DEGASSER_ANAMET

1. **The sample weighting** (Manual 3P micro,vapor series_SW_10.06, Appendix 2, page 69):
   a) **Weight the vial** with the orange cap, and write down the value. Use the clear vial with the orange cap from the box in the cabinet labeled "BET". For adding a powder using the glass cylinder (sample weight according to table 6-1 in BET manual), usually 0.1 – 0.5 g. It is recommended not to have powder on the wall of the vial stalk. If it happens, it is possible to put away the powder with a wooden stick with cotton (possibility of contamination, reweight it). If you work with a '*flying*' sample, use a sintered glass filter without a ball and weigh the sample with the sintered glass filter instead of the orange cap.
   b) **Weight the vial** with the orange cap and a sample. Use the Antistatic kit (Fig. 1) for discharging by passing through the ionizer for an accurate value (switch on the device, throw the ´bridge´ with the vial full of powder). **Weight**. Write down the stable value (vial with a cap and a sample).

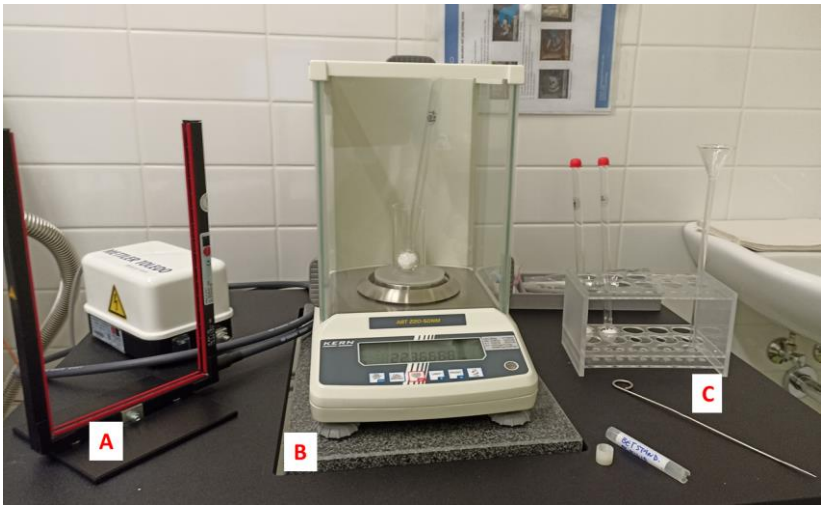


Fig. 1: Antistatic kit (A), Microbalance (B), Holder with vials, and cylinder (C).

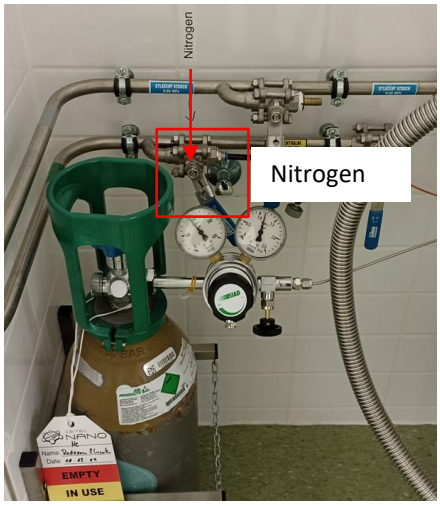


Fig. 2: Main seal for nitrogen.

2. **Degassing (Manual 3P micro,vapor series_SW_10.06, page 75):**
   a) **Open the nitrogen line** - the main switch (Fig. 2). The maximum pressure should be around 0.07- 0.1 MPa. The reduction valve is set, do not touch the valve.
   b) **Switch on the electrical plug labeled ´Degasser´** (behind the PC monitor): Switch on the degasser (Main switch on the right side of the instrument, Fig. 3).
   c) **Switch ON the turbocharge**: Pusch the main button (Fig. 4A) and the on/off button on display (Fig. 4B).
   d) **If the vacuum is ON**, Switch it off ´VACCUM,´ push ´GAS IN,´ and wait until the atmosphere pressure (around 0 kPa, green) is reached (Fig. 3). Switch off ´GAS IN´ (no nitrogen goes to the room atmosphere). Only then the measuring cells could be removed. Never change the heating mantle from one port to another. The heating mantels are labeled.
   e) **Screwing vials:** Take away the stainless steel pins from the stations you plan to use (it is impossible to do so during degassing). Add all parts on the stalk (Fig. 5B-C), insert the sample cell into the instrument's corresponding port, tighten it carefully, and place the magnetic holder and heating mantle (Fig. 5A). Be careful – you can break the stalk of a glass vial easily. NEVER move the glass vial after the installation. Move only with the magnetic holder.
   f) **Switch on the VACUUM** - Wait a few minutes to reach the vacuum (-98.5 KPa - red number). If the vacuum is not reached, check the vial connections.
   g) **Switch ON** the exact degassing station (the upper button in the degasser display, Fig. 3). press RUN (▽) to start the programmed degassing process. An example of a program is in Fig. 6.

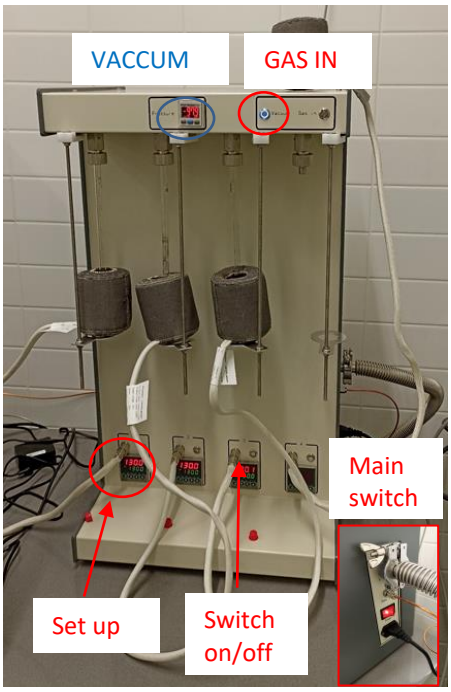


Fig. 3: Degasser.The explanation of primary functions.

Fig. 4: The turbocharge: Main switch (A). The switch button on display (B). Open/close of vacuum (C).

h) After the degassing process, switch off ´VACCUM,´ push ´GAS IN,´ and wait to reach the green level. Switch off ´GAS IN.´

i) Unscrew the vial. Cover the vials with an orange cap. Go throgh the antistatic kit. Weight the vial. Write a mass of degass sample.

j) Add all parts to the pin. Screw it to the degasser, and push ´VACCUM´. Wait till the vacuum is reached. Switch off the instrument. **Switch off the turbocharge, wait several minutes to slow the rotation speed, then turn the screw to the left to vent the pump (Fig. 4C) and turn it back.**

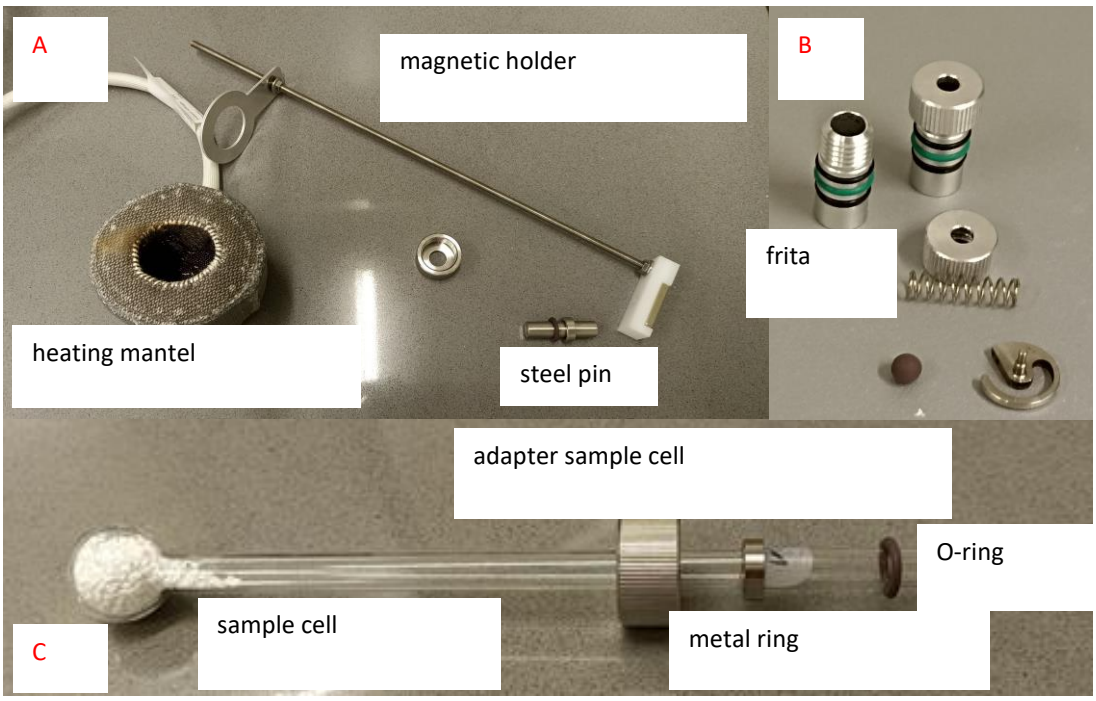


Fig. 5: Degasser parts (A). Completed and distributed frita (B). Preparation of the sample cell for installation in the degasser (C).

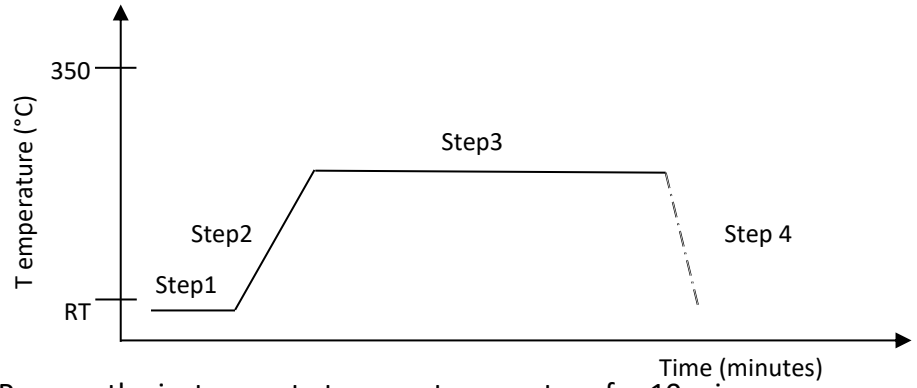
Set up of temperature program:

1. Ensure the station is OFF ("STOP" flashes on display). If not, press STOP (△). Then briefly press ◁; this will allow you to program the set point temperature (SP) in °C and the time (t) in minutes for each step. The device will enable you to program up to 30 individual steps in one degassing process. If you want to return to the segmented menu, wait a couple of seconds without pressing any buttons.
2. First, program the starting point **SP1** and time ***t1***. The ◁ button changes the decimal place ▽ and △ will change the set values. Then press ↻ to save the current step. You can automatically change other steps (***SP2***, ***t2***, ***SP3***, ***t3***...). To end the program, it is necessary to give high negative values at the last step (e.g., ***SPX*** = -100, ***tX*** = -100).
3. Before starting the programmed degassing process, it is recommended to check the entire sequence once more. Press briefly ◁ and then press several times ↻ until the end of the sequence. To activate the programmed sequence, press RUN (▽) for a few seconds to start heating. To terminate the program prematurely and to stop degassing process, press STOP (△)

Notice

- In the beginning, please ensure that the controller is not a RUN/HOLD state but a STOP state. If not, press STOP.
- It is good practice to start the first step for a few minutes at room temperature (e.g., ***SP1***=25, ***t1***=5).
- The individual steps are time-controlled, not temperature-controlled. If you want to choose a rapid increase/decrease in temperature, give the program enough time to reach the desired temperature. Otherwise, after the set time has elapsed, the program will automatically go to the following sequence, which may have a negative effect on the shape of the temperature ramp.
- If a longer time than 16.5 h (999 min) of heating is needed, set another set point (***SPy***, ***ty***) to the appropriate temperature and time (e.g., SP3=350, t3=600, SP4=350, t4=600 means to hold the temperature at 350 °C for 12h)
- Be careful where the dot is placed in the number (decimal point).
- Never use small negative values to terminate the process. Values from -1 to -30 do not end the program but are a command to run individual steps in the sequence (e.g., SP= -4 automatically starts the fourth step in the sequence). To terminate the program, use high negative values, e.g., SP= -100 or negative values with a decimal place, e.g., SP=-4.3)

More details are available in the manual (Manual 3P micro, vapor series_SW_10.06, page 7).



Step1: Prepare the instrument at room temperature for 10 min.
SP1 = 25, *t1* = 10
Step2: Heating from RT to 220 °C within 30 min
SP2 = 220, *t2* = 30
Step3: Hold the temperature at 220 °C for 90 min
*SP*3 = 220, *t*3 = 90
Step4: Cooling proccess back to RT
*SP*4 = -100, *t*4 = -100

Fig. 6: Example of Degass sequence