

BET_ANAMET

- GENERALLY:** Use stations S1 and S2 for measurement with nitrogen gas and liquid nitrogen. Check the amount of liquid nitrogen before the measurement. Use station S3 for measurement with argon (gas). Two days before measurement, the liquid argon must be arranged with Marek Eliáš (Marek.Elias@ceitec.vutbr.cz), and BET Dewar labeled 'Ar' must be used. **If the BET machine is not used** for a few days, it is necessary to fill it with proper gas (measurement with N₂- use nitrogen, measurement with Ar – use argon) during sample degassing. If the instrument is not used for longer than a week, it is better to gasify the device overnight.
- Switch on nitrogen OR argon** (move the lever to the ON position (just a little bit - you don't have to open it entirely). The main switch is labeled "Nitrogen" (Fig. 1B) /"Argon" (Fig. 2). The pressure of N₂ /Ar should be around 0.07 MPa, max. 0.1 MPa, the reduction value is permanently preset to the required value. Do not touch the reduction valve on the nitrogen/argon line. The description of a gas cylinder is in Fig. 3.
- ALWAYS Switch on Helium** (Fig. 1A) – open the main valve on the Helium cylinder - use excessive force to open it. It is better to fully open the valve and then turn it back a little. This will prevent the jamming of the thread. The pressure in the He line is preset at 0.5-1 bar. If the pressure is lower, contact the instrument guarantor.

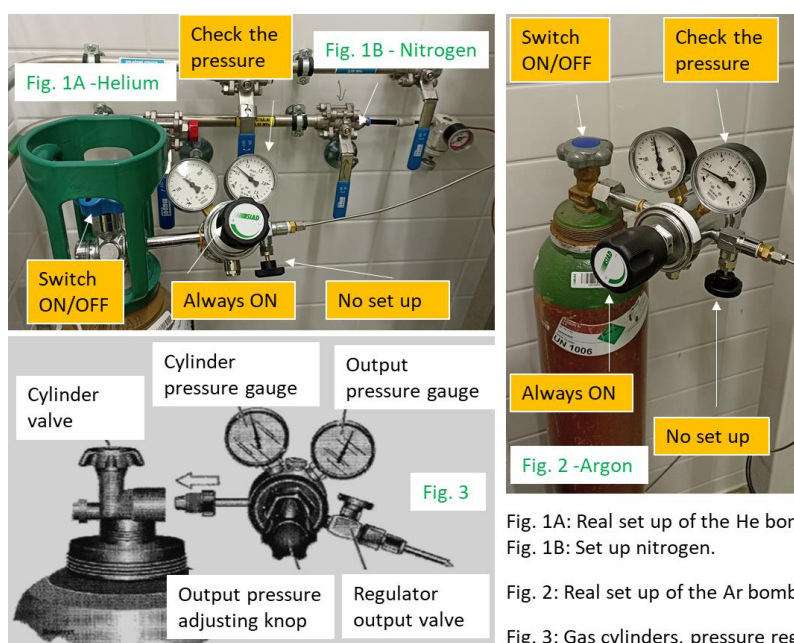



Fig. 1A: Real set up of the He bomb.
Fig. 1B: Set up nitrogen.

Fig. 2: Real set up of the Ar bomb.

Fig. 3: Gas cylinders, pressure regulators.

- Switch on the electrical plug** labeled 'BET' (behind the screen). It is a plug for the instrument and vacuum pump.
- Switch on the BET instrument** (Fig. 3A) and PC - **program PAS.exe** (Fig. 3B).
- Set up the program** by the icon . You can find more details about the setup of the instrument in the manual (Manual 3P micro, vapor series_SW_10.06, Appendix 2, page 28).
- The sample cell installation:**
 - Check the pressure** in the station (it must be done individually for stations S1, S2, and S3). The pressure level should be around zero – NO vacuum, and TURBO must be OFF. (**Vac.1-3**) must be switched off (no lights) (Fig. 3C). If a vacuum is present in the station, it is necessary to fill stations S1 and S2 with a nitrogen – button in the software 'Dose(N2)'. **Station S3 is permanently connected to Ar, dose Ar (in software labeled 'Dose(N2)'). Check that the station is set up for measurement with Ar(87K) (Software: Set up – Parameters- tick Ar).**
 - If the **BET Dewars are placed** on the BET Dewar holder, **take it away** (there is a risk of damaging it during the sample cell installation).
 - Prepare the sample cell according to Fig. 3D. **Be careful** when placing a filler rod in the sample cell. Follow pictures 6-4 in the Manual (Manual 3P micro, vapor series_SW_10.06, Appendix 2, page 57). If you used a fritta for degassing, use it for BET measurement as well.
 - Unscrew the blind steel pin from the station and place the sample cell with all components (Fig. 3D). Carefully tighten it with your hand.

8. Fill the BET Dewar with liquid nitrogen/argon and place it on the holder. Push the P0 tube close to the sample cell Fig 3E. Try the correct position of the BET Dewar by command “Dewar Up/down” - An unrestricted stroke for the BET Dewar must be provided. (see Fig. 4 - 2 - ‘Dewar Up/Down’).
9. **Push ‘EVACUATED’** and wait till the evacuation of the station is finished.
10. **Import prepared methods** (Pore Analysis System – folder: 1METHODS) if it is applicable.
11. **‘START ANALYSIS’** We recommend waiting until the “Leak test” is successfully performed. You can track the program’s progress by clicking on the **note icon Fig. 4-4**. In case of a failed Leak test, wait until the program ends. Evacuate the sample again. Leave the sample in a high vacuum for a few minutes and restart the program with the command – **“Start Analysis” Fig.4-3.**
12. **After finishing the measurement**, check the pressure in the station. Switch off the turbo, put away BET Dewar, dose N₂/Ar, unscrew the vial, and place the steel pin back. Fill the line with N₂/Ar (in the main menu in the software, see Fig.4.).
13. Pour out the liquid nitrogen from BET Dewar back into stored Dewar. Pour Ar out outside on the grass.
14. **Close the PAS software and switch off the BET instrument (main switch). Switch off the electrical plug with the BET pump.**
15. **Close N₂/Ar with the lever to the OFF position and the He valve on the gas cylinder.**

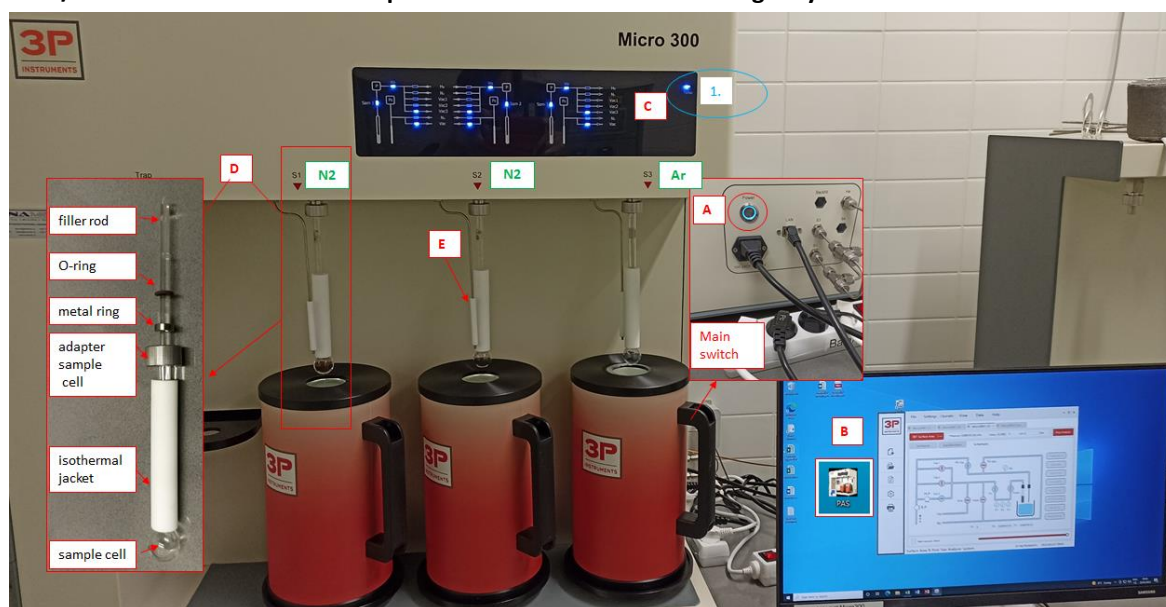


Fig. 3: 3A - Main switch of BET instrument, 3B - BET software, PAS.exe, 3C - The control of switching on/off TURBO, 3D - The installation of the sample cell. 3E – P0 tube.

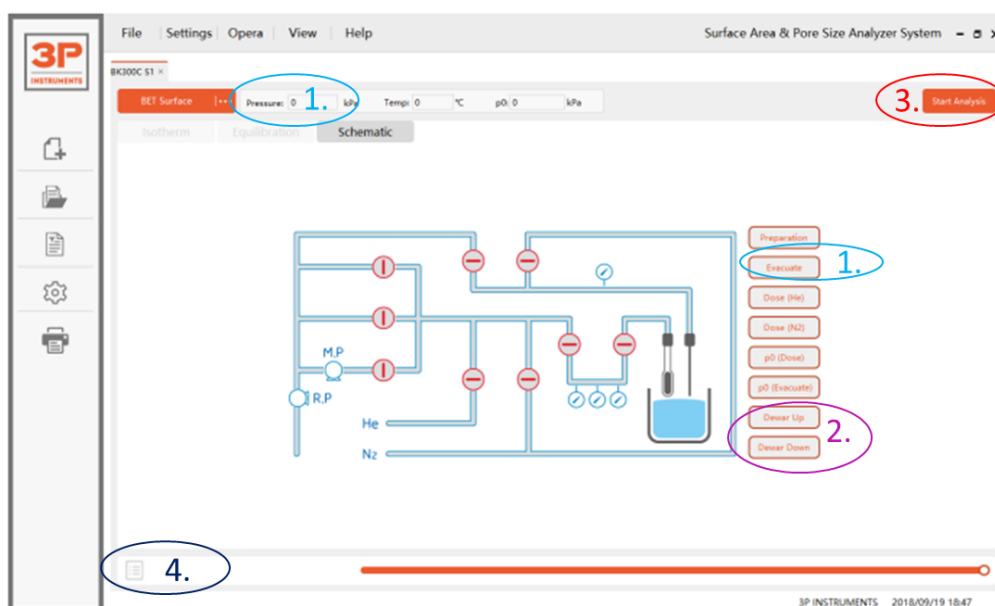


Fig. 4: Schematic picture of toolbars in the software of the instrument.