TA Instruments DSCRun™ Software Getting Started Guide

Instrument Types Supported

DSCRun software is used to control the operation of the Nano DSC Differential Scanning Calorimeter.

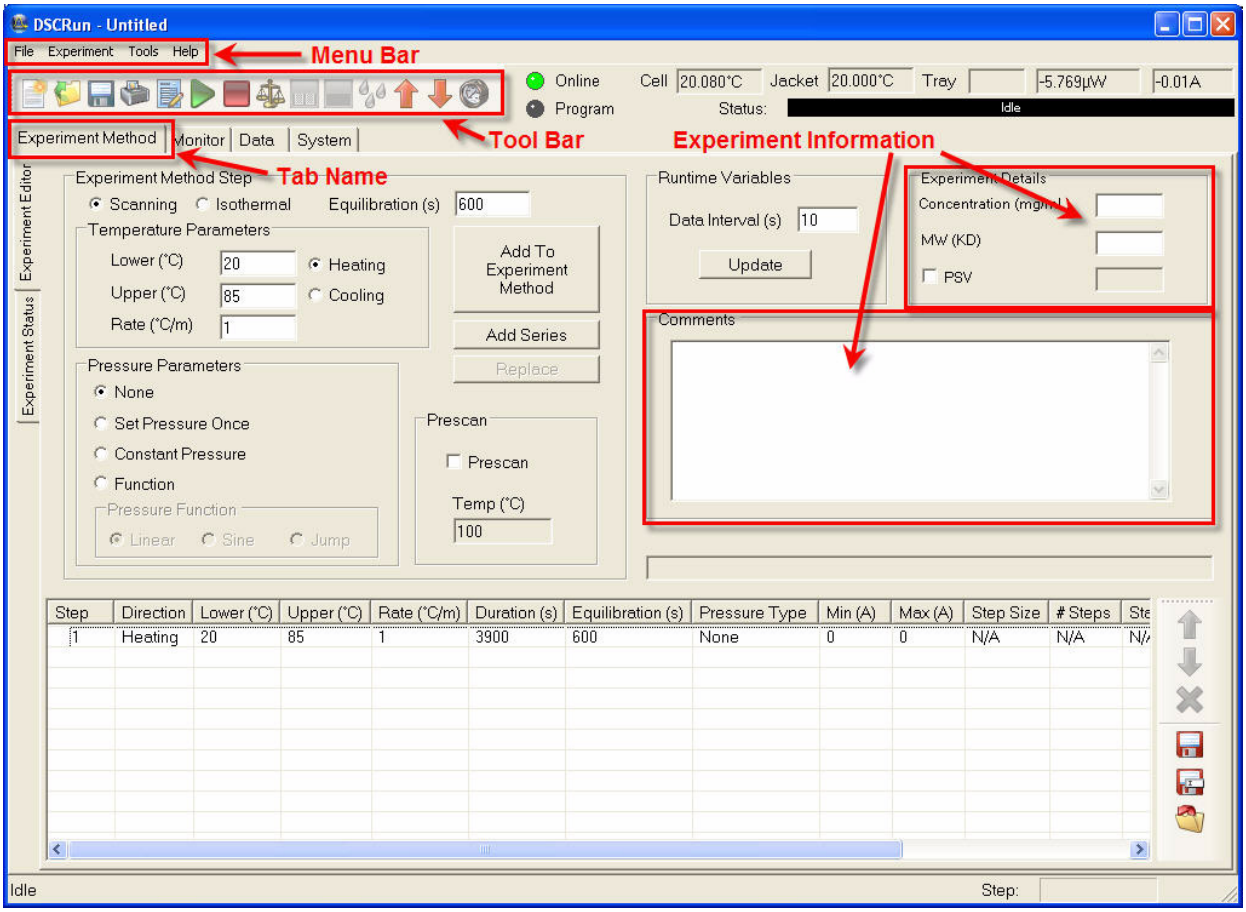
Getting Ready

1. Install the DSCRun software, instrument drivers, and National Instruments DAQ interface card before connecting the Nano DSC instrument to the computer.
2. Connect a power cable to the instrument and a power outlet.
3. Connect the SCSI cable between the instrument and the computer.
4. Set the power switch (located at the rear panel of the instrument) to on. A green light appears on the front window of the instrument.

Note for IT personnel: Since the data control and collection software depends on accurate timings, it is highly recommended to set the computer BIOS settings for performance rather than for power savings. Some computer manufacturers may have different names for this. For example, some Dell computer have a setting called “C-States” that includes the C1E setting, which should be disabled. Other computer manufacturers may call it “Enhanced Halt State”. Other settings that should be disabled (if available) are EIST (Intel SpeedStep) and AMD’s Cool ‘n’ Quiet.

Starting DSCRun Software

1 Double-click the **DSCRun** desktop icon or select the **DSCRun** shortcut in the Windows **Start** menu. The program opens a window on the desktop.



2 The following functions are located on the **Menu** bar:

- File
- Experiment
- Tools
- Help

3 The following functions are located on the toolbar:

- New File
- Open File
- Save File
- Print
- Run Experiment
- Balance Cells

- Gauss
- Pulse
- Pressurize
- Connection status indicator (green circle)
- Experiment in progress indicator (circle, red when activated)
- The system status is displayed at the upper-right of the **Experiment Method** page.

4 Additional toolbar Items

- Cell temperature
- Jacket temperature
- Heat rate
- Pressure in atmospheres

5 New Feature: Experiment Details

- Enter sample information here before starting an experiment. The data is automatically transmitted to NanoAnalyze software during data analysis.

Experiment Method Page

Instrument Controls

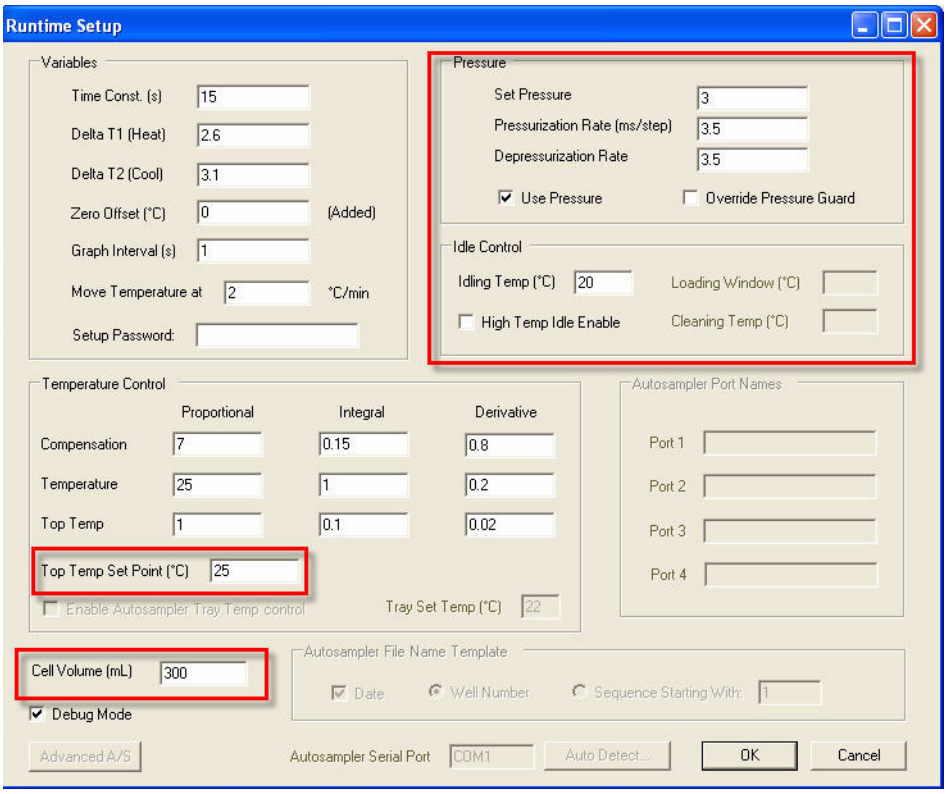
Experiment Setup: Select scanning or isothermal operation, temperature(s), rates, and the direction of the initial scan. When the **Heating** direction is selected, the instrument initially moves to the lower temperature limit. The first scan is then collected in the heating direction.

Pressure Parameters

Options include no pressure applied, set pressure once, constant pressure, and a user-defined pressure modulation function.

Runtime Variables

Set the data acquisition interval and enter the DSC cell volume in microLiters (one time setup). The cell volume is saved in the data file and is automatically entered into NanoAnalyze software during data analysis.



Scan Schedule

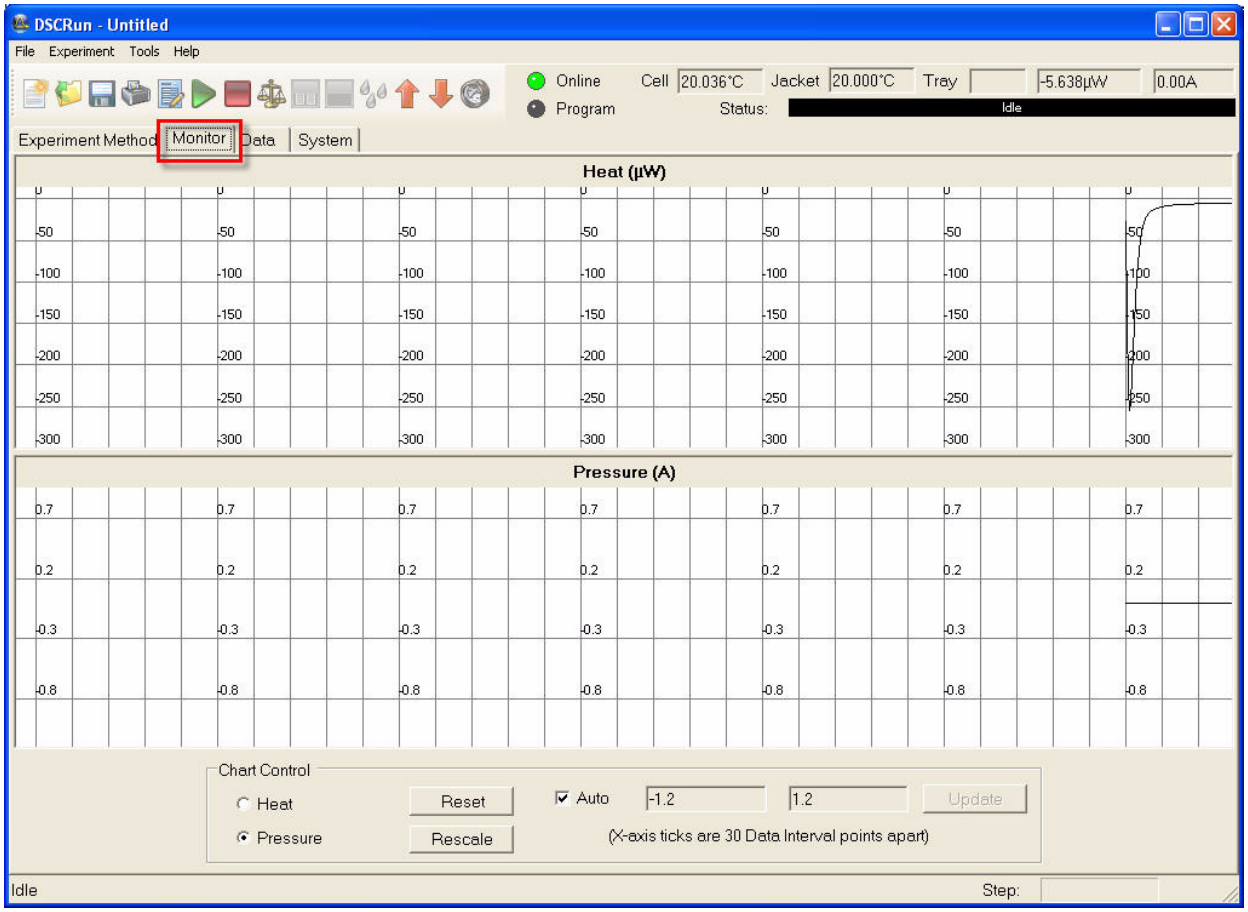
This is a table that lists the individual scans to be performed in the experiment. Use the **Experiment Setup** control group to define the steps, and use the **Add** or the **Add Series** buttons to populate the schedule table.

The DSC operating mode status display is the black bar at the upper right of the screen. The modes are:

- **Idle**: The DSCRun program is active but is not running scans.The Idle mode is the default mode of operation when DSCRun starts. The software holds the cell at the idle temperature. The idle temperature defaults to 25°C but can be changed by the user in the **Tools** menu, **Setup Runtime Variables**.
- **Moving Temperature**: In this mode the DSC is preparing to start the scan. To begin the scan, it moves the temperature of the cells to the starting temperature of the scan.
- **Equilibration**: When the Nano DSC finishes a scan and before it starts the next scan in the series, there is a 10 minute equilibration period. This can be changed in the runtime variables.
- **Heating/Cooling mode**: Heating or Cooling status will be shown in the status bar. A scan is in progress and data is collected and recorded in the specified data file.
- **Balance Mode**: Used only when the Nano DSC is running a balance.

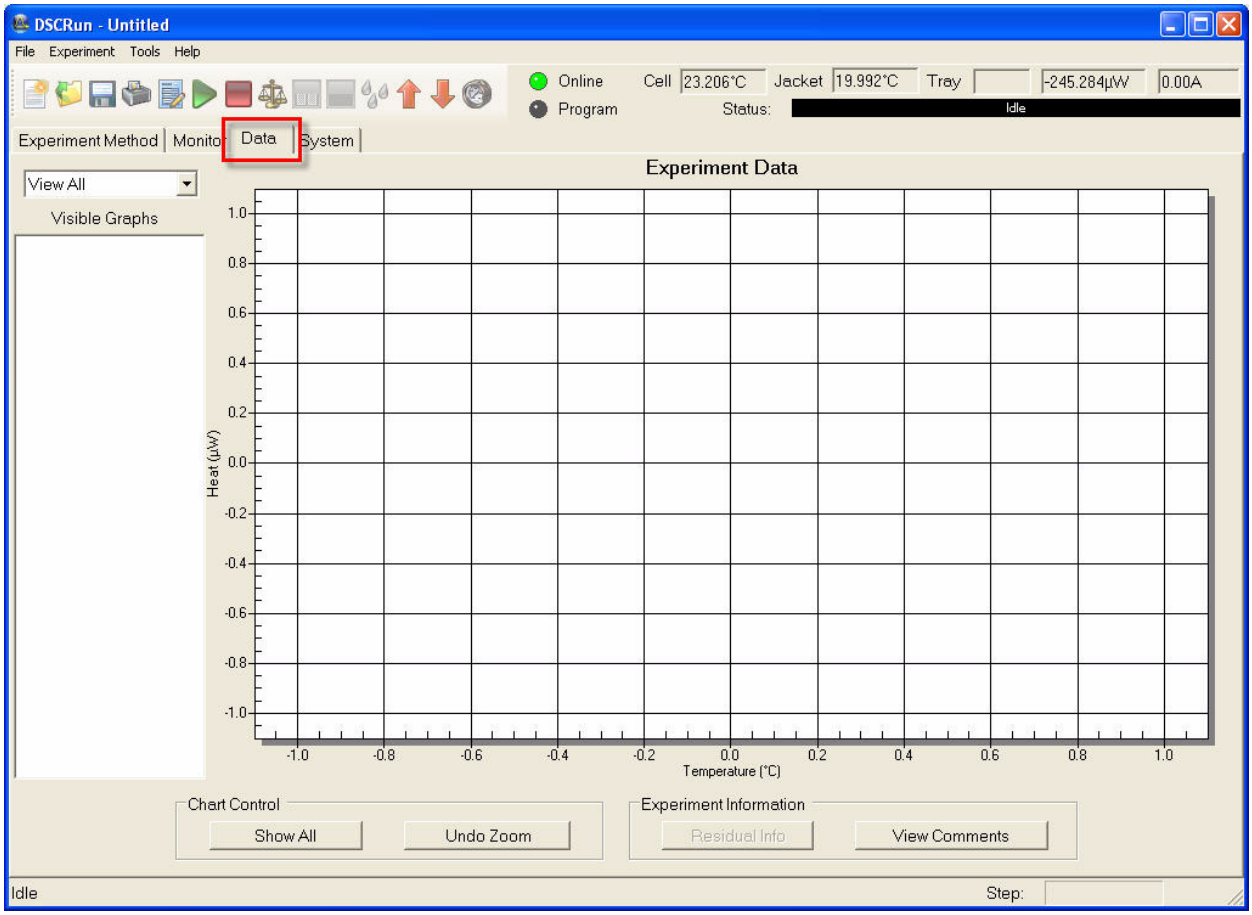
Monitor Page

The **Monitor** page is used to observe the heat and pressure signals. The charts run continuously to indicate the recent conditions of the heat and pressure channels. They can be auto-scaled or manually scaled to user-selected Y-axis ranges.



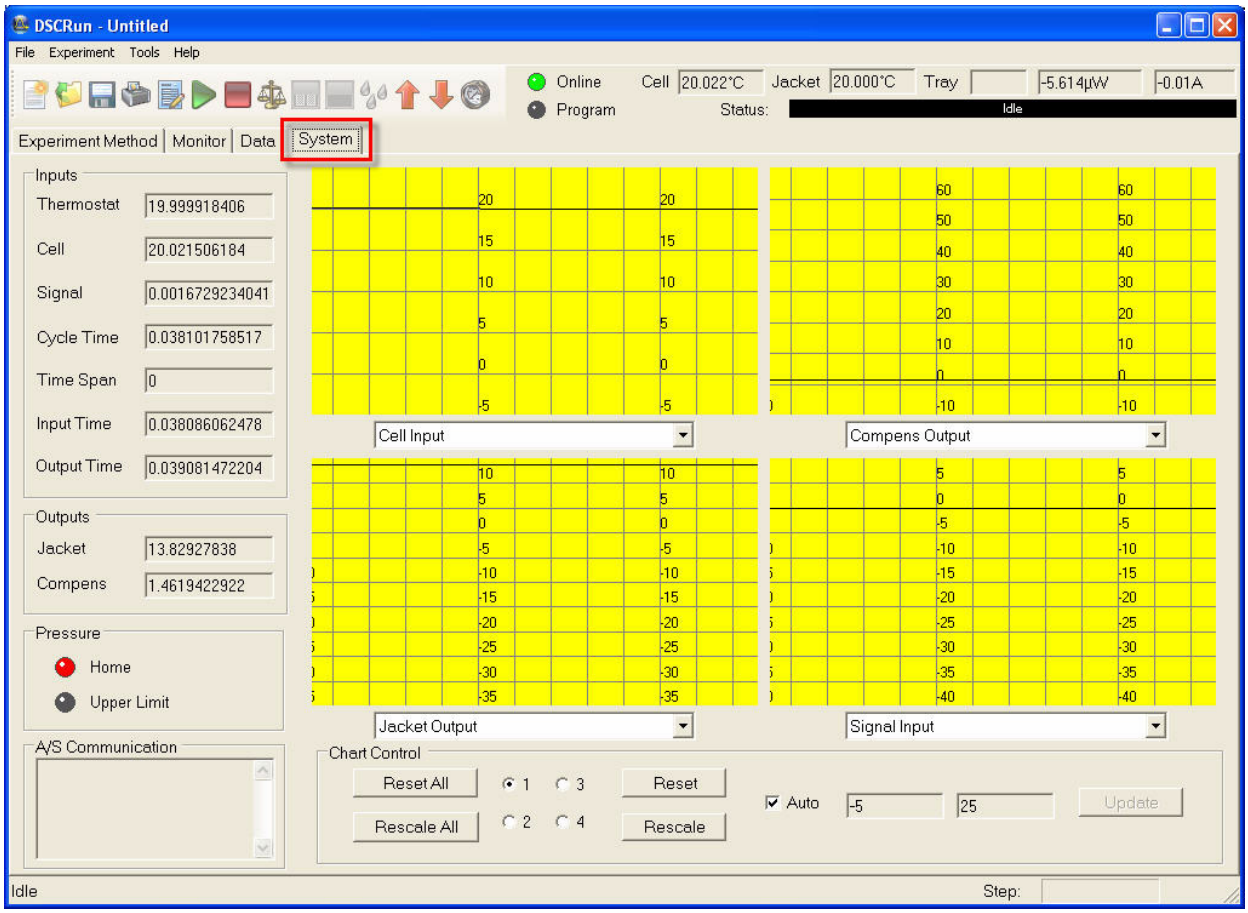
Data Page

The **Data** page is activated when an experiment begins. Using the drop-down menu, the data graph can be set to display scans only, isothermal steps only, or all steps. Experiment data charted on this page remains visible after the completion of an experiment.



System Page

The **System** page is used to display the recent and current status of internal zones of the instrument. The data continuously scrolls through to the left side of each chart. Each chart can be manually or automatically scaled. The most recent measurements are shown numerically on the left side of the page.



Experiment Overview

A typical Nano DSC experiment involves the following:

1. Preparation of the buffer/solvent and sample solutions
2. Running the baseline and the sample solution scans
3. Cleaning the calorimeter
4. Performing a thermodynamic analysis

Sample handling and cleaning are discussed in the *Nano DSC Getting Started Guide*. Thermodynamic analysis is discussed in the *NanoAnalyze Software Getting Started Guide*.

Instrument Preparation

The Nano DSC is shipped from the factory ready to run experiments with clean and dry sample and reference cells. It should be noted, however, that chemical conditioning is always recommended any time a new buffer solution is used; this also applies to the first-time operation after delivery. One important function of the conditioning is to improve the wetting behavior of the interior surfaces of the cells in order to facilitate complete filling with no air bubbles. The cells can be conditioned by performing a scan across a broad temperature range using buffer in both the sample and reference.

Two tools in the software are used to obtain smooth and flat baselines, the balance scan and residual scans. The data collected during the balance scan is stored in the computer that is connected to the Nano DSC. New Nano DSC instrument systems are sold with the personal computer that was used during preparation and testing at TA Instruments, and the balance scan is already in place. If the Nano DSC is subsequently used with a different PC, a new balance scan must be performed. Residual scans are optional, and their data is also stored on the PC that is connected to the instrument.

The Nano DSC sample and reference cells were balanced at the factory before delivery. The cells are matched for thermal characteristics before assembly, but there are very small remaining differences that result in small heat flow differences, and therefore result in baseline tilts. The information obtained from the balance scan is used to keep the cells in thermal balance during all subsequent operation, and it is therefore required in order to obtain the best possible experiment data.

The balance may gradually shift over time, which would cause scan baselines to shift. If a water/water scan yields data that do not fall within a +/- 100 microwatt window, then the balance may need to be updated.

Performing Balance Scans

1. Load the reference and sample cells with degassed, deionized water. It is imperative that no bubbles are present in either cell. Any distortions in the balance will impair all data collected while that degraded balance is in place.
2. Install the pressure handle and click **Pressurize**. Double-check that the pressure does reach a value close to 3 atmospheres. If not, then there may be a problem with the o-ring seal.
3. Prepare the instrument by performing at least one (and preferably multiple) sequential heating scans that run from near-ambient temperature to an upper limit of approximately 120°C. This ensures a full wetting of the cell surfaces and prevents bubble formation during the balance.
4. From the **DSCRun** menu, select **Tools** > **Balance Cells**. The calorimeter performs a 0 to 125°C scan, both up and down. After the balance scan is complete, save the balance scan.

Performing Residual Scans

Sample scans that are collected within a narrower temperature range than the balance scan will contain scan artifacts of several microwatts due to the slightly differing thermal history prior to the start. These are cosmetic only and will cancel out during data analysis because the buffer scans that are run under the same conditions as the sample will contain identical artifacts. However, it is sometimes desirable to improve the visual presentation of the data while an experiment is in progress in order to more easily monitor the experiment and ensure that it is progressing as planned. For this reason, there is a **Residual scan** feature in DSCRun. The residual is subtracted from the data before the data is stored. The same residual scan is used to correct the sample and the baseline scans. It is important to keep in mind that using residual scans is optional, but whether you choose to use them or not, blank and sample scans must be performed under identical conditions. The same residual scan (or none) must be used for both the blank and sample experiments.

Without a residual scan, the signal subtraction works as indicated in Equation 1, in which "S" = sample heat effects, "B" = buffer heat effects, and "A" = scan artifacts.

Equation 1: (S + B + A) - (B + A) = S

Removing the artifacts from both the sample and blank scans replaces Equation 1 with the mathematically equivalent Equation 2:

Equation 2: (S + B) - (B) = S

The Residual Database

DSCRun software will automatically use a residual scan during an experiment according to these selection rules:

- The residual scan and the experiment scan must start at the same temperature.
- The scan rate in degrees per minute must match between the residual and the experiment.
- The upper temperature limit in the residual is at least as high as what will be used in the experiment.
- If multiple residual scans meet the criteria as listed above, the first one in the list that matches will be used. If desired, the residual database may be re-ordered so that a different qualifying residual will be used.

Follow these guidelines for the use of residuals:

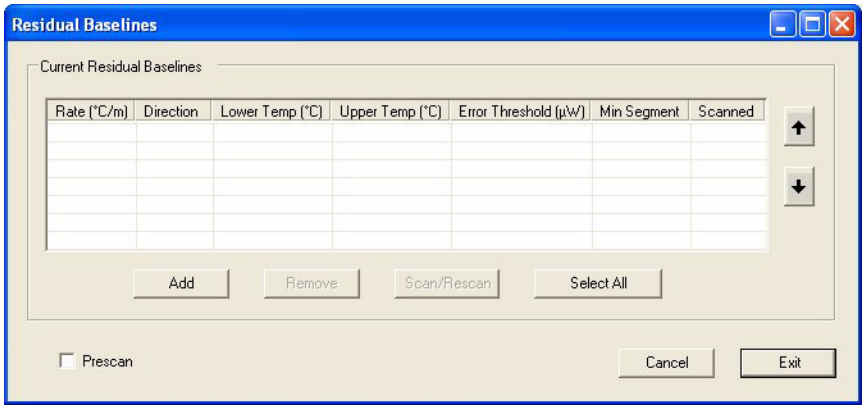
- If you choose to not use residuals, then both the blank and sample experiments should be run without them. Delete any matching residual scan files that may be present before running the experiments.
- If you choose to use a residual, use the same one for both the blank and sample experiments. Be sure that a matching residual is in place before running the experiments.
- If a new balance scan is performed, delete every residual in the database. Afterwards, create new residual scans as needed.
- If baselines are consistently several tens of microwatts above or below zero, a new residual and/or a new balance scan may need to be run.
- If a new residual scan is performed with identical parameters to an existing one, the prior scan will be replaced.

When a residual scan that has been collected over the same temperature range of the sample scan is available, the data is processed as follows:

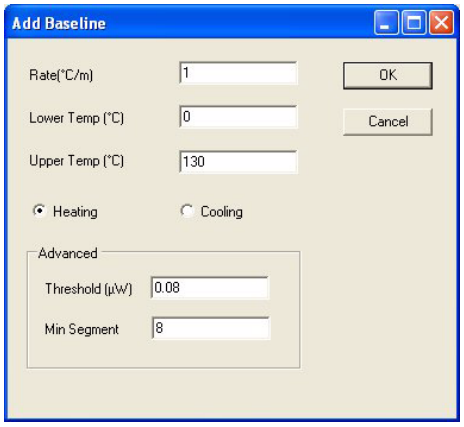
1 The balance information is used to fine-tune the thermal scan during an experiment. Raw data is acquired during the scan.

2 The data screen display will take the result of step 1 and subtract the residual scan data point by point along the scan. This same subtracted data is saved in the data file in the computer.

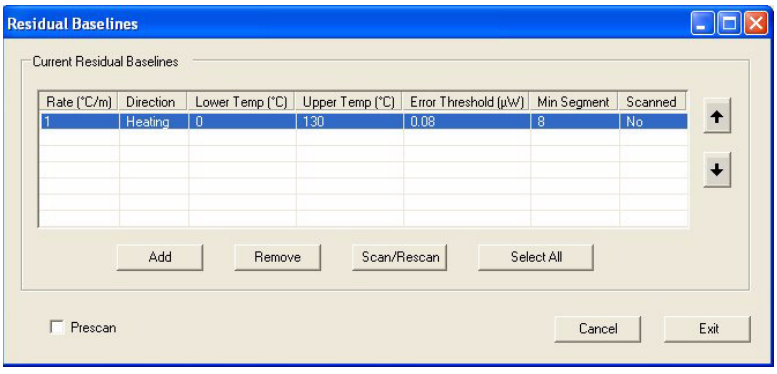
3 Prepare for the residual scan in the same method used for the balance scan (degassed deionized water in reference and sample cells, pressurize to 3 bar, with a conditioning scan performed beforehand). Select **Tools** > **Manage Residual Data Base**.



4 Click **Add**. Input the rate and temperature limits that match a sample experiment you will perform later. Click **OK** to save the scan parameters.



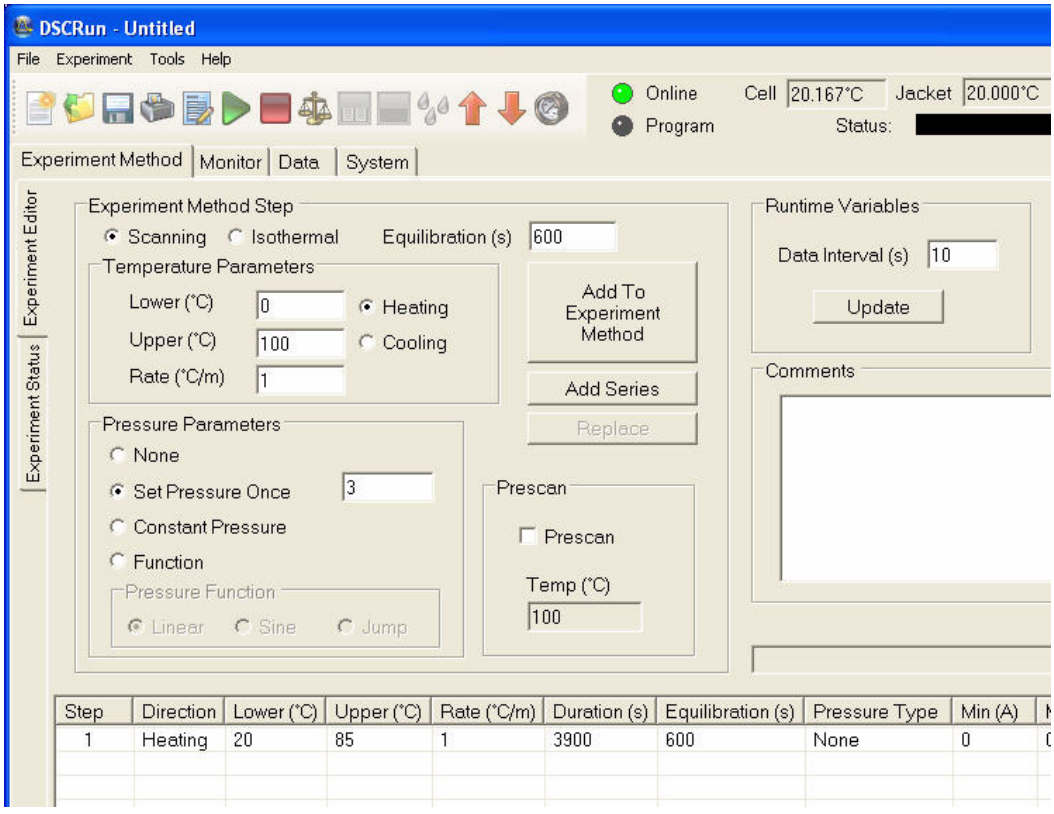
5 The residual scan is now resident in the database but is marked as not scanned. Select the scan by clicking the mouse cursor on it. This enables the **Remove** and **Scan/Rescan** buttons. Click **Scan/ Rescan** to allow the Residual scan to begin. Click **Exit** to proceed.



Loading the Buffer and Running a Baseline

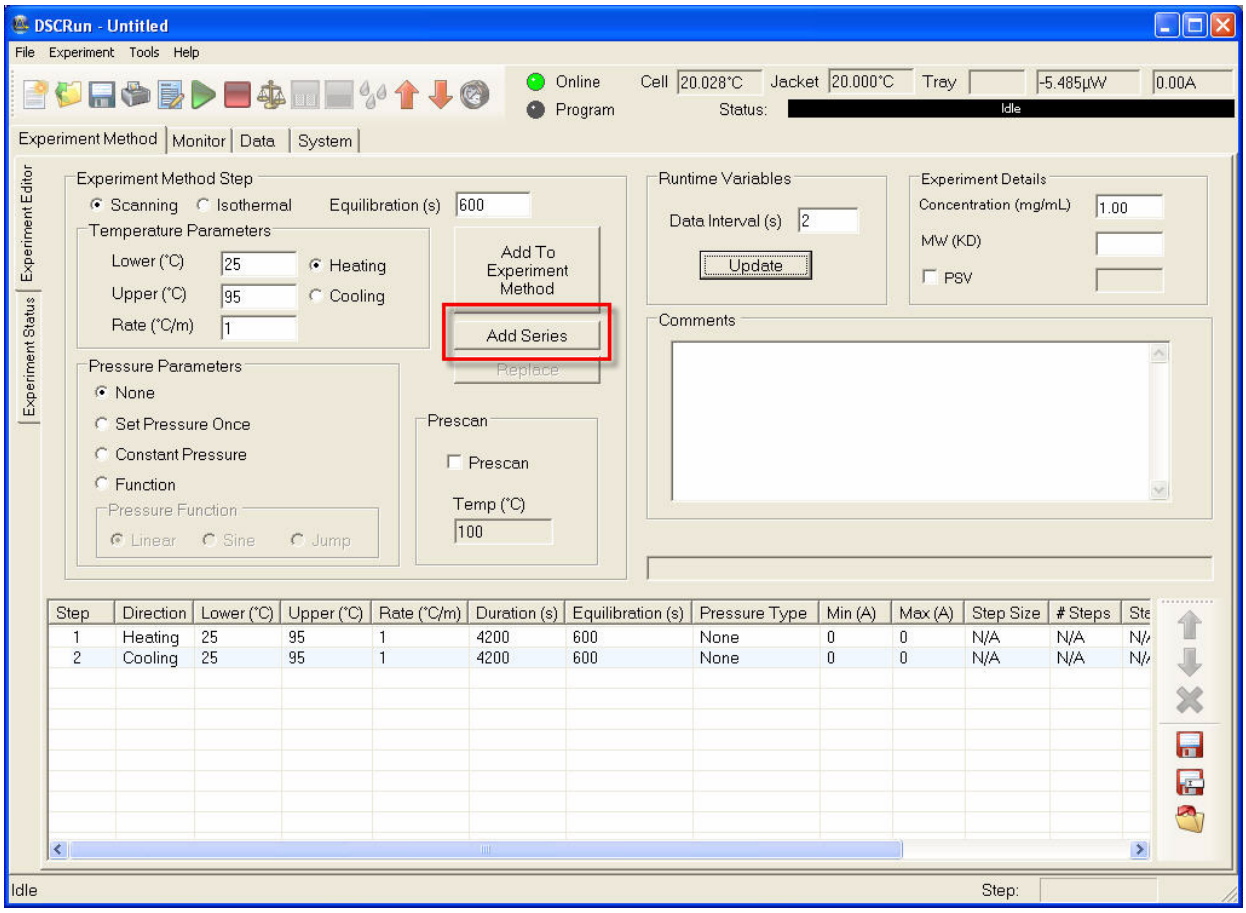
1. If the Nano DSC is pressurized, first set the cells to a temperature in the range of 20 to 30°C.
2. Release the pressure by clicking the down arrow on the main window, or selecting **Depressurize** in the **Tools** menu. The pressure and temperature can be monitored in the stats windows at the upper right of the program window, and also in the **Monitor** page.
3. Remove the pressure handle. The cell access ports are exposed when the pressure handle has been removed. The ports on the left access the reference cell and the ports on the right access the sample cell. “R” and “S” marks are present at the ports.
4. A baseline scan is performed with both the reference and sample cells filled with degassed buffer. Refer to the *Nano DSC Getting Started Guide* for detailed sample handling instructions.
5. Re-attach the pressure handle and pressurize the system by clicking the up-arrow control on the main window, or selecting **Pressurize** in the **Tools** menu. Typically set the pressure to 3 bar (atmospheres) when aqueous solutions are scanned up to 100°C.
6. On the **Experiment Method** page, select the desired **Pressure Parameters**.
7. If a number is entered in the **Pressure** section of the **Runtime Variables**, when the **Pressure Parameters** on the **Experiment Method** page is set to **None**, the Nano DSC pressure system automatically sets the pressure in a scan to the **Runtime Variables** value when the **Pressurize** up-arrow is activated or **Pressurize** is selected in the **Tools** menu.

NOTE: If there are program steps listed in the schedule, delete them. To delete a step, select it by clicking it, then press the **Delete** key on the computer. A series of steps can be deleted by selecting the first step, holding down the **Shift** key while selecting the final step, then pressing **Delete**.



8 A typical Lysozyme/Glycine demonstration scan would use limits of 25°C and 95°C, heating direction. 1°C/min scan rate, # of scans = 2. Set the temperature parameters in the **Experiment Setup** group, then use **Add Series** to add two scans to the schedule.

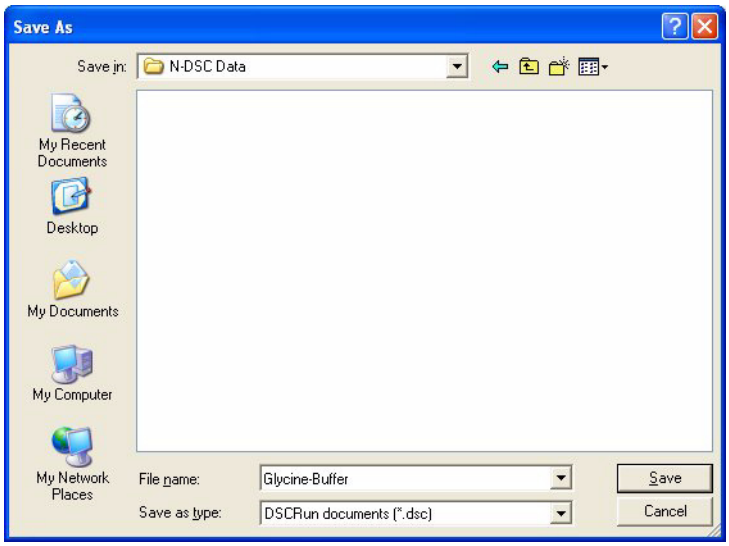
- A single individual heating scan can be added by setting the **Lower (°C)**, **Upper (°C)** and **Rate (°C/min)** and then clicking **Add**.
- To remove a step or steps from the list, highlight the step or steps to be removed and click **Remove**.
- To replace a step or steps already on the list, set the desired **Temperature Parameters**, highlight the step or steps to be replaced, and click **Replace**.



Performing Conditioning Scans

Initial scans should always be conditioning scans. The actual buffer baseline scans are performed later. Any chemical interactions that happen between the buffer and the cell walls, or residual cleaning solutions on the walls, take place during this scan, and significant shifts or noise may occur. The experiment steps described in this example will require about 1.5 hours to complete.

1 To start the conditioning scan, add the necessary scan steps with the **Temperature Parameters** identical to what the first buffer baseline scan will be. Then select **Start** in the **Experiment** menu, or click the **Run Experiment** icon in the main program window. Enter the file name for the data storage.



2 The status updated to **Moving Temperature**, the **Program** indicator turns red, and the first experiment step becomes highlighted. The instrument moves the cells to the start temperature at this time.

3 When the condition scans are complete, replace the old buffer with fresh buffer. The conditioning can be thought of as the last step in the cleaning process in preparation of actual sample data. Do not perform any additional cleaning steps at this time.

4 Before beginning the buffer baseline scans, clear the conditioning scans by selecting **File** > **New**.

Performing Buffer Baseline and Sample Scans

Perform these scans in the same way as the conditioning scans. The first scan starts with buffer loaded into both the reference and sample cells. After it completes, load the sample “on the fly” for the best results. The second scan, therefore, will have the sample solution in place in the Sample cell at the beginning of the second scan.

It is important that the buffer used for the buffer baseline scan and the sample be equilibrated at the starting temperature of the scans. This will ensure the that the instrument will come to a stable equilibration in the shortest time possible.

Loading the Sample on the Fly

When the purpose of the experiment is the measure the partial molar Heat Capacity Cp, the signal takes the form of the shift in baseline between the sample and blank scans. However, the initial scan in an experiment will often already have a shifted baseline because it has a unique thermal history. This is caused by the timing of the heat flows throughout the instrument that occur during dynamic scanning conditions.

When measuring Cp, the sample should be loaded during a down-scan at a near-ambient temperature condition of the sample cell. Sample and blank scans should be obtained in the same way, with the pressure handle re-sealed at matching cell temperatures. This prevents unwanted differences in cell pressure. (In all other experiment types, load the sample in a static condition at the idling temperature.

1 After the initial buffer baseline scan is complete and the instrument's temperature is moving down toward the starting temperature (approximately 10–15°C higher than the scan's lower temp), depressurize the instrument and remove the pressure handle.

2 At this time, load the degassed sample into the sample cell while the calorimeter is still moving down. The reference cell does not need to be reloaded.

3 As you load the sample in the calorimeter, the microwatt reading goes off scale. Do not be alarmed; it should return to a normal setting before it reaches the 0°C equilibrium point.

4 After the 10 minute equilibration, the calorimeter scans up, and when finished, will move the temperature down to the idle temperature, as before. When the scan is finished, the calorimeter idles at the programmed idle temperature (e.g., 25°C).