Application Note 73

Degas Backfill Gas Selection for Micromeritics Gas Adsorption Instruments

Introduction

Specific surface areas and pore volume distributions are often determined using gas adsorption techniques. Prior to analysis, any adsorbed gas or vapor phase (such as water or other volatiles) should be removed from the sample. This process is often referred to as degassing the sample.

Degassing usually involves either heating the sample or flowing an inert gas across the sample during evacuation. In either case, molecules being desorbed from the surface of the sample are removed from the sample tube. After degassing, the sample tube can be sealed and removed in one of the following conditions:

- **Under vacuum** using the TranSeal™, an apparatus which is inserted into the sample tube. It is designed to close when removed from the degassing port and open automatically when installed on the sample port, maintaining a vacuum-tight seal. Request Product Bulletin No. 86 for additional information.

- **Backfilled**, using the seal frit or rubber stopper provided in your instrument accessories kit, or the TranSeal.

Nitrogen is often used as the backfill gas because it is readily available, inexpensive, and works quite well for many applications. Nitrogen, however, is unsuitable for materials such as carbon and microporous samples containing pores of less than 2 nm in diameter. These type of materials adsorb nitrogen at room temperature. And, since adsorbed gas is one type of contaminant that you wish to remove during degassing, using nitrogen as the backfill gas partially defeats the purpose of degassing. Obviously, nitrogen is not a true inert gas when it comes to microporous materials.

Therefore, for materials which tend to adsorb nitrogen, helium is a better choice as the backfill gas. Helium adsorption at room temperature is negligible, even in the high energy pores of most microporous samples.

There are some highly microporous materials in which helium (if used as the backfill gas) is extremely difficult to remove due to diffusion. Complete removal requires great care as well as time-consuming tasks. Otherwise its presence can severely distort an adsorption isotherm.

For samples where neither nitrogen nor helium are ideal, a vacuum-sealed transfer is the most desirable method for transferring the sample to the analysis port.

Weighing the Backfilled Sample Tube

If using helium, remember that it has a lower molecular weight than nitrogen and, therefore, is a less dense gas. When placed on a laboratory balance, a sample tube filled with helium weighs less than a sample tube filled with nitrogen. Care must be taken when weighing tubes containing degassed samples backfilled with helium.

Normally, sample weight is determined by (1) weighing the empty sample tube, (2) weighing the

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tube after adding the sample, and (3) calculating the difference between the two. The tube must be filled with the same gas both times it is weighed (with and without the sample). If not, there will be an error in the sample weight due to the buoyancy created by the density difference between the two gases. The error is negligible when the density difference is small, such as between air and nitrogen. However, the density difference between helium and nitrogen can be quite noticeable. For example, if you use nitrogen to weigh the empty sample tube and helium to weigh the sample tube with sample, you can get an error of as much as 0.02 gram. Often only 0.2 gram of sample is used when analyzing micropores; therefore, a 0.02-gram error represents a 10% error in the sample weight.

There are two methods for avoiding this problem.

Method 1

1. Evacuate the empty sample tube on one of the degas ports; then backfill with helium.
2. Quickly cap or stopper the sample tube to avoid loss of helium.
3. Weigh the empty sample tube.
4. Add sample to the sample tube; then degas the tube.
5. Weigh the tube containing the sample (backfilled with helium).
6. Subtract the empty tube weight (step 3) from the weight of the tube containing the sample (step 5) to obtain the weight of the sample.

NOTE: This method must be used with samples analyzed on the ASAP 20xx series micropore analyzers if helium is connected to the Backfill inlet.

Method 2

1. Weigh the empty sample tube containing air.
2. Add the sample to the tube; then degas and backfill with helium.
3. Weigh the sample tube containing the sample.
4. Subtract the empty tube weight (step 1) from the weight of the tube containing the sample (step 3) to obtain the weight of the sample.

Remember, this weight is incorrect by as much as 0.02 gram. At the end of the analysis:

5. Reweigh the sample and tube because the tube is filled with nitrogen (analysis gas) at the completion of the analysis.
6. Subtract the empty tube weight (step 1) from the after analysis sample-plus-tube weight.
7. Enter the corrected sample weight (step 6) and generate the analysis report.

The true sample weight is slightly overestimated by this second method. It will be incorrect by the weight of the nitrogen adsorbed on the surface at the end of the analysis. (Remember, it is because of room temperature adsorption of nitrogen that helium is being used as the backfill gas.) The weight of nitrogen adsorbed is proportional to the weight of the sample analyzed. Using more sample does not reduce the effect of this error.

While the first weighing method is more accurate than the second, it is more difficult to perform. In some applications, the improvement in accuracy is worth the extra time investment. In others, time may be more important than the small improvement in weighing accuracy. Regardless, never subtract nitrogen-filled tube weights from helium-backfilled sample-plus-tube weights unless enough sample is being analyzed such that a 0.02 gram weight error is acceptable.