



PROCEDURE FOR BET ANALYSIS

Preparations for Sample Analysis

1. Take liquid nitrogen from Chemical Department (Next building) (Just write the volume – 4L, and write Biswas on the log notebook)
2. Fill **Cold Trap Dewar** with liquid nitrogen (3/4)
3. Cold trap dewar is then mounted to **Autosorb-1**
4. Open He and N₂ cylinder valves and ball valves located after pressure regulators. Confirm cylinder pressure regulators are set to **10 psi**
5. Operator must sign in log book

Degassing Samples Prior to Analysis

1. Choose appropriate cell size for sample (6, 9 or 12mm)
* We only have one cell (9 mm) with fill glass rod for sample.
2. Using a 5-place analytical balance, weigh and record the weight of the empty sample cell with fill glass rod.

(Weigh 3 times for each sample and then calculate the average) Record this as the **initial weight**.
3. Remove fill glass rod and insert sample (powders) into the glass cell using a **funnel** (½ to full volume of the cell will be appropriate)
4. Replace the fill glass rod into cell
5. Weigh the sample cell with the glass rod and sample (Weigh 3 times and then calculate the average) Record this weight as the **Weight before Degassing**. Calculate the sample weight by subtracting the **initial weight** from the **Weight before Degassing**.
6. Put the cell into a **heating mantle** (pinch mantle to open the mouth) using a **clamp** to secure cell into mantle.
7. Attach the cell and mantle to the sample preparation station (There are two stations. We are now using **Station 2**)
 - a. First remove the **Knurled retainer ring, plunger with o-ring, and cell adapter** from the sample preparation station.

(Be careful! **Don't leave system open any longer than necessary!**)

- b. Place ring, adapter and o-ring onto sample cell
 - c. Insert the cell into the station hole and tighten the nut gently (**Tighten by hand only!**)
8. **Set temperature manually** (200 °C is normally used. 120 to 350 °C for water evaporation)

Turn heat on by lifting the **toggle switch** to the **up position**. (The green light is NOT ON at this moment. It starts to blink after degassing has been started.)

9. Degassing may take more than **1-2 hrs** depending on your sample conditions

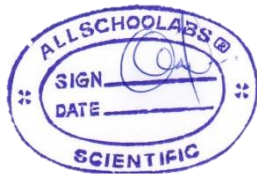
THEORY

The BET method is the most widely used procedure for the determination of the surface area of solid materials and involves the use of the BET equation.

$$\frac{1}{W[(P/P_0) - 1]} = \frac{1}{W_m C} + \frac{C - 1}{W_m C} \frac{P}{P_0}$$

in which W is the weight of gas adsorbed at a relative pressure, P/P_0 , and W_m is the weight of adsorbate constituting a monolayer of surface coverage. The term C , the BET C constant, is related to the energy of adsorption in the first adsorbed layer and consequently its value is an indication of the magnitude of the adsorbent/adsorbate interactions.

The BET equation requires a linear plot of $1/[W(P/P_0)-1]$ vs P/P_0 . which for most solids, using nitrogen as the adsorbate, is restricted to a limited region of the adsorption isotherm, usually in the P/P_0 range of **0.05 to 0.35**. This linear region is shifted to lower relative pressures for microporous materials.



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