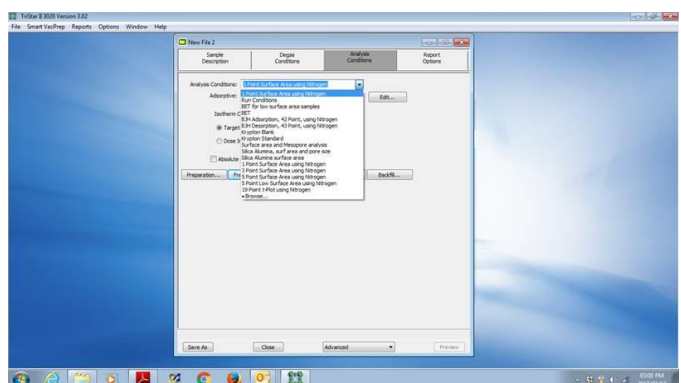


Image 3

The following recommendations should be followed to improve the quality of your Gas adsorption analyses.

- Be consistent in using seal frits; for example, use the same seal frits for the analysis as you did for the empty tube test.
- The cold free space is dependent on the adsorptive and the bath temperature; perform a test for each bath temperature (for example; liquid nitrogen, liquid argon, or others) to be used.
- Please use filler rods and isothermal jackets. The top of the isothermal jacket must be in the same position for the sample analysis as it was for the empty tube test.
- Uptake - Y axis is low against a low sample mass and a large free space = huge free space error. Use more sample and / or move to a smaller tube. As an example, going from a 3/8" tube to a 1/4" tube reduces your free space error by 70%! Free space is your enemy!
- Don't use fast evacuation - you should only use this with 'large' solid pieces - never with powders. With the filler rod in place you create an annulus and the draw up the tube is very high.
- The IUPAC recommendation on specific area is 30 m² in the tube for BET and 50 m² for porosimetry. Some customers can't do that due to bulk density, the TriStar (with a 3/8" tube) will work well down to 0.6 m² - but more is better for sure.