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| Standard Operating Procedure for:  **Using the BET** | PPE required:        **Liquid N2** |
| **Introduction**  The BET (Brunauer, Emmett and Teller) measures the specific surface area of a sample– including the pore size distribution. The volume of gas adsorbed to the surface of the particles is measured at the boiling point of nitrogen (-196°C). The amount of adsorbed gas is correlated to the total surface area of the particles including pores in the surface. The calculation is based on the BET theory. Traditionally nitrogen is used as adsorbate gas. Helium is used to measure dead volume.  **Requirements: Before carrying out this technique you MUST have cryogen and gas cylinder training.** |
| **Prepare samples**   1. **Check the individual experimental risk assessment for risks from samples.** 2. Take clean sample tubes and bungs (including one tube to act as a blank), also, think about whether you want to include reference carbon black sample in your measurements. 3. Label the sample tube and then weigh an empty tube to 4 dp (without bung) using polystyrene holder and record the value. An anti-static device could also be used. 4. Add the sample (usually 0.5-2g – more sample for larger particle size) careful not to have sample on sides of tube, ideally the sample should not come more than 1cm up the side of the tube. You can use a long cotton bud to clean sides if necessary. 5. For surface area 5m2/g you need 2-3g 6. For surface area 10m2/g you need 2-3g 7. For surface area 30m2/g you need 0.5-1g 8. For surface are 60m2/g you need 0.25-0.5g   If unsure, assuming particles are spherical, can use equation:   1. Weigh the tube and sample together and record result. 2. Place clean bung in neck of tube to avoid contamination. 3. On the degasser, wipe the metal needle with methanol and allow it to dry. 4. Open the nitrogen cylinder and check that the pressure is at the mark on gauge. 5. On the degasser turn on the individual line switches for N2 gas. 6. Place tube in degasser and treat as appropriate. Minimum treatment is cold flow of N2 overnight (18 hours). Keep bung in during this process. Samples can be heated up to 300oC, but must remain 30oC below the sample decomposition temperature. 7. After degassing is finished allow the sample to cool. Make sure sample is not left long before testing. Sample should be left with N2 flowing to reduce reabsorption of gases. 8. Reweighed the sample (and tube).   **Cleaning sample tubes and bungs**   1. **Check the individual experimental risk assessment for risks from samples.** 2. BET is a non-destructive technique so you can keep your sample. Otherwise dispose of powder as discussed in your risk assessment. 3. Rinse the tube with methanol or soapy water if required. 4. Gently use bottle brush to clean. Be VERY careful not to scratch the bottom of tube. 5. Flush again with methanol and sit upside down in tube holder. 6. Wash and wipe bungs with methanol and dustless tissue. 7. Place tubes and bungs in drying oven at 40ºC.   **Starting up BET – minimum 30 mins before use**   1. The BET should be left on during normal use. However, if it off when you arrive switch on all parts of the BET at the plug sockets and turn on the computer. 2. Ensure that both N2 and He cylinders are open to the marks on the gauges. 3. Collect liquid nitrogen and add to Dewar **WEARING APPROPRIATE PPE.**   **Taking a P0**   1. Check that the liquid nitrogen level in the measurement Dewar is no more than 2cm from top. Top up if necessary **WEARING APPROPRIATE PPE.** 2. Remove the right hand empty sample tube and place your blank sample tube in its place. Make sure plastic sleeve and O-ring are present and screw cuff is finger tight. 3. Go to **Unit 1 🡪 Start P0. measurement** 4. After ~20mins the P0 measurement should be complete. 5. Write the value down in the log book. 6. The P0 value can be found in **Unit 1🡪 Unit configuration.**   **Running a sample**   1. Check liquid nitrogen level in the measurement Dewar is no more than 2cm from top. Top up if necessary. 2. Remove previous sample/empty tube from BET. 3. Remove new sample from degasser turning off flow of gas to that line **and REWEIGH**. 4. Place plastic sleeve and O-ring into place around the new sample tube and screw tube into place on BET - finger tight. 5. Go to File 🡪 Open 🡪 Sample Information. 6. Type in new file name and press open. Software will ask if you want to create a new file, answer OK. Save the file in your folder. 7. Click on REPLACE ALL and pick the most appropriate file with the desired parameters. Use the “Standard method file” if appropriate. 8. Edit the parameters as desired ensuring you change the mass of tube and tube with sample. Please check with a technician before changing any of the other parameters. 9. Exit this and go to Unit 1 🡪 Start analysis (the browse to find your file) and click start. 10. Wait to ensure that the vacuum forms. If you haven’t screwed the tube in correctly the vacuum will not form and the sample will not run. 11. After the sample has run (about 45mins) save the report into your folder, 12. Check the Correlation coefficient is four or five nines (e.g. 0.99999 or 0.9999). Three nines would suggest your results are not robust.   **Closing down BET**   1. Fill in the log book for the work you have done. 2. Make sure there are empty sample tubes on the BET. 3. Close gas cylinders. 4. Results are saved at C:\GeminiVII\data\if you want to take them away. 5. Leave the equipment on unless shutting down prior to a holiday in which case shutdown the computer and turn off all pieces of equipment at the plug sockets. | **Hazard symbols:**  Compressed gas |
| **Significant hazards:**   * Gas cylinders * Liquid nitrogen |
| **Hazard phrases (H):**  See individual experiment risk assessments. |
| **Can it be done out of hours?**  **The BET should not be used out of hours** due to requirement for liquid nitrogen and cylinder gases. |
| **This SOP is not relevant in the following circumstances:**   1. SOP does not cover specific experimental risk these must be covered by user’s assessments. 2. Any other situation where the procedure may result in harm to yourself or others. | |