

Instructions for Perkin Elmer DSC 4 and System 4 Controller

To Set the Temperature Program on the System 4 Controller

The System 4 controller is used to set the minimum and maximum temperatures and the heating rate. In the instructions below, keys on the controller are shown in [brackets]. The general procedure is as follows:

1. Make sure the [MANUAL] light is on.
2. Press [SET UP].
3. The Tmin light should now be on. Type in your Tmin and [ENTER].
4. The Tmax light should now be on. Type in your Tmax and [ENTER].
5. The HEAT RATE light should now be on. Type in your heat rate and [ENTER]. $20^{\circ}\text{C min}^{-1}$ is a good starting point, unless you are told otherwise.
6. The COOL RATE light should now be on. Type in 0 and [ENTER]. A cool rate of 0 gives the fastest cool rate possible. When the cool rate is 0 the system automatically returns to Tmin when the temperature program is completed.
7. The T SPAN light should now be on. Just press [ENTER]. Leave the T SPAN set at 400; this setting is used with x-y recorders, and has no effect on our method.
8. The [SET UP] light should now go out.
8. Press [HEAT] or [COOL] to begin the program depending on the direction you set up.

To Get to the Starting Temperature

When the instrument is turned on, the instrument is set at 25°C . Usually you want to begin your run at some other temperature. To get to the starting temperature:

1. Press [LOAD TEMP] and [MODIFY].
2. Type in your desired starting temperature and [ENTER].
3. Press [RESET] and [GO TO LOAD] at the same time. The system will go to the load temperature at the maximum rate.
4. When the system is equilibrated at the load temperature, the Load light will come on and the [GO TO LOAD] light will go out.

If your Tmin is well above room temperature, it is best to load the sample pan at 25°C and then [GO TO LOAD] to get to your Tmin.

Step-by-Step Instructions

To load your sample:

1. Load your sample at or near room temperature.
2. Lift the lever on the furnace cover. Swing the cover to the right.
3. Gently lift the sample cover by inserting the tips of the cover forceps into the holes. The covers are made of platinum and are very easy to deform. *Please be very gentle with the covers to avoid damage.*
4. Center your sample pan in the holder .
5. Replace the cover. Be sure to align the holes in the cover in the same orientation each time.

The orientation of the holes changes the heat capacity of the cell.

6. Swing the furnace cover back and depress the lever. You should feel a small amount of resistance as you press the lever. If you don't, tighten the knurled nut at the back of the furnace cover slightly.
7. Turn on the nitrogen flow, and set the regulator at 20 psi. The nitrogen valve and regulator is mounted on the wall.
8. Get to the starting temperature, see above.
9. Set the Temperature Program, see above.

Starting Up Logger Pro Data Acquisition Software

10. Log in to the Mac and start up Logger Pro (double click on the logger Pro Icon).
11. Pull down the file menu and choose Open. Switch to the "Documents" folder.
12. For most purposes you will use the "DSCautostart.cmbl" data set up. However, if you wish to record the baseline before the temperature program begins, choose the "DSCdelayedstart.cmbl" set up. The DSCdelayedstart.cmbl is the appropriate set up for determining the heat capacity of your sample.
13. Enter your temperature scan rate and starting temperature in the Calculations window for the Temperature column as follows. Double click on the Temperature column heading in the spreadsheet window. The Column Options window will open. Click on the Column Definitions tab if it is not already selected. In this window the default temperature scan setting is 20 °C/min and the starting temperature as 25°C giving the definition: "Time"/60*20+25. Change these values to your conditions. For example if you are using a 10 °C/min temperature scan rate and the starting temperature as 50°C:

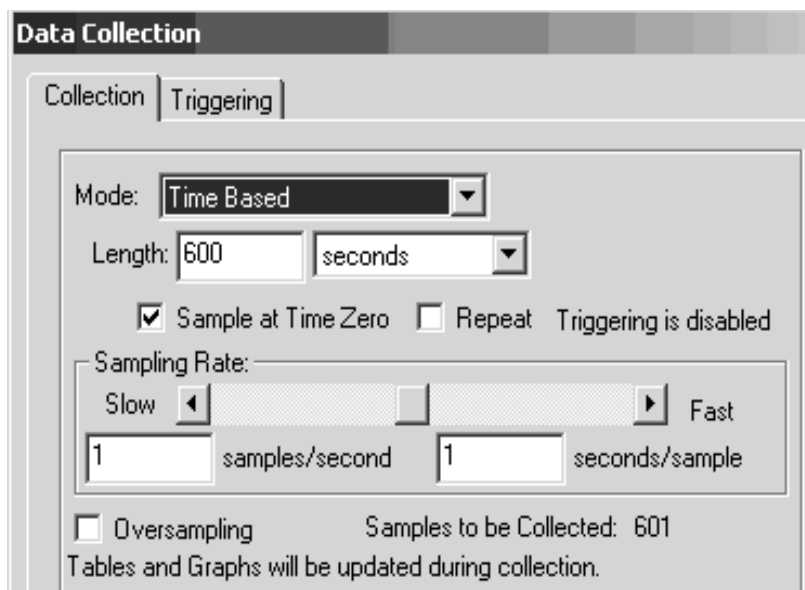
The screenshot shows the 'Column Definition' dialog box with the 'Options' tab active. The 'Labels and Units' section contains a text field for 'Name' with the value 'Temperature', a text field for 'Short Name' with the value 'T', a text field for 'Units' with the value '°C', and a dropdown menu for 'Insert Symbol'. The 'Equation' section contains a text field with the equation '"Time"/60*10+50'. Below the equation field are three buttons: 'Functions >', 'Variables (Columns):', and 'Constants:'. At the bottom left, there is a checkbox labeled 'Show Live Readouts' which is currently unchecked.

Then click on Done.

If you are using the delayed start version, the default Equation will be listed as ("Time"/60 - 0.5)*20+25. The "-0.5" provides a 30 sec initial period to record the baseline before the temperature scan begins. Once again, you should change the scan rate and starting temperature to the values you are using. Click on Done.

14. The data acquisition rate is set to 1 sample per second for 600 total seconds. These settings are fine for most work. If so skip to the next step. If you need to change the data acquisition

rate, click on the “stop watch” icon in the top icon bar. The Data Collection window will open.



Change the values to fit your experiment. For slow temperature scan rates you should use a larger value for the seconds per sample setting. For 2°C per minute, you might try 10 seconds per sample for 10 – 20 minutes.

Your Run

15. In Logger Pro, if you have a previous data set, pull down the Data menu and choose Clear All Data.
16. If you used [GO TO LOAD] wait until the load light is on.
17. Lift the door on the front of the DSC-4 console. Set the desired RANGE using the large black knob.
18. Using the ZERO knob on the front of the DSC-4 console, adjust the output voltage to a value near the bottom of the chart scale (near 0.2 mV). You can read the output voltage in the Logger Pro window on the left-hand side just under the icon bar and above the spreadsheet window.
19. Click on Collect in Logger Pro. Logger Pro will start the scan automatically.
20. You may stop a run at any time by pressing the COOL button on the DSC controller and clicking on STOP on Logger Pro. At the end of the run save you data to a new file name. Use the Documents folder to save your data. Then do the required data analysis steps.
21. After the system returns to T_{min}, you can repeat your run after the READY light is lit.
22. To change samples, if your T_{min} is not close to room temperature, use the [GO TO LOAD] procedure, above, to return the DSC to 25°C.