Application Note 112

Optimize Free-Space Correction for Low Surface Area Samples Using the Gemini Analyzer

The unique balanced measurement method used in the Gemini permits small amounts of surface area to be measured with nitrogen gas that otherwise would be measurable only with krypton. Low surface area samples often displace many times more nitrogen than they adsorb, especially if composed of low-density materials of large particle size. The standard, built-in, helium free-space difference measurement and mathematical compensation routine typically can remove the effects of more than 99% of this sample displacement, but the small amount remaining uncompensated can still be significant.

A technique for adding a compensating volume with negligible surface area into the balance tube has been developed. It can reduce the size of the initial imbalance to low levels and allow precise measurement of adsorbed gas. Either straight-wall or bulbous sample tubes may be used. Hanging filler rods are recommended but not required.

1. Load the sample tube with an appropriate quantity of sample.

2. Load the balance tube with glass beads that have a total volume approximately the same as the sample volume.

   a. Determine the volume (v) of the sample in cm^3:

   $$v = w + \rho$$

   where

   w = mass of sample (g)
   ρ = density of sample (g/cm^3); if density is unknown, refer to your laboratory handbook

   b. Determine the number (n) of glass beads needed to equal the sample volume:

   $$n = v \div 0.014\, cm^3$$

   where

   $0.014\ cm^3$ = approximate volume of one bead

3. Outgas the sample in the sample tube at an appropriate temperature for an appropriate amount of time.

micromeritics®
One Micromeritics Drive, Norcross, Georgia 30093
T. (770) 662-3620 www.micromeritics.com

4. Install the sample tube (containing the outgassed sample) onto the analysis port and the balance tube (containing the glass beads) onto the balance port. Use hanging filler rods for best results.

5. Set up the Gemini for a one-point measurement (P/Po = 0.05 to 0.1) so that the initial free-space measurement can quickly be determined; then perform the measurement.

6. Using the “measured free space” absolute value and the following relationship, determine the mass of glass beads to remove from (or add to) the balance tube to reduce the free-space imbalance:

$$\frac{free\ space\ cm^3\ \ x\ 2.515\ g\,/\,cm^3}{3.53} = mass\ of\ \ glass\ beads\,(g)$$

where

2.515 g/cm^3 = density of glass beads
3.53 = thermal correction (no units)

Note that the volume of one glass bead is approximately 0.014 cm^3. Therefore, if the measured free space is less than 0.02 cm^3, it is unnecessary to correct the free space.

7. Use a beaker of warm water to bring the balance tube to room temperature before removing it from the balance port of the Gemini to remove (or add) glass beads. This prevents condensation of moisture from the laboratory atmosphere onto the cold glass beads.

8. Remove the balance tube:

   - If the measured free space is negative (–), add the calculated mass of glass beads into the balance tube.

   - If the measured free space is positive (+), remove the calculated mass of glass beads from the balance tube.

9. Reinstall the balance tube onto the balance port of the Gemini analyzer and proceed with the analysis.

For subsequent samples of the same material, you may simply use the same weight of sample that was used for the initial sample so that the original bead quantity may be left undisturbed on the balance port.