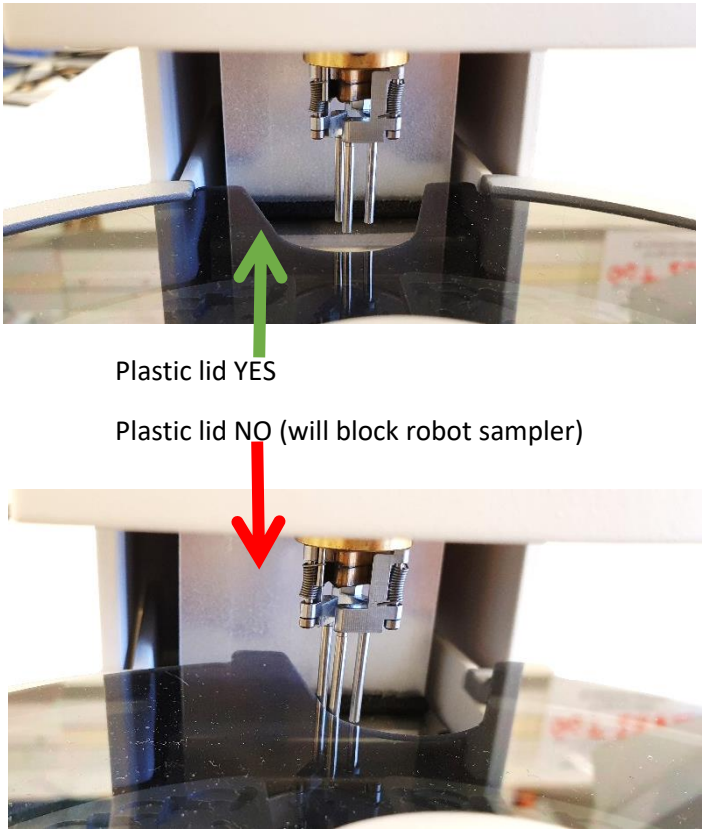
Mettler DSC2 short checklist

Note: All experiments must be finished before 16.00 on Fridays, as the cooler is switched off over the weekend. If you are the first user of the week, you may need to switch on the cooler (use touchscreen).

The DSC temperature range is -100°C to 500°C. DSC experiments at temperatures exceeding 500°C can be carried out in the combined TGA/DSC 3+ instrument – contact the instrument responsible.

1. During sample preparation: always wear gloves, make a note of the sample weight (typically 1-5 mg), and make sure that the pan is clean and not deformed as this may cause problems with the robot sampler.
2. Once you have a suitable method, open the Experiment window.
3. Select a physical sample position and check that it is not the same as the running sample. After placing your sample, make sure that the plastic lid is replaced in its correct position.
4. Add 100 to the sample position, i.e. physical position 23 = 123 in the Experiment window. Send the experiment.
5. Register your experiment/s in the DSC logbook. You can check the experiment time in the DSC module window.
6. In case of questions, do not hesitate to contact the instrument responsible.

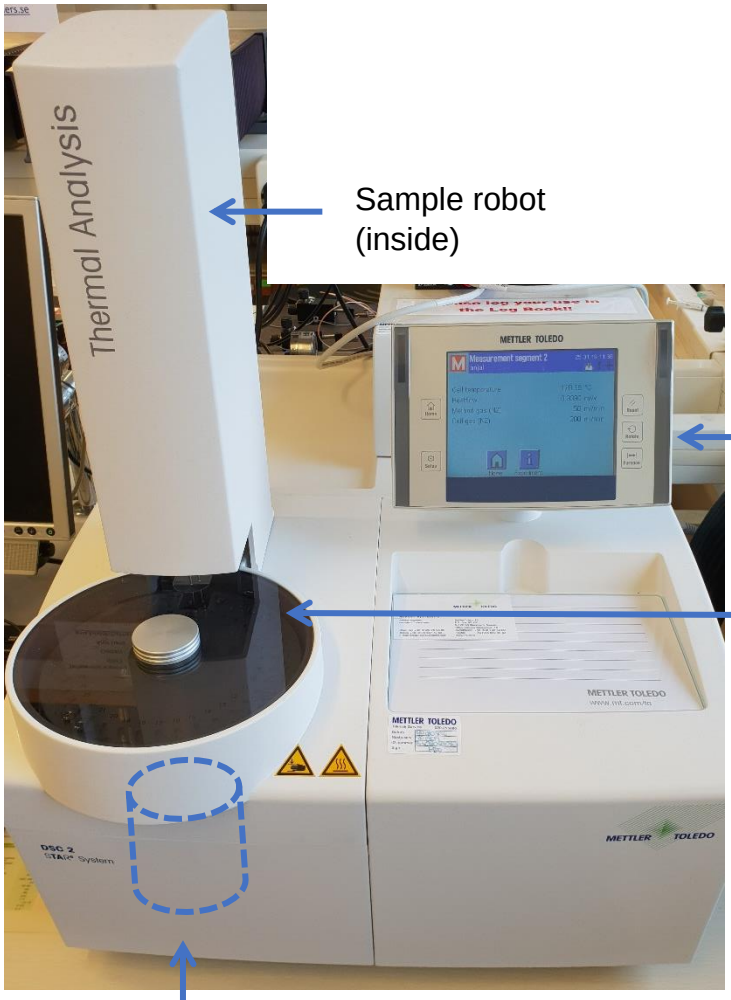


Mettler DSC2 User instructions

Differential scanning calorimetry (DSC) principles

In DSC, the amount of heat required to increase (or maintain) the temperature of a sample is measured as a function of temperature or, in the case of isothermal experiments, as a function of time.

The instrument

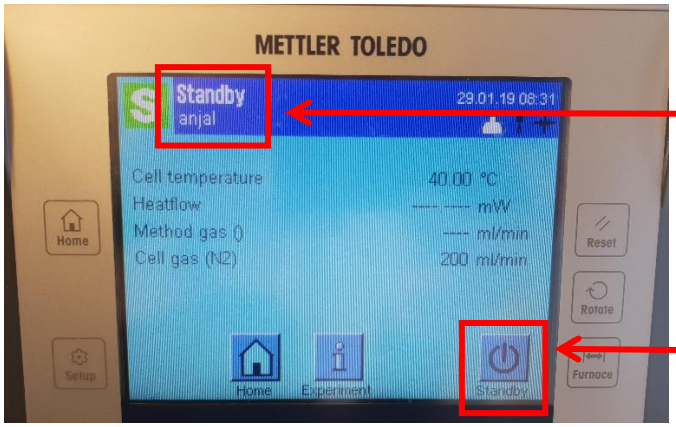


Measuring cell: furnace with sensors for sample (left) and reference pan (right)

Basic operation

Note: the sample crucible is placed directly on the sensor. Therefore corrosive materials are not allowed in this instrument. The DSC is used to monitor transitions, not reactions.
Sample decomposition in the DSC must also be avoided – run a TGA experiment first if you do not know the decomposition temperature of your material.

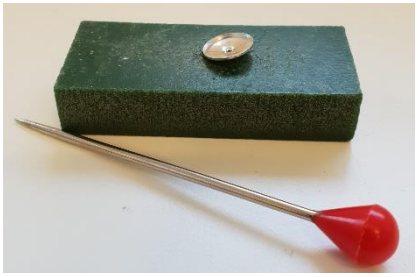
Make sure the cooler is running before you start your experiment. If not, make sure that the instrument is in standby (see below), then switch the cooler on and allow an hour for start-up.

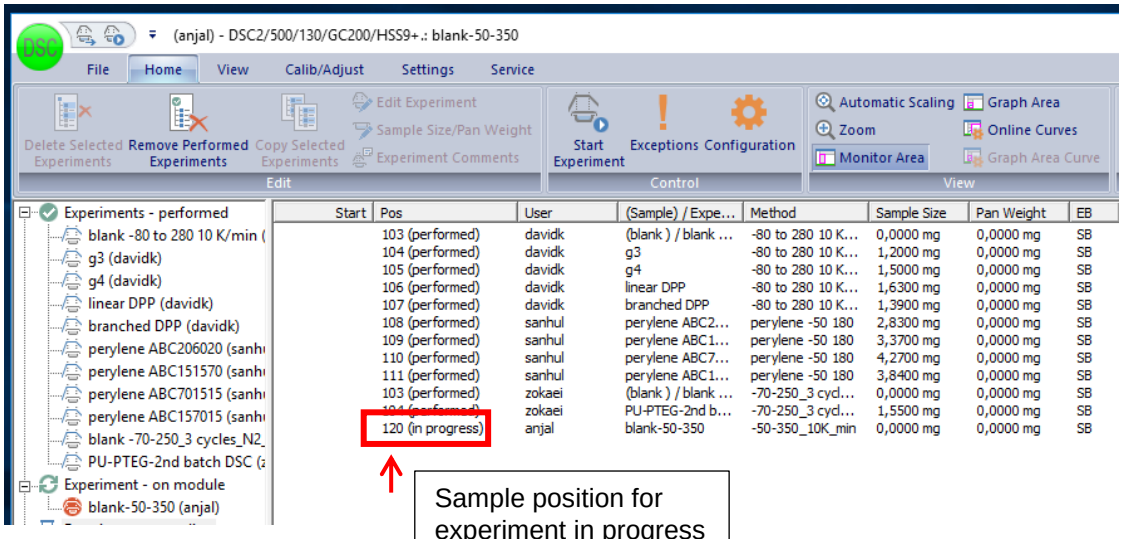


DSC is in Standby mode

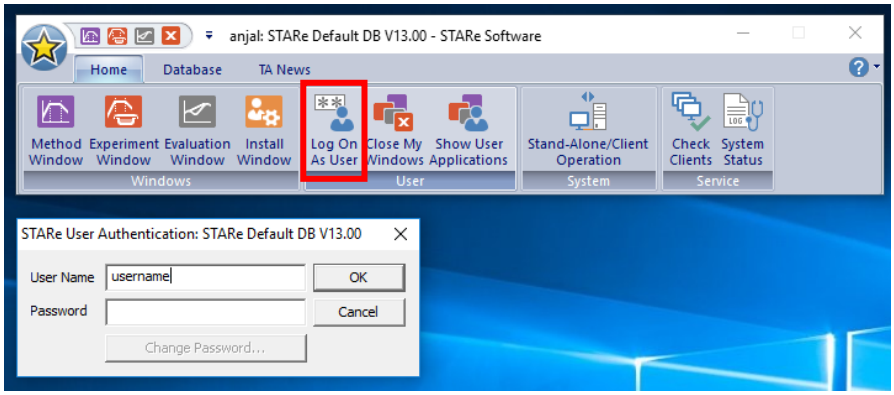
If the instrument is in a different mode, select standby here

1. Prepare your sample:
    a. Wear gloves!
    b. Measure 1-5 mg of your material into a crucible. Make a note of the weight.
    c. Pierce the crucible lid, from the inside out. Seal the crucible.
    d. Make sure there is no sample residue or other dirt on the outside of the crucible, then place it on the sample tray.
    e. Note: make sure not to select the same position as for the experiment in progress, by checking the module window.

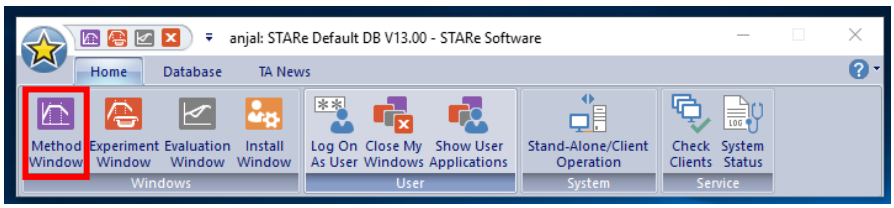




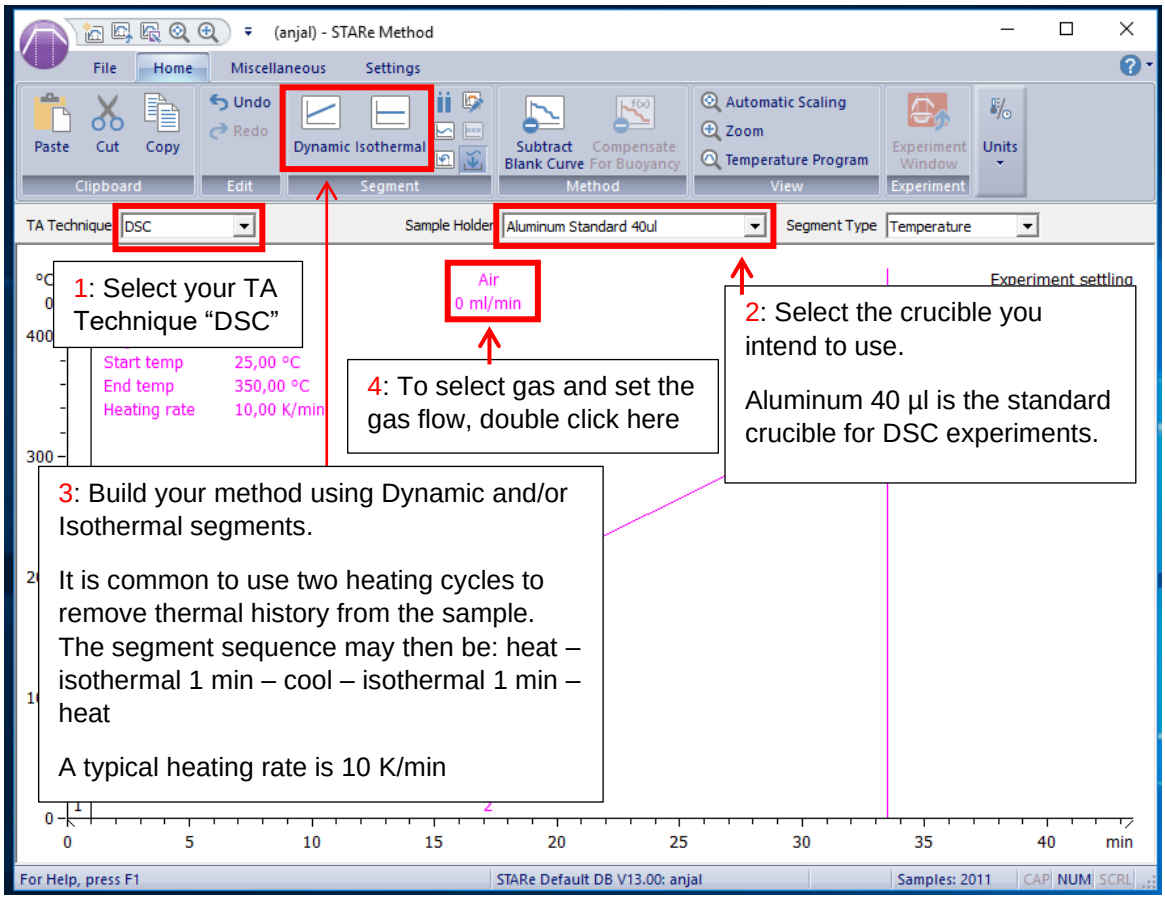
2. Log on to the STARe software



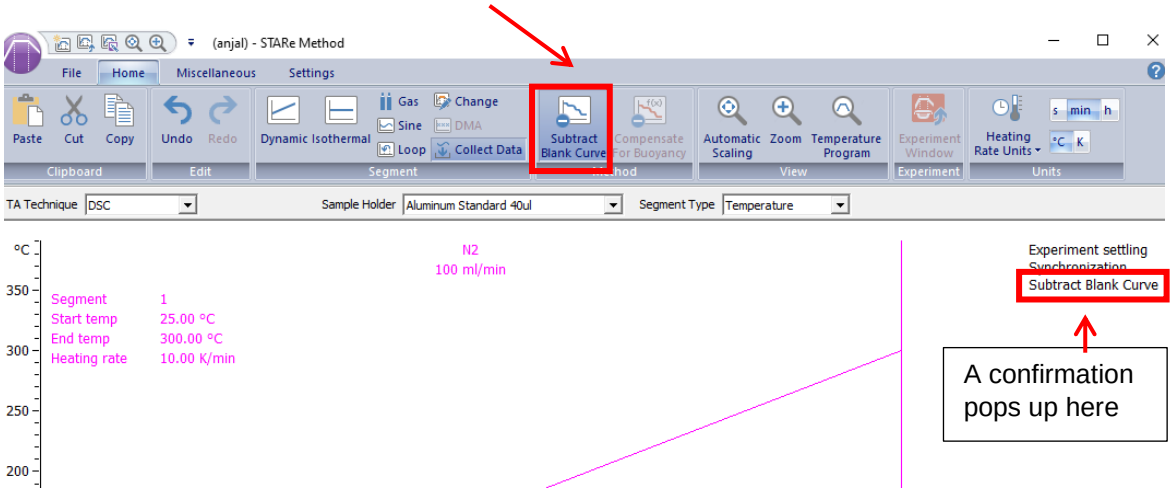
3. Prepare your method using the software. If you already have a suitable method, skip this and go to step 6.
    a. Open the method window



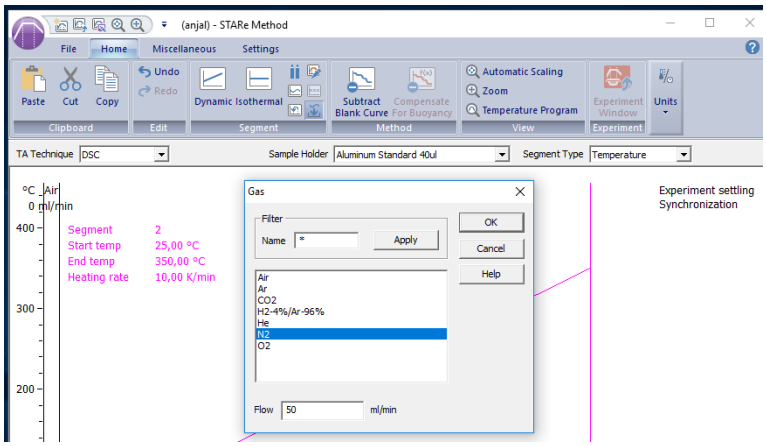
    b. Prepare your method



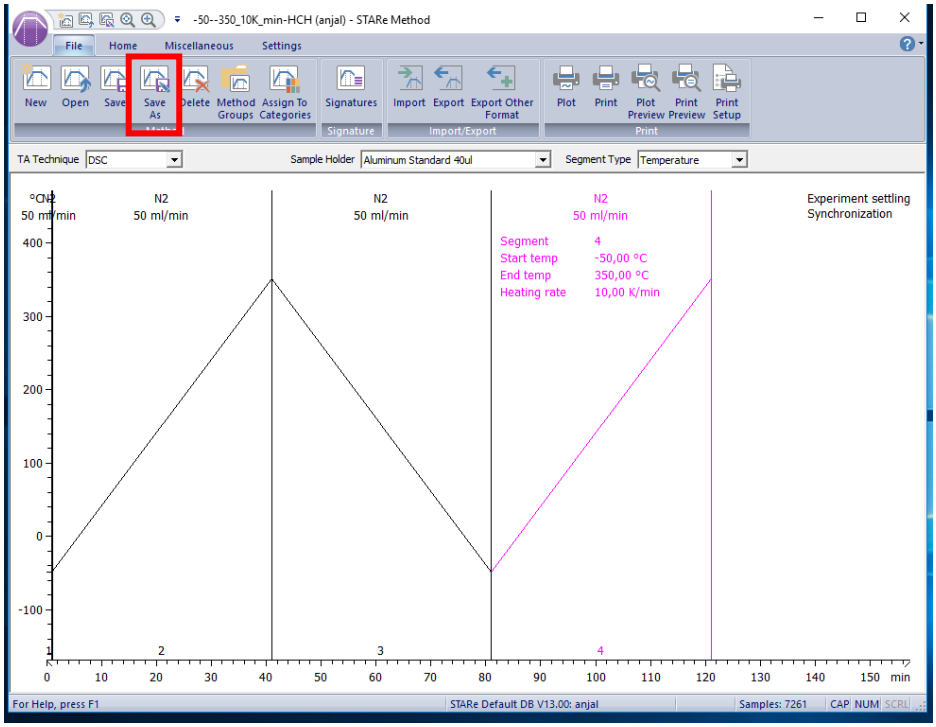
c. IF you want to run a blank first (highly recommended for c_p measurements and for samples with weak transitions), add that to the method by clicking "Subtract Blank Curve".



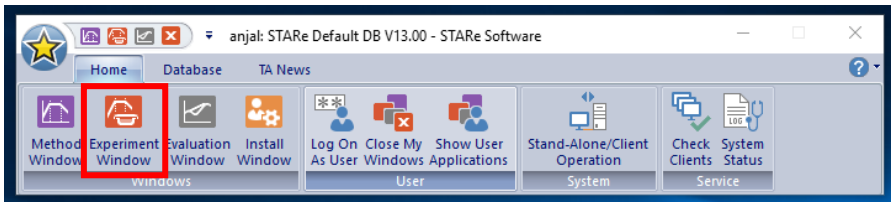
d. Always use N_2 at 100 ml/min to purge the cell.



e. Save your method using a descriptive name

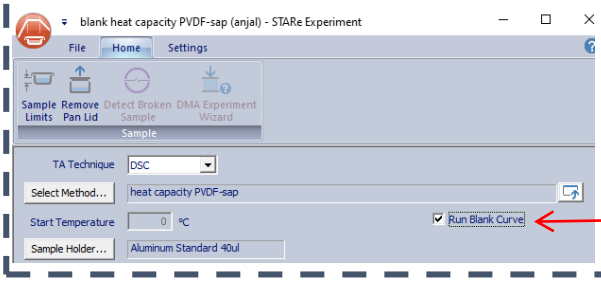


4. Program your measurement
    a. Open the experiment window

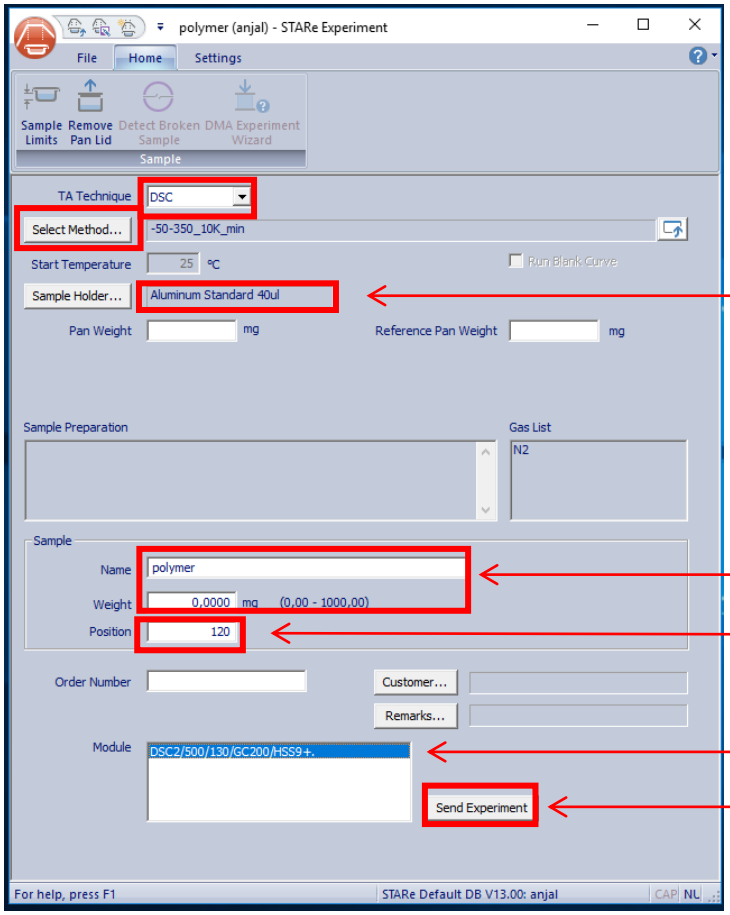


    b. Select your technique and method.

    i. IF you included "Subtract Blank Curve" in your method, you should run a blank (an empty crucible) first. Tick the "Run Blank Curve" box, prepare an empty crucible, and program the run as described further below. For all following measurements with the same method, this blank curve will be subtracted from the result.



For blank curve measurement



1: Double check that you are using the correct crucible

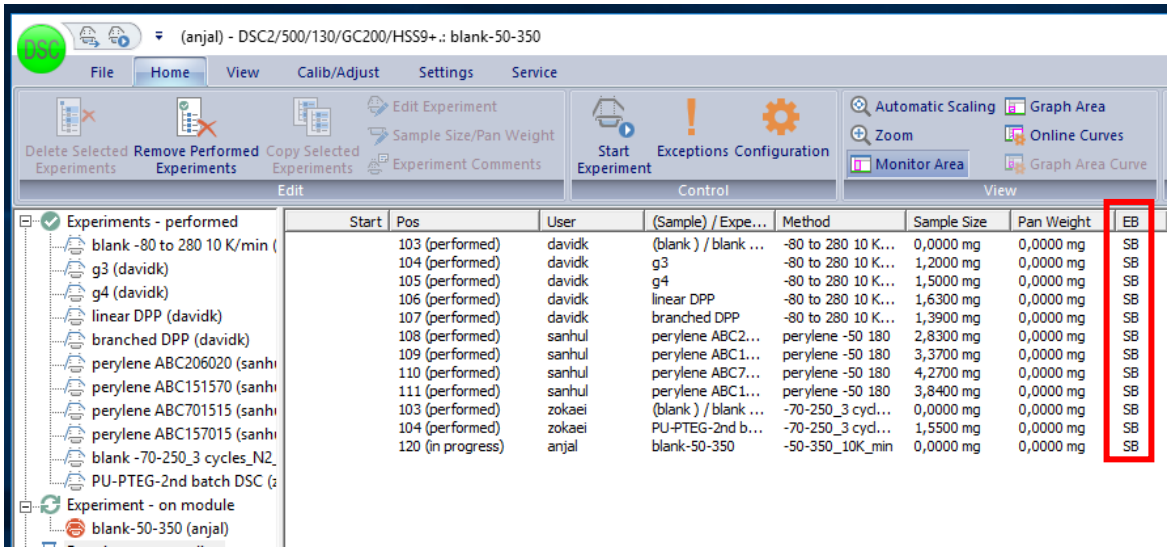
2: Fill in sample name and weight

3: Give the sample position + 100

4: Select module DSC2

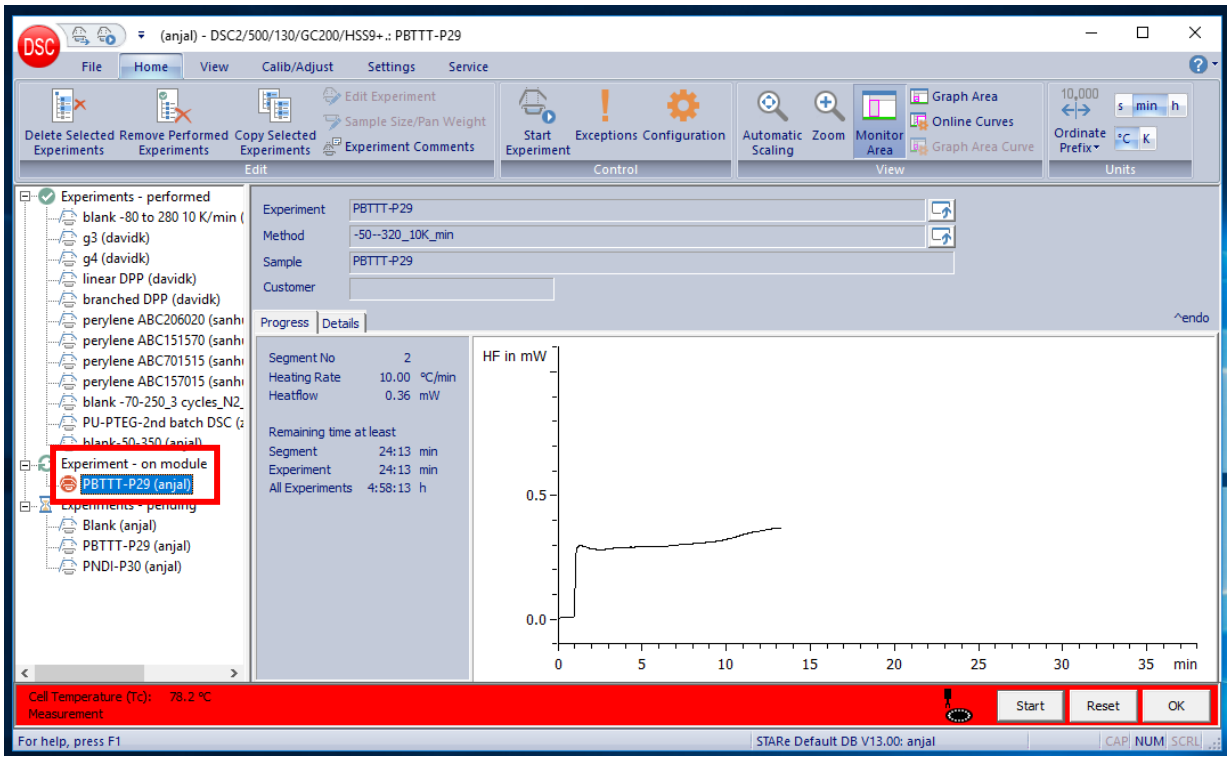
5: Send the experiment

5. Check in the module window that your EB (end behaviour) is SB (standby). If it is not, contact the instrument responsible for help to change it.

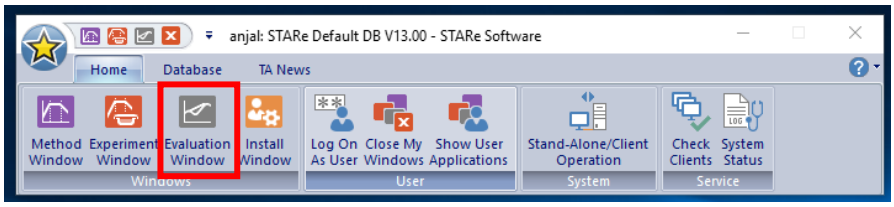


6. If you want to do several experiments, add them according to points 3 and 4.
7. Fill in the logbook

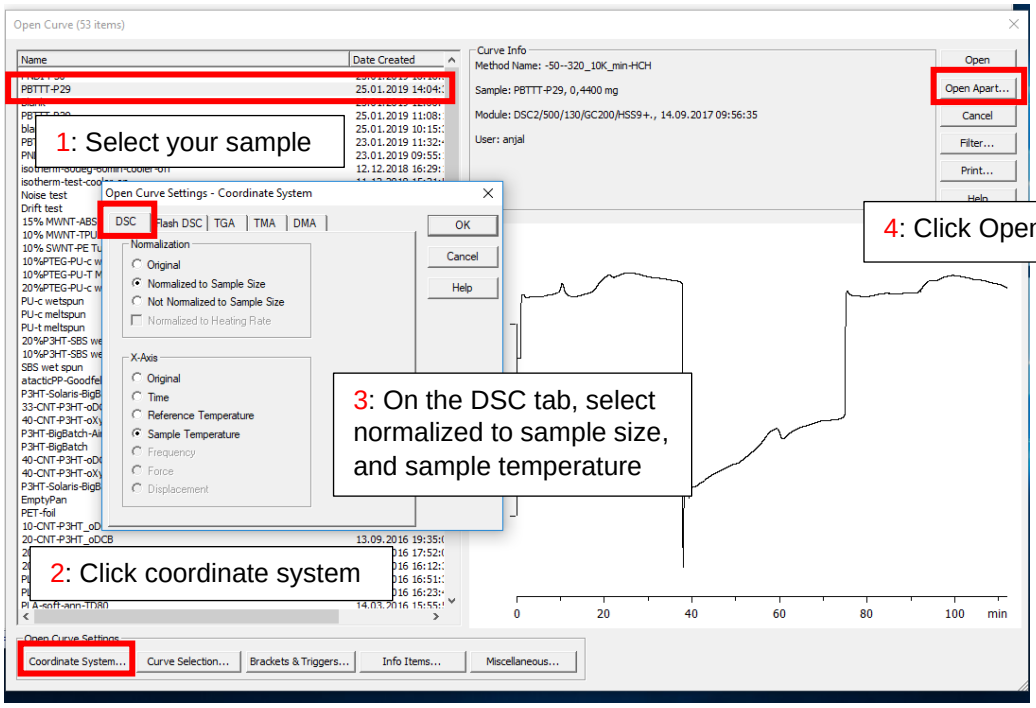
Tip: to check how much time your, and others, experiments will take – select the running experiment in the module window.



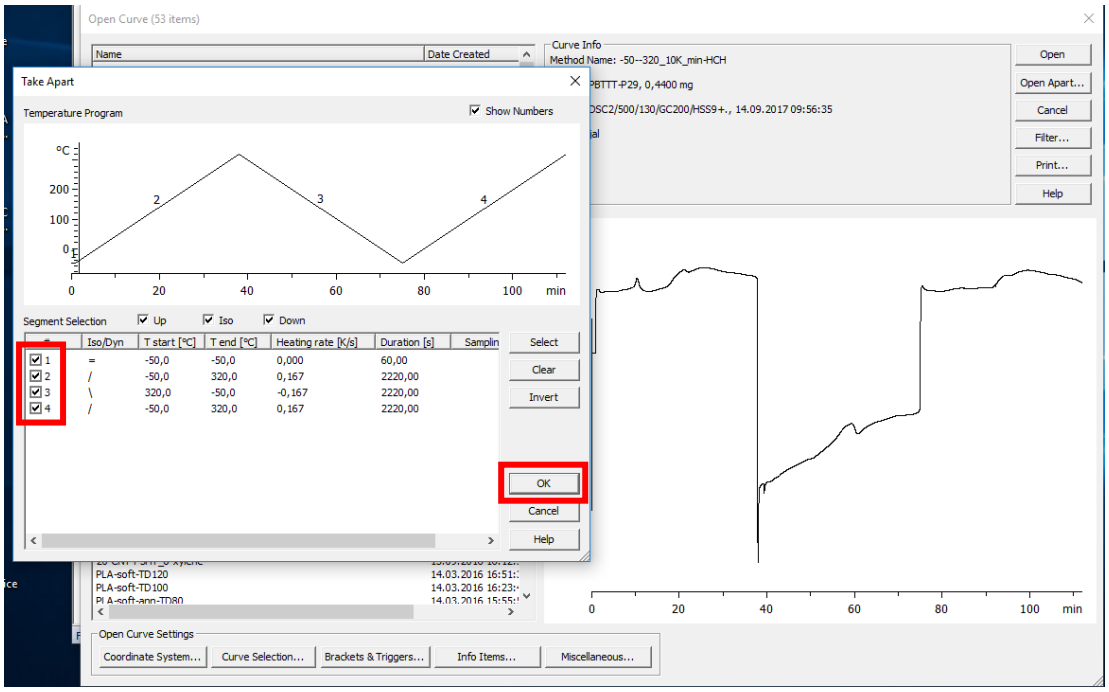
8. After the run - Export your data
    a. Open the Evaluation Window



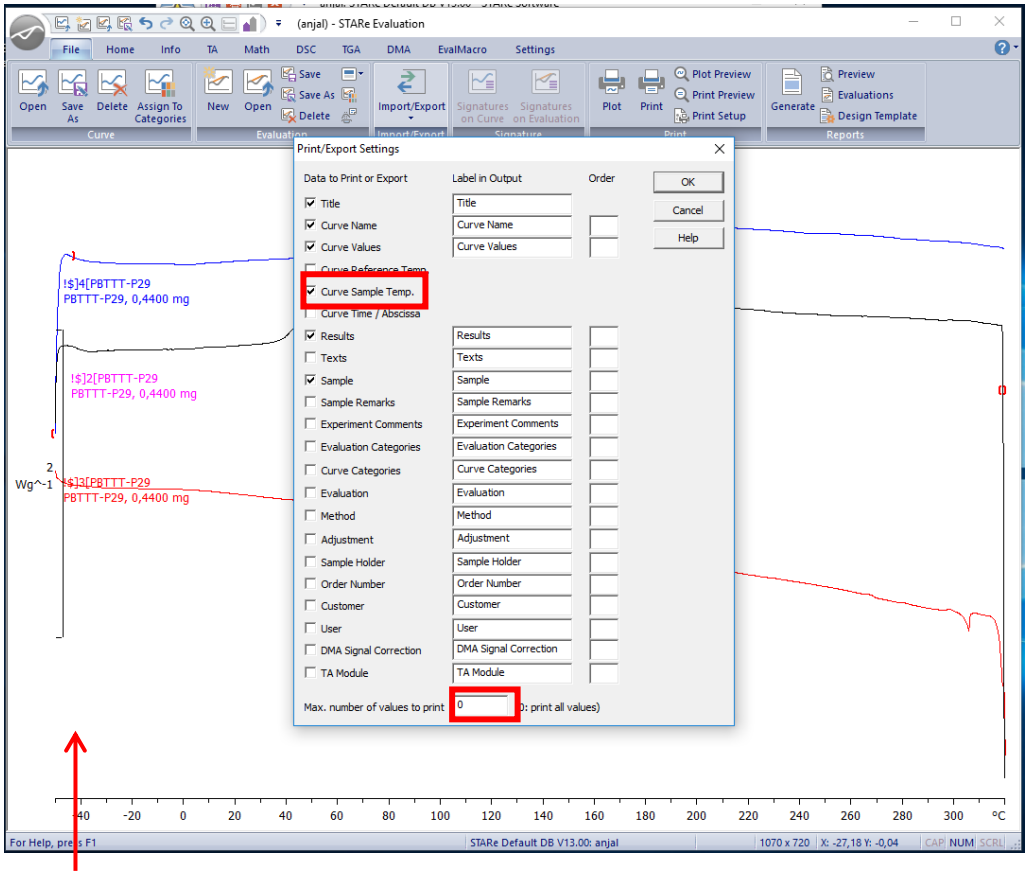
    b. In the Evaluation Window, go to File -> Open



    c. In the pop-up window, select the test segments of interest.



d. Select File -> Import/Export -> Export other format -> Select .txt format. In the Print Export Settings dialogue box, make sure to tick the box for Curve Sample Temp. and to set the Max. number of values to print to “0” (!).
If you don’t get this prompt, close and re-open the Evaluation Window.



note: red brackets indicate unreliable data

The evaluation software is available for free download from Mettler Toledo:

https://www.mt.com/se/sv/home/perm-lp/product-organizations/ana/ta-freesoftware.html