

DIFFERENTIAL SCANNING CALORIMETER (DSC) OPERATING INSTRUCTIONS



- ❖ Measure your sample weight by sensitive weighing machine carefully.
- ❖ Put your sample into the small pans and put the cover onto the pan and press it by pressing machine.
- ❖ Prepare your measurement conditions. If you want to do your experiment in N2 environment, open N2 tank valve and adjust it max. 0.5 atm.

CAUTION

If the pressure you give into the system is above 0.5 atm, the flowmeter of the instrument gets out of order.

- ❖ In order to perform very accurate experiment, do a correction experiment before you do real experiments. Correction file minimizes the effects of instrument errors on results of the sample measurements.
- ❖ Ensure that you have enough liquid nitrogen for cooling system.
- ❖ From the file menu, select new for correction file.

DSC 204 F1 Measurement Header

Measurement Type: Correction
 Sample+Correction
 Sample

Laboratory: x
Project: x
Operator: x
Date: 10/06/08; 18:14:44
Material: [dropdown]

Instrument Setup Information
Crucible Type: Pan Al, pierced lid
Sample Carrier: DSC 204F1 t-sensor
Sample Carrier TC: E
Furnace: Standard DSC 204F1
Furnace TC: E
Measurement Mode: DSC
Temp. limit: No special temp. limitations

Sample
Ident: [text]
Name: aerogel-D5
Sample Mass: 0 mg
Crucible Mass: 0.000 mg

Reference
Name: x
Reference Mass: [text] mg
Crucible Mass: 0.000 mg

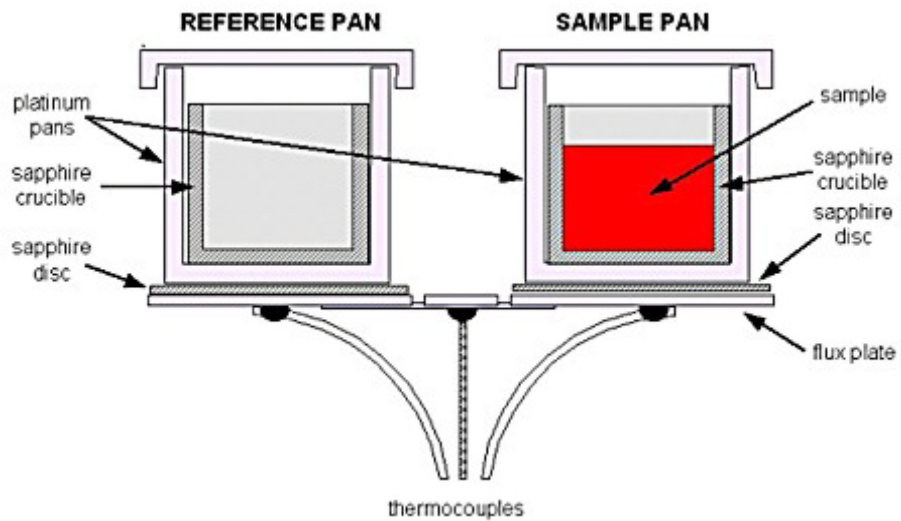
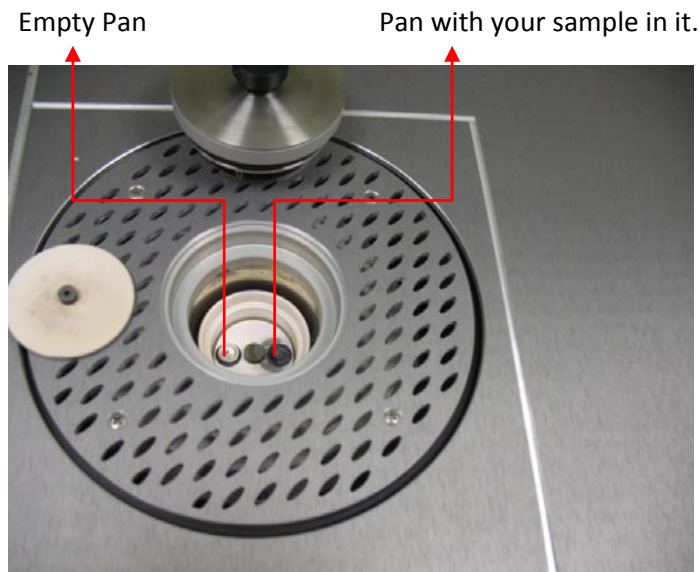
Remark: [text area]

Current hardware temperature range is from -195 °C to 600 °C

Buttons: Help, CANCEL, OK, Continue ->

Fig. 1 Correction File Window

- ❖ Before you do correction file preparation, put a blank pan in right side of the sample holder and a blank pan on the left side and close the cover of the sample compartment.



- ❖ Select correction radio button and fill all other areas in the window. (Ref mass=0mg)
- ❖ Select the calibration file and sensitivity file after pressing continue button.
- ❖ After that you will have a popup window that represents the temperature profile and selection of gas environment.

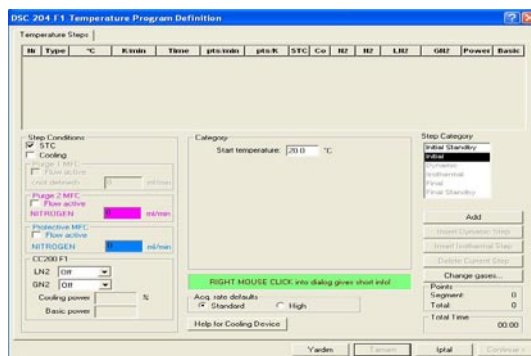


Fig. 2 Temperature Profile Preparation Window

- ❖ The window above, define the heating and cooling steps from the start point till the final step by simply adding individual dynamic segments.

CAUTION

1. If your experiment is under gas environment, select protective and purge 2 gases and write down the flow rates. (For example for protective gas, it should be around 70 ml/min and the sample compartment gas should be 30-40 ml/min).
2. Do not forget to open liquid nitrogen which must be used during cooling segments. You must open it only cooling steps by just clicking **LN2 tab** related these steps.

- ❖ Save the correction file.
- ❖ After experiment done, correction file created.
- ❖ From the file menu, this time open the correction file.
- ❖ Then you will have a window below that explains all the experiment details like steps you done, gases you used, flow rates, heating and coolings rates you done in correction file.,
- ❖ Click on sample+correction radio button. Fill out the other areas. Give your sample weight in sample mass area and write down 0 for crucible mass.

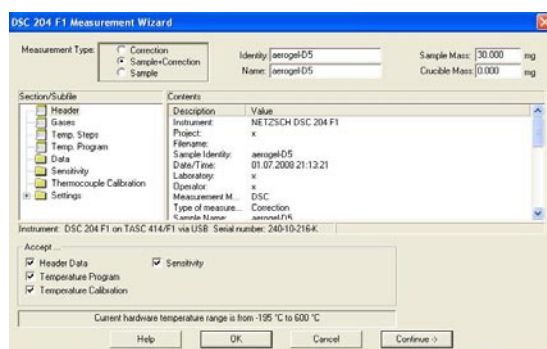


Fig.3 Correction File Open Window

- ❖ Put your put the pan with the sample on the right side and take out the empty one. (left handside empty pan remains)
- ❖ After experiment finished, investigate your data with the help of analyse program.