Department of Engineering – Risk Assessment

Ref No. B887

Title of project/experiment/activity **Use of Quantachrome NOVA 2200e Surface and Pore Volume Analyser (BET Instrument)**	
Location of activity Cambridge Graphene Centre, Ground floor: Energy Storage Lab	**Start and end dates** 01/08/2016 - continuous

Brief description (or attach procedure/protocol)

The NOVA 2200e high-speed, automated surface area and pore size analyser, is a fully automated vacuum volumetric, gas sorption system. This system is capable of degassing and measuring two samples. and must be used in accordance with the manufacturers instructions.

This BET instrument is a self-contained instrument with a powerful built-in microprocessor with automatic coolant level control that ensure constant, small void volume for accurate data throughout the measurement.

The instrument is capable of operating a variety of gases (e.g. nitrogen, helium, argon, carbon dioxide) and coolants (e.g. liquid nitrogen, liquid argon, ice/water) for a variety of applications. The default system is set up with N_2 as the sorption gas and liquid N_2 as the coolant. Helium (He) is also connected to the instrument. Measuring the void volume of the sample cell could be done immediately prior to sorption measurements - in the presence of sample - using He. This risk assessment is only for N_2, He, and Ar. An additional risk assessment is required for any other sorption gas/coolant combination with considerations for chemical reactivity, toxicity, and control measures at room and elevated temperatures.

Various samples can be measured e.g. graphene, 2d materials, metal oxides, etc. (user should refer to personal risk assessments for sample handling).

Due to the variety of materials measured in the BET instrument, separate risk assessments must be carried out for every material measured in the instrument with consideration of sample form and size, aerosol/particle handling, material handling, toxicity, sensitivity, carcinogenicity, temperature, gaseous and non-gaseous products, etc. This list is only an indicator and not an exhaustive list of factors that need to be assessed.

Hazard	Effect	Control measures	Residual risk
General hazards in lab	Inhalation of solvents	Wash bottles containing volatile and combustible solvents, such as acetone, ethanol, and isopropanol, are present and used in the lab to clean items or equipment. (Likelihood: 1, Severity: 1) There is appropriate ventilation in place. The solvent will be contained in a wash bottle. Eye protection and lab coat will be worn while in the laboratory. The Energy Storage Lab rules will be followed.	Low risk
Electrical hazard	Electric shock	High voltages are present inside this instrument. Death can result from normal voltage of 240 V causing currents of greater than 30 mA to flow through the body for more than 40 ms. Minor shocks may also cause injury following involuntary muscle contraction. (Likelihood: 1, Severity: 3)	Low risk

		As a result, the instrument will not be opened by the user. Only a qualified technician or expert is allowed to open the instrument enclosure. The instrument must be disconnected from the mains before doing any maintenance or repair work that requires opening the cabinet covers.	
Chemical hazard	Fire; Inhalation of potentially toxic gases;	The sample atmosphere is controlled during degassing and sorption experiments by connecting N_2 to the system. High purity N_2 (chemical purity grade) is connected for degassing and sorption. No other gas beside N_2, He, and Ar must be used without a separate risk assessment. (Likelihood: 1, Severity: 1) Under no circumstances will hydrogen or any other explosive gas be used in the instrument. Corrosive gases shall not be used in this instrument. O_2 and CO_2 may be used as a purge gas, but a separate risk assessment must be filed. The instrument is not intended for materials that undergo chemical reactions at either room or elevated temperature of the degassing. As such, the degassing temperature selected must be such that the no chemical reactions or vaporization takes place. In addition, a separate risk assessment must be made for every material measured in the instrument with its thermochemical stability assessed and appropriate degassing temperature selected. A separate risk assessment must be done for every material measured in the instrument. If measuring samples that vaporizes, undergo decomposition, or may emit harmful gases, an exhaust must be connected to the vacuum pump.	Low risk
Compressed gases	Explosion; Asphyxiation;	If the regulator fails, the gas is released suddenly with possible fatal consequences, but fortunately failures of regulators that have been purchased to the correct standard, and selected correctly for the service are rare. If a compressed gas cylinder tips over, causing the valve block to be sheared off, the rapid release of high pressure gas may cause the cylinder to be violently accelerated, potentially causing property damage, injury, or death. (Likelihood: 1, Severity: 3) Size V chemical purity grade N_2, He, and Ar bottles are installed in the lab for used with the instrument and chained to a rack to prevent falling and breaking.	Low risk

		Compressed lab gas supplies are equipped with suitable regulators. Installing and replacing gas cylinders will be done by trained personnel. Compressed gas poses asphyxiation hazard – a condition of severely deficient supply of oxygen to the body that arises from abnormal breathing – such as choking. This could be fatal. (Likelihood: 1, Severity: 3) Proper ventilation is maintained to prevent injury or death due to asphyxiation in case of leaks. Also, an oxygen monitor is located next to the BET instrument and will alarm if oxygen levels fall below 20%. The maximum pressure supply to the instrument is 20 psi for the experiments. The pressure is set to 10 psi by default and it is normally retained.	
Thermal hazard	Skin burn; Fire	The outer surfaces of the heating mantle and the sample holder/sample may become hot during use, up to 350 °C. If a sample is removed whilst the sample tube is still hot, there is a risk of burns to the user. (Likelihood: 2, Severity: 2). The heating mantles shall not be held without wearing thermally insulating gloves until it is at a safe handling temperature, <60 °C. Fingers shall never be inserted inside the pocket of the mantle to determine if the mantle is heating up. The temperature will still be displayed when the switch is OFF, so that the temperature of the mantle will always be monitored, even when cooling down. The heating mantles are designed not to operate if the thermocouple is not plugged into its jack. Wash bottles containing volatile and combustible solvents, such as acetone, ethanol, and isopropanol, are present and used in the lab. (Likelihood: 1, Severity: 2). The sample stage is enclosed to prevent accidentally touch by user or contact with solvents. The door must be closed at all times when the sample is not being loaded or removed.	Low risk

		Liquids shall not be allowed to come into contact with the heating mantle and heating mantles shall not be handled with wet hands. Heating mantle shall not be disconnected and removed from the BET instrument under to prevent possibility of combustible compounds coming into contact.	
Liquid Nitrogen	Asphyxiation; Frostbite	Liquid nitrogen poses asphyxiation hazard by displacing oxygen in air. It can cause rapid suffocation without warning. This could be fatal. (Likelihood: 1, Severity: 3) Proper ventilation is maintained to prevent injury or death due to asphyxiation in case of leaks. When decanting LN2 to fill the instrument's dewar in the lab, the lab entrance door will be opened to enhance ventilation. An oxygen monitor is located next to the BET instrument and will alarm if oxygen levels fall below 20%. LN2 could cause serious frostbite when in contact with skin and it could cause serious damage to eyes. (Likelihood: 3, Severity: 2) Continued exposure of unprotected flesh to cold atmospheres can result in frostbite. Though there is usually sufficient warning by local pain whilst the freezing action is taking place, care must be taken not to pour LN2 on the skin. In addition to wearing rubber gloves, lab coat and glasses, a pair of thermally insulating gloves and face shield will be worn whilst filling the LN2. The instrument's dewar flask is not designed for transporting LN2. LN2 must be transported in a safe, designated dewar with a lid and must be decanted into the instrument's dewar flask for use. Only the required amount of LN2 must be transported into the lab for instant use. There shall be no LN2 storage in the lab for future use. The dewar flask will be checked to ensure that it is clean and dry.	~~Low~~ risk MODERATE

Personal Protective Equipment required [*eye/face protection, respiratory protection, gloves, lab coat etc*]

Department of Engineering – Risk Assessment Ref No.

Lab coat and eye protection (safety specs) required in the lab at all times.
Emergency Instructions & First Aid General advice: Consult a physician. Show this risk assessment to the doctor in attendance.
Any special monitoring required [*e.g. hearing test, vibration monitoring, health surveillance*]
Further control measures required? If yes, list with actions. In the case of equipment malfunction/failure, you need to cut off the power supply. Merely closing the control software will not terminate the measurements. To cut off power from the instrument, the power switch is located at the rear of the instrument.
Biological/Laser/Radiation Approval [*requires relevant Specialist Safety Officer signature and date*] N/A
Out of hours/Lone working Measurements may be run overnight. Once the measurement has been started, the instrument completes the measurements based on the preset procedure. An unattended experiment form must be filed by the user and signed by a designated super user of the BET instrument, the person in charge of the BET instrument, or the lab leader for such an experiment to be run. Also requires permission from Head of Division.

Signature to confirm that this is a suitable and sufficient assessment of risk and that stated control measures are in place. This risk assessment should be reviewed if additional risks not covered in this assessment are identified or if there is any reason to indicate that the control measures are insufficient.

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Name of Supervisor Prof. Andrea Ferrari Email: acf26@cam.ac.uk	**Signature**	**Date** 2/9/16

Local Safety Coordinator	**Signature** D Hanks	**Date** 27/10/16
Departmental Safety Office IAN SLACK	**Signature**	**Date** 9 NOV 2016