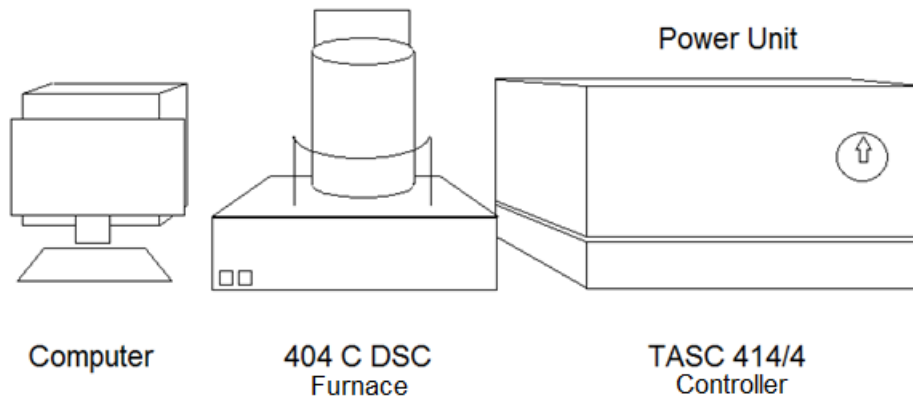


404 C DSC "Pegasus" Quick Reference Guide



Powering on the instrument:

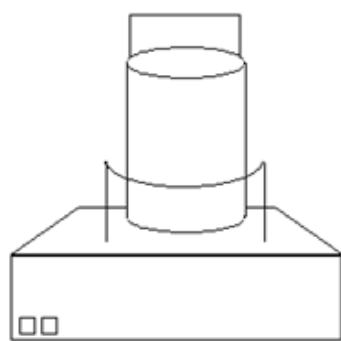
1. Each instrument component is powered on by a physical switch on their cases.
 - a. The 404 C DSC switch is located on the left side at the back of the case.
 - b. The TASC 414/4 switch is located on the right side at the back of the case.
 - c. The power unit is controlled by a red dial on the front of the case. It must be rotated to on.
2. Allow the components a minimum of 15 minutes to warm up.
3. If you are planning to perform a measurement in a specialized environment then verify that the correct gas cylinder is connected to the instrument and that the cylinder valve is open.
4. Log into FBS and activate your reservation for the instrument to unlock computer use.

Note: The instrument will usually be powered on already.

Prepping your sample:

1. Each measurement requires two lidded crucibles of matching types. One will hold the sample and one will be empty and function as a reference. Both Al_2O_3 and Pt-Rb crucibles may be used. When choosing a crucible **make sure that your sample will not react with the crucible** type and that the crucible is appropriate for your measurement and temperature range.
2. It is generally wise to pre-heat a crucible to your desired maximum heat before using it in the DSC.
3. Take the mass of your sample crucible and reference crucible with their respective lids.
4. Take the mass of your sample. **All samples must be tested in the TGA for volatility or decomposition before they are used in the DSC.** Samples showing decomposition in their measurement temperature range cannot be run.

Note: Failure to use the correct crucible or adequately test for volatility could destroy the sensor head. The advisors of negligent users may be responsible for paying for the \$8,000 replacement.



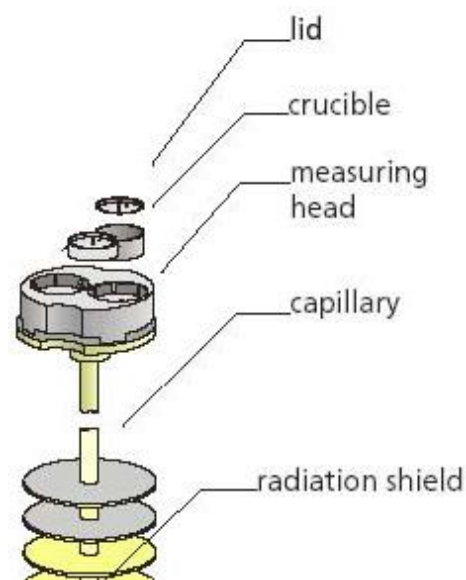
404 C DSC
Furnace

Loading your sample:

1. Open the DSC 404C software on the computer and verify that the DSC is at room temperature by looking at the status in the bottom right corner of the screen.
2. Verify that the “Vacuum” panel on the front of the furnace does not have a red led light on.
3. To open the furnace hold the safety button located on the right side panel of the furnace base and press the button with the upward arrow on the front of the furnace base. Both buttons must be pressed for the furnace to move.

Note: Do not open the furnace unless it is below 100 °C.

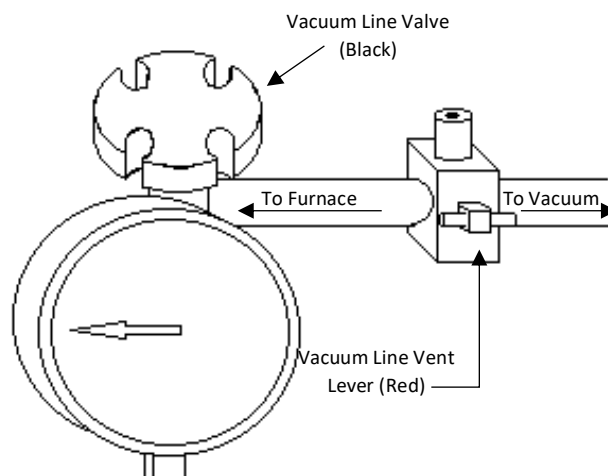
4. Once the furnace has raised an appropriate height use tweezers to gently place your crucibles on the sample holder.
 - a. The reference crucible is placed in the back.
 - b. The sample crucible is placed in the front.
5. You must be very gentle when placing the crucible on the sensor head as minor disturbances will affect the baseline. Use the green fencing around the furnace to stabilize your hands.
6. Ensure both crucibles sit flatly on the sensor and are generally in the center.
7. Lower the furnace by holding the safety and downward arrow buttons. **Visually inspect to make sure that the sample holder will not hit the furnace.** Once you have checked that it is clear, completely lower the furnace. Once fully lowered a green led will appear on the downward arrow on the front of the DSC.



Purging the Furnace Chamber:

1. Check for a green LED on the downward arrow on the front of the DSC furnace base to make sure the furnace is fully lowered.
2. Check the front of the furnace base for the absence of green LED indicator lights on the purge 1 and 2 buttons. No gas should be flowing.
3. On top of the furnace, **above the sample chamber**, turn the arrow shaped black lever to the right to close the sample chamber vent.
4. Turn on the red vacuum pump located on the shelf above the DSC.

- The vacuum pump has a line connecting to the right-back side of the DSC furnace unit. The line is fitted with a manometer, black handwheel and a small valve with a red lever. Rotate the red lever until it is parallel to the tubing, as shown in the diagram to the right. This closes the vacuum line.
- Slowly open the vacuum line valve by rotating the hand wheel. Opening the valve rapidly may displace the crucibles in the furnace. The manometer should show the pressure decreasing.
- Allow the furnace to purge for approximately fifteen minutes. A red light should display on the front of the furnace indicating when a vacuum is established.
- Close the line by using the handwheel to close the vacuum line valve. Turn off the pump and turn the red level until it is perpendicular, to vent the line.
- On the front of the module press the button for the purge gas you intend to use.
- Allow the furnace some time to backfill, but watch the pressure using the manometer at the back of the DSC. Once atmospheric pressure has been reached open the exhaust valve on the top of the furnace sample chamber. The black arrow should be pointing away from you. This process should generally be repeated three times to ensure an oxygen-poor environment.



Note: The exhaust valve on the top of the furnace must be open when performing a measurement.

Running a Measurement:

- Open the DSC 404C software on the DSC computer. The icon is located on the desktop.
- At the top of the software select the *File* tab and from the drop-down menu select *New*.

DSC 404C Measurement Header

Measurement Type: Correction
 Sample+Correction
 Sample

Laboratory: MRL
Project: Black Pearl
Operator: Jack Sparrow
Date: 02/26/09; 11:37:46
Material: baseline

Instrument Setup Information
Crucible Type: DSC/TG pan Pt-Rh
Sample Carrier: DSC(TG) HIGH RG 2
Sample Carrier TC: |S|
Furnace: STD Pt-Rh
Furnace TC: |S|
Measurement Mode: DSC
Temp. limit: No special temp. limitations

Sample
Ident: SP245
Name: black pearl
Sample Mass: 0 mg
Crucible Mass: 252.400 mg

Reference
Name: empty pan
Reference Mass: mg
Crucible Mass: 261.400 mg

Remark:

Purge Gas 1: argon
Flow Rate: 50 ml/min

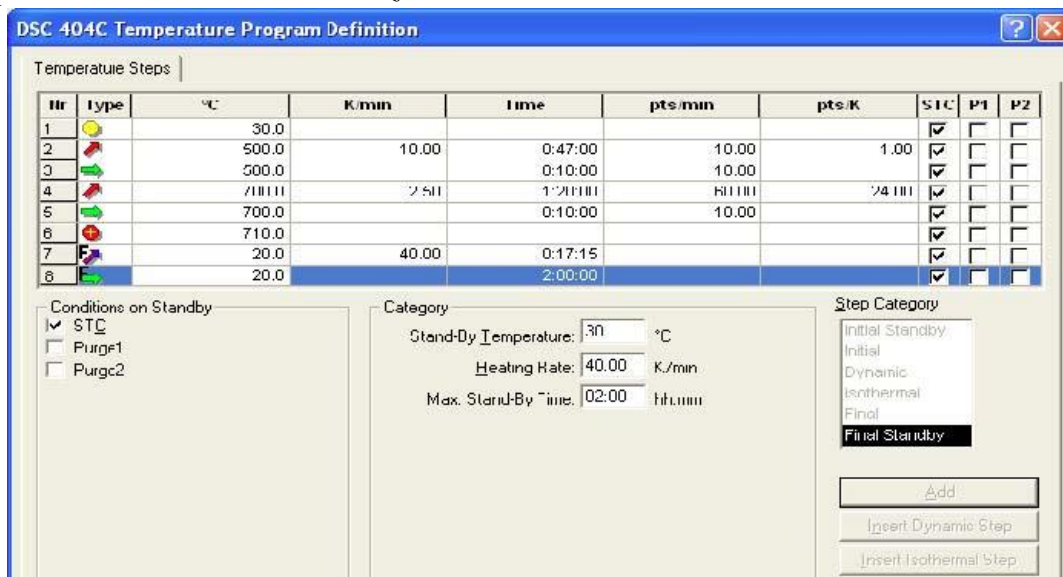
Purge Gas 2:
Flow Rate: ml/min

Help on Crucible Selection

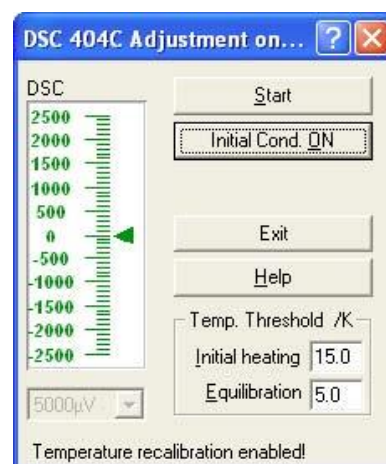
Current hardware temperature range is from 0 °C to 1500 °C

Buttons: Help, CANCEL, OK, Continue ->

3. In the Measurement Header window that appears enter the relevant information for your measurement. This will include
 - a. Your sample identity
 - b. Your sample crucible and lid mass
 - c. Your sample mass if you are running a sample or standard
 - d. The reference crucible mass
4. Confirm that the Crucible Type is correct for your crucible and then select *Continue*
5. Select the most recent calibration file and then select *Continue*.
6. Select a sensitivity file. The sensitivity calibration file must match your experimental parameters including heating rate, gas atmosphere and crucible type. If you are performing a measurement for phase transition data select *senzzero* and then *Continue*.



7. Design your temperature program. During this time you will set
 - a. Your heating rate
 - b. Your data acquisition rate
 - c. The gas type for your experiment
8. When complete select *Continue*.
9. Name the data file and select where your data will be saved.
10. In the following window you may select *Initial Cond. ON* to instruct the furnace to begin heating to your start temperature. Once it reaches the starting temperature the system will attempt to equilibrate.
11. Before starting, **visually confirm that the vent on top of the furnace sample chamber is open and the arrow is pointing toward the back of the instrument.**
12. Press *Start* to begin the measurement.



DSC User Application

Name: _____

Date of DSC Training: _____

Department/Company Name: _____

PI Advisor/Supervisor: _____

Full E-mail Address: _____

Campus Phone #: _____

Position at UCSB:

Undergraduate Student

Graduate Student

Post Doc

Visitor

Staff

Other _____

If you are a visitor what is your home institution or company? _____

What 13 digit recharge number will be used for your measurements?

8-_____-_____-3

Have you taken UCSB EH&S Laboratory Safety training? When and in what format?

What type of samples will you be running on the DSC?