KRYO-FLEX Low Temperature Device (for D8/PLATFORM) User's Manual

Purpose
These instructions cover use of the KRYO-FLEX Low Temperature Device software, which controls the low temperature device and dewar that cool the sample for various applications.

NOTE: This procedure assumes that the KRYO-FLEX software (which may be a standalone program or part of the PROTEUM program suite) has been installed on your system.

Responsibilities
All procedures are to be performed by general users, site administrators, and trained Bruker AXS personnel.

Disclaimer
All configurations and specifications are subject to change without notice.

Referenced Literature
• none

1 Features

Before using the program, you will want to be familiar with the main window (Figure 1) and its features. This section explains those features.

Temperature Units
The Temperature Units subwindow allows you to select the temperature units that you wish to use. Two unit options are available: Kelvins (K) and Celcius (C) When setting the units, click on the circle next to your desired option (K or C).
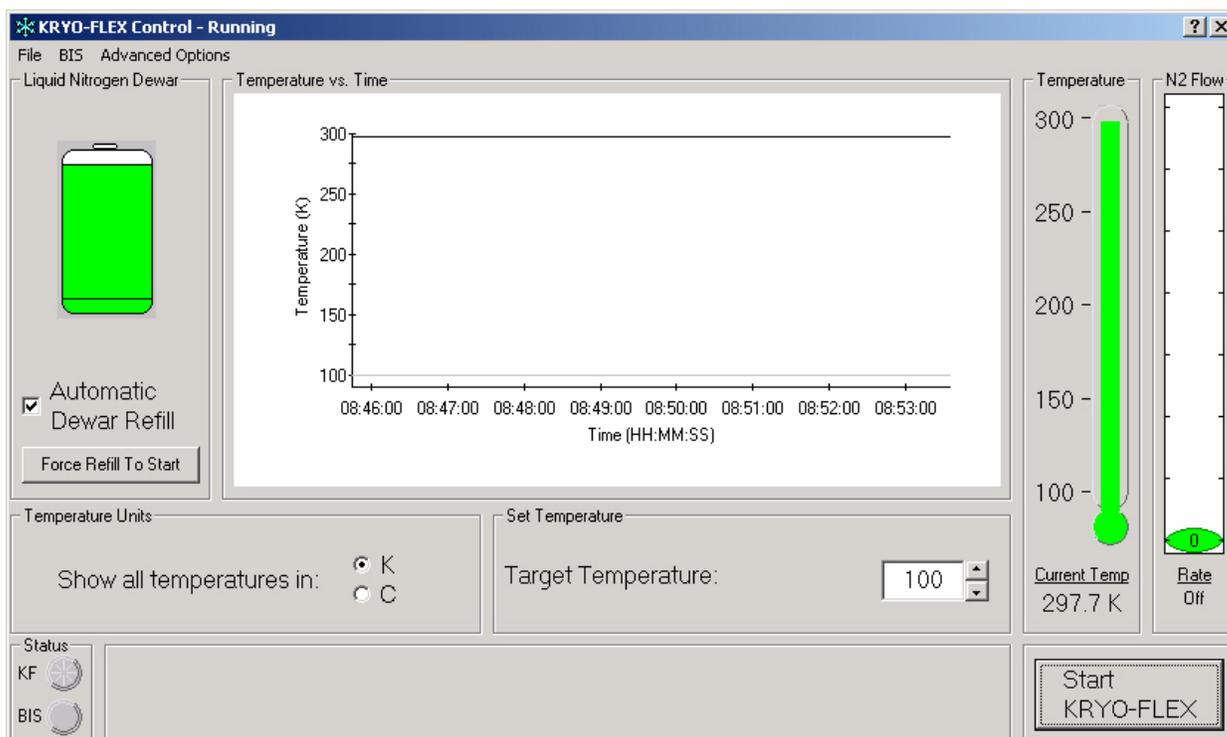

Figure 1. KRYO-FLEX Control-Running window

Note that the change will immediately affect all temperatures in the main window.

Current Temperature
The current temperature of the sample displays both as a thermometer (that indicates the maximum and minimum range of the temperature) and as text below the thermometer, with the temperature displaying in those units you have chosen.

A flashing thermometer indicates an error related to temperature. A message in the text subwindow by the Status lamps may explain the error.

Set Temperature
The Set Temperature subwindow is where you set a target temperature, which is the temperature you will want the system to cool to, stabilize at, and maintain throughout the experiment once you start KRYO-FLEX. Here you simply enter a target temperature in the provided field or scroll to a desired temperature using the arrows beside the field. A range of 90° to 300°K or -183.5° to 26.5°C is allowed by the system. You will be warned if you enter values outside this range or if you accidentally enter a nonnumeric value.

Temperature vs. Time
The current temperature also gets recorded in the Temperature vs. Time graph (center subwindow) to show you what KRYO-FLEX is doing over a period of time.

During the cooling operation, a dark line forms (see Figure 1 example near 300° mark) to mark the exact temperatures at different points along the timeline. Two light lines form (see Figure 1 example near 100° mark—lines are so close, they appear as one) to indicate the acceptable range of temperatures for the current target temperature (the range being target temperature ±0.1). These lines appear on the graph so that you can visually verify the temperature stability. During non-cooling, temperatures will rise to room temperature, and these will record in the graph for the extent of that period.

Automatic and manual zoom modes allow you to view the graph to monitor operation or check particular information.

The graph automatically zooms out (displaying the entire range of temperatures—cooling and non-cooling) when the temperature is not stable and zooms in (displaying the temperature range acceptable for stable operation) when the temperature

has stabilized at the target temperature. This auto-zoom feature allows you to readily see the current temperature in relation to previous temperatures for stability monitoring (that is, to see the temperature's likely rate of change).

You may also manually zoom in on or out of the graph and see temperatures over a period of your choosing. To zoom in on a specific period, left-click drag across the temperature points you wish to magnify. Upon releasing the mouse, your selected area will enlarge and fill the subwindow. This feature is recursive, meaning that you can select a time range within a time range (further zoom) to narrow the specific range shown. When you want to return to the entire time range recorded in the chart, right-click.

N2 Flow
The N2 (nitrogen) flow status displays as a scale in the N2 Flow subwindow, and as text below the indicator.

The KRYO-FLEX control is default set with a nitrogen flow rate of 5 liters per minute. However, when first cooling, the KRYO-FLEX control may switch to a Turbo Mode that uses a higher flow rate of 7.5 liters per minute to speed the cooling process.

Status Lamps
In the Status subwindow, two status lamps indicate KRYO-FLEX operation.

The KF lamp indicates specific KRYO-FLEX control status as follows:

- A lamp not lit indicates that the control is not actively cooling.

- A yellow lamp indicates that the control is cooling but the temperature has not stabilized. A message appears next to the lamp that explains the status and states the target temperature.

- A green lamp indicates that the control has stabilized at the target temperature. A message appears next to the lamp that explains the status. Stabilization occurs when 90% of the observed temperatures fall within the "acceptable range" (±0.1) over a period of 3 minutes.

- A red lamp indicates that a serious error has occurred and that the control is no longer cooling due to that error. A message appears next to the lamp that explains the status.

The BIS lamp shows if the KRYO-FLEX control is connected to the Bruker Instrument Service. If the lamp lights green, the control is connected. If the lamp is not lit, the control is not connected.

Liquid Nitrogen Dewar
The Liquid Nitrogen Dewar subwindow shows you the level of liquid nitrogen in the dewar and provides two options for refilling the dewar. A dewar graphic shows three states at which the nitrogen supply may exist in the dewar: full, within operational range, and empty. These states are controlled by two level sensors in the dewar, which detect the amount of liquid nitrogen in the dewar. The first (full-level sensor) senses when the dewar is full. The second (low-level sensor) senses when the dewar is almost empty. Figure 2 shows the different stages.

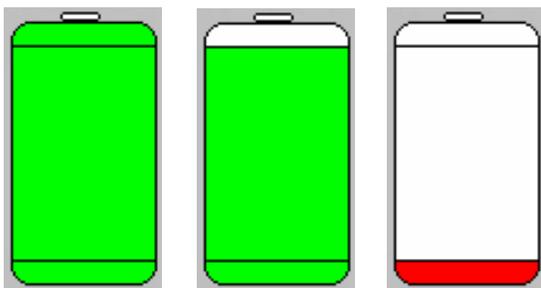| Full	Within Range	Empty
Full dewar, indicated by fully green graphic	Partially depleted dewar, indicated by partially green graphic	Empty dewar, indicated by white graphic with low level in red to stress refill urgency

Figure 2. Stages of liquid nitrogen level in dewar

A flashing dewar indicates an error related to liquid nitrogen. A message in the text subwindow by the Status lamps may explain the error.

To ensure smooth operation and prevent damage to your sample (in the event that the liquid nitrogen would run out), you can have the KRYO-FLEX system automatically refill the dewar when the liquid nitrogen level reaches the low-level sensor (if a refill tank with sufficient liquid nitrogen is connected to the dewar). To enable the refill feature, simply check the Auto Dewar Refill box in the Liquid Nitrogen Dewar subwindow.

When the automatic refill option is enabled, you can also force the refill to start and stop by pressing the Force Refill to Start button

(button text changes between Start and Stop when toggled).

Start/Stop KRYO-FLEX
You press the Start KRYO-FLEX button (bottom right corner of the main window) when you are ready to cool a sample. (Button text changes to Stop KRYO-FLEX.) When you are done collecting data for a sample and no longer need to cool it, press the Stop KRYO-FLEX button to stop flow of liquid nitrogen. Button text will change back to Start KRYO-FLEX.

2 Startup

With the KRYO-FLEX system set up as explained in the KRYO-FLEX Installation Manual, you should be able to run the KRYO-FLEX control software to control the hardware instead of having to manually control the hardware.

1. Double click the KRYO-FLEX icon (snowflake in standalone program, thermometer in the PROTEUM program) to start the KRYO-FLEX program. The KRYO-FLEX Control-Running window appears (recall Figure 1).

3 Operation

3.1 Basic Use

To prepare the system for basic use:

1. In the Temperature Units subwindow, select the temperature unit (K=Kelvin or C=Celsius) that you wish the KRYO-FLEX control to use. Note that the change will affect all temperatures in the main window.

2. Set (or change) the target temperature by entering a new value in the Target Temperature field of the Set Temperature subwindow.

3. Press the Start KRYO-FLEX button to start cooling.

NOTE: Press the Stop KRYO-FLEX button when you want to stop cooling.

3.2   Create a Log File

1. Click File > Log File Settings to have the KRYO-FLEX control create a log file of the recorded temperatures (Figure 3).
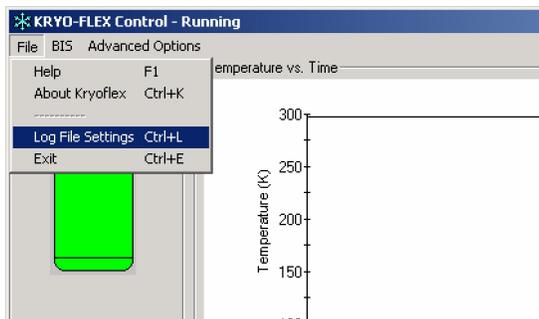

Figure 3. KRYO-FLEX Control-Running window
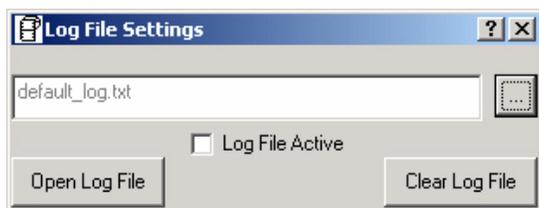
The Log File Settings input panel appears (Figure 4).

Figure 4. Log File Settings panel

2. Click on the browse button (…) to select a logfile.

A standard Windows browse window appears to let you open a previous logfile or create a new one (Figure 5).

Figure 5. Browse window

3. Highlight a current logfile or enter a new name in the File name field. Then click Open.

The Log File Settings panel now shows your selected (or new) filename (Figure 6).

Figure 6. Log File Settings input panel

4. Check the Log File Active box to tell the KRYO-FLEX control to activate the log file and record the temperatures into the log file.

5. Click File > Close (or the [x] box) to close the panel and return to the Control window.

If, at some point, you wish to view or edit the logfile, simply return to this panel and press the Open Logfile button. The notepad appears and displays your logfile. Or if you find that you are finished with the logfile at some point, return to this window and press the Clear Log File button to delete the file.

3.3   Connect to BIS

Connected to the Bruker Instrument Service (BIS), the KRYO-FLEX control can communicate the current temperature to BIS and other programs (such as PROTEUM) that also communicate with the Bruker Instrument Service. This connection lets the programs record the temperature to which a crystal was cooled each time a frame from that crystal is collected.

To connect to BIS:

1. Click on BIS > Connect to BIS.

The BIS Socket Connection window appears (Figure 7) to request a host name. A host name is the network name or the IP address of the computer to which the Bruker Instrument Service is connected.

Usually, the KRYO-FLEX control will be run on the same computer on which BIS is running because BIS controls the frame-collecting hardware and the KRYO-FLEX control runs the KRYO-FLEX low-temperature device. The KRYO-FLEX control assumes this arrangement and, by default, sets the host to an IP address that is universally accepted as a local connection. You may, however, enter any network name or IP Address that you wish.

Figure 7. BIS Socket Connection window

2. At the Host field, accept the default IP address, enter the IP address for the Bruker Instrument Service, or enter the host name you desire.

   If you need to set the KRYO-FLEX control back to the default IP Address, click the Set To Local button.

3. Click the Connect to BIS button.

   If you have entered a valid host name, the KRYO-FLEX control will connect to BIS and you can close the BIS Socket Connection window.

   To show that the KRYO-FLEX control is connected to BIS, the BIS lamp in the lower left corner of the KRYO-FLEX Control-Running window turns green.

3.4 Obtain Information from System Monitor

While the main window displays commonly needed information, the control stores more information in the system monitor (should you need to access those details).

1. Click on Advanced Options > System Monitor (Figure 8).
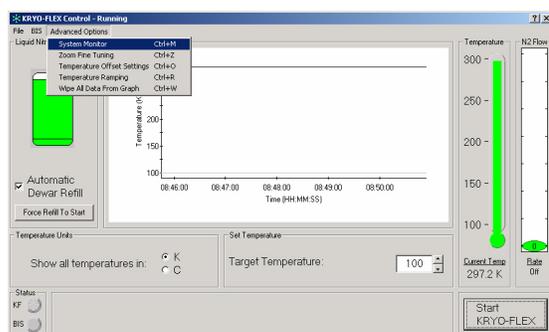
Figure 8. Advanced Options menu

   The System Monitor window appears, giving you specific details for both the Evaporator and the Nozzle heaters. It also gives you information on the dewar, the nitrogen flow, and the date and time (Figure 9).
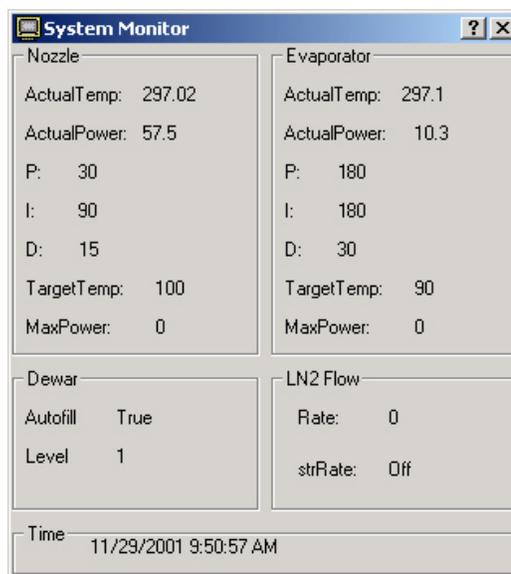

Figure 9. System Monitor window

3.5 Use of Zoom Fine Tuning

There are times when you may wish for finer control of the scale of zoom that the KRYO-FLEX control uses when the temperature has stabilized. Conversely, you may wish to zoom in even when the temperature is not stabilized, or you may wish to zoom out when it is stabilized. Do so as follows:

1. Click on Advanced Options > Zoom Fine Tuning.

   The Temperature vs. Time graph shrinks slightly, and the Zoom Fine Tuning tools show up in the new space created (Figure 10).
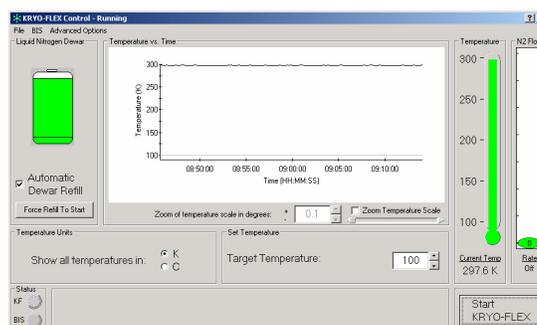

Figure 10. Zoom Fine Tuning tools appear under graph

2. Check (or uncheck) the Zoom Temperature Scale box of the new tools to turn on (or off) the zoom.

   When the Zoom Temperature Scale box is checked, the Temperature vs. Time graph will be zoomed in on the target temperature according to the scale settings.

3. Enter a scale value in the Zoom of temperature scale in degrees field (Figure 11) or adjust the slider bar below the heading Zoom Temperature Scale.
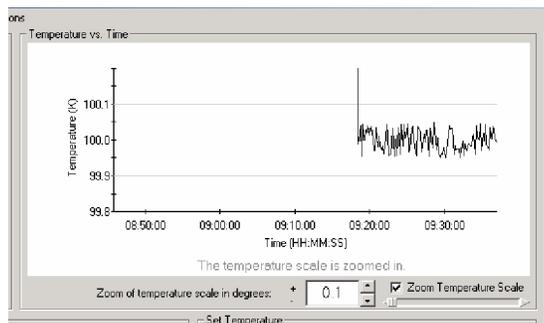

Figure 11. Zoom of temperature scale in degrees

To deactivate the Zoom Fine Tuning tools, click Advanced Options > Zoom Fine Tuning again to uncheck it.

3.6   Set Temperature Offset

The KRYO-FLEX control assumes that the temperature of the nitrogen stream (dispensed and monitored slightly above the sample) is the temperature of the crystal. This assumption may or may not be accurate. Sometimes the nitrogen stream can warm up between the nozzle and the crystal, if the nozzle is far away from the crystal. If you know how much the nitrogen stream warms (by taking an infrared reading at the crystal and calculating the difference between it and the target temperature), you can enter this value to set the temperature offset, as follows:

1. Click the Advanced Options > Temperature Offset Setting. The Temperature Offset input panel appears (Figure 12).

Figure 12. Temperature Offset panel

2. Check the Use an offset of: box to tell KRYO-FLEX to start using an offset value.

3. Enter the offset value (difference between infrared reading and target temperature) in the field to the right of the box.

4. Close the panel [x] and KRYO-FLEX will begin using the offset.

3.7   Temperature Ramping

Normally KRYO-FLEX cools down rapidly and may overshoot your target temperature. This rapid change is suitable for most applications, but could damage a crystal. If your application requires a more controlled temperature reduction, you may want to use temperature ramping. This feature works with a start temperature, a scale temperature (amount by which to slowly reduce), and an end temperature. When this feature is set, the KRYO-FLEX control will cool down to the start temperature and stabilize. Then it will cool down in incremental stages by the scale that you set, briefly stabilizing at each stage, until it finally reaches the end temperature that you set. Thus, if you set a start temperature of 200°K, a scale of 10°K, and an end temperature of 100°K, the control will cool to 200°K, then cool down 10°K at a time in stages until it reaches 100°K.

To set temperature ramping:

1. Click Advanced Options > Temperature Ramping to activate the temperature ramping.

   Temperature ramping controls will appear in the Set Temperature field (Figure 13).
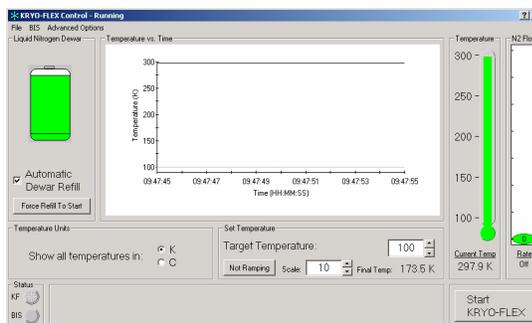

Figure 13. Temperature ramping controls

2. Enter a start temperature in the Target Temperature field, as you normally would.

3. Click on the Not Ramping button to enter settings and start ramping. (Click it later to stop ramping.)

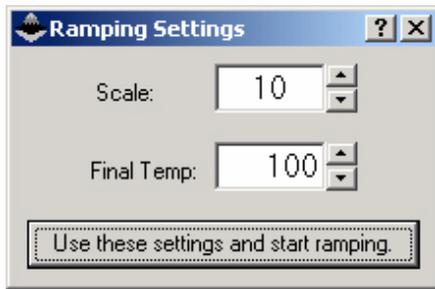   The Ramping Settings input panel appears (Figure 14).

Figure 14. Ramping Settings panel

4. Enter in the Scale field the amount by which you will want the temperature to incrementally fall.

5. Enter an end temperature value in the Final Temp field.

6. Click the Use these settings and start ramping button to activate the KRYO-FLEX control to use your settings.

    The Ramping Settings panel will close and the KRYO-FLEX control will start ramping.

3.8   Wipe All Data From Graph

The Temperature vs. Time graph normally holds all data since the software was started. If you run the software for prolonged periods, the graph can consume a large amount of system memory, which may make the software behave abnormally. To free this memory, you may want to periodically clear data from the graph.

To clear data from the graph, click Advanced Options > Wipe All Data From Graph. The data will then be cleared.