Technical Note

Mercury Support
Oxford Instruments
tel: +44 (0)1865 393311
fax: +44 (0)1865 393333
email: helpdesk.nanoscience@oxinst.com
www. mymercurysupport.com

Using a Mercury iTC with thermocouples

Abstract and content description

This technical note describes how to make accurate and reliable temperature measurements using an Oxford Instruments Mercury iTC. There is also an important note about heater interlocks when using cryogenically referenced thermocouples. This document should be read in conjunction with the latest version of the Mercury iTC manual.

Date: 21/3/2012.
Author: Rod Bateman.

Thermocouple principles (background)

A thermocouple consists of two parallel conductors of dissimilar materials, usually alloys, joined at one end, possibly both ends but with a break-out for voltage measurement. One junction is placed on the sample of unknown temperature, the other at some reference temperature, so that along both conductors there is a temperature gradient, ΔT. Electrons at the hot end of each conductor will have, on average, larger kinetic energies than electrons at the cold end, giving rise to an average diffusion of electrons to the cold end. This results in a lower electron density in the hot end forming a potential gradient in the conductor, termed the thermoelectric emf $\Theta(T)$. The potential gradient $\Theta(T)$ opposes the net drift of electrons due to the temperature gradient, ΔT. An equilibrium state is reached between the number of "hot" electrons diffusing toward the cold end driven by ΔT, and the number of "cold" electrons driven to the hot end due to $\Theta(T)$. Hence, in the steady-state, $\Theta(T)$ is dependent on ΔT.

The precise form of $\Theta(T)$ is determined by scattering of the conduction electrons by the lattice, which is in turn energy-dependent. Therefore, the shape of the Fermi surface of the metal or alloy, and the conduction electron mean-free-path, λ, are significant factors in determining the detail of $\Theta(T)$. As the detailed electron energy versus scattering relation can be very different from one material to another, this is the reason thermocouples are useable as thermometers and is termed the Seebeck effect. This effect is a *temperature gradient* phenomenon rather than a *junction* phenomenon. That is to say, an identical temperature difference in different alloys is required to observe the effect. What is necessary is that each pair of ends of the different metals are at the same temperature.

The property used to define this effect for some material, A, is the thermopower $S(T)$ where

$$S_A(T) = \frac{d\Theta_A(T)}{dT}\,. \qquad (1.1)$$

So the thermoelectric emf for a typical thermocouple constructed of a pair of conductors A and B is a unique function of the cold junction temperature, T_C, and the hot junction temperature, T_H

$$\Theta_{AB}(T_C \to T_H) = \int_{T_C}^{T_H} \left[S_A(T) - S_B(T) \right] dT\,. \qquad (1.2)$$

At high-temperatures, $T > \theta_D$, where θ_D is the Debye temperature for the material, for pure metals and alloys over large ranges of temperature, $S_A(T) \sim \alpha T$ where α is a temperature independent constant which is proportional to the sum of the rate of change of the Fermi surface area with energy, $k_B T$, where k_B is Boltzmann's constant, and the rate of change of λ with energy

$$\alpha = \frac{-\pi^2 k_B^2}{3e} \left(\frac{1}{\xi}\frac{\partial \xi}{\partial \varepsilon} + \frac{1}{\lambda}\frac{\partial \lambda}{\partial \varepsilon} \right)_{\varepsilon=\varepsilon_F} \qquad (1.3)$$

where ξ is the area of the Fermi surface. For simple metals with a spherical Fermi surface, $\partial\xi/\partial\varepsilon$ is always positive, as in general should $\partial\lambda/\partial\varepsilon$, therefore α should always be negative. This is indeed observed for group I metals (except Li) at high-temperatures. It is also observed, over a wider range of temperature for the group X transition metals, Ni, Pd and Pt. However, the largest values of thermopower, occur in the group VI transition elements, namely Cr, Mo and W, here α is positive, implying that

$\partial\xi/\partial\varepsilon$ must be negative. The fact that the group VI transition metals have the largest values for $S(T)$ corresponds to previous findings that the maximum values for $S(T)$ in binary alloys with Ni occurs at 10at% of Cr, Mo or W, each with 9.6 (*s*-band + *d*-band) electrons in the outermost incompletely filled bands.

Deviations from this ideal behaviour are introduced by inhomogeneities in the conductors contributing small additional thermopowers which have position dependent effects. That is, an inhomogeneity in a hot region of the thermocouple will generate a greater thermopower contribution than if it were in a cold region. The resulting fluctuations in $\Theta(T)$ can make it difficult to distinguish between genuine temperature transients in the measuring environment and the effects of inhomogeneities within the thermocouple.

At intermediate to low-temperatures, $T < \theta_D$, for pure metals and alloys, there is an additional "phonon-drag" term in α. This arises as $\lambda_{ph\text{-}ph}$ becomes greater than $\lambda_{ph\text{-}el}$, the lattice no longer appears in equilibrium in the electron frame of reference, thus the diffusion of the electron gas is now subject to changes by the phonon-electron interaction, which in general impedes the electron gas diffusion. At the low-temperature end of the scale, the phonon-drag term is proportional to the lattice specific heat, thus it disappears as T^3. In the intermediate to low-temperature region, $S(T)$ tends to be small, and has an even greater dependence on inhomogeneities. Indeed if an inhomogeneity exists near the room temperature region of the thermocouple, it can generate a thermopower much larger than the low-temperature signal thermopower, causing large fluctuations in $\Theta(T)$. Consequently resistance thermometry is now preferred to thermocouples for low-temperature work.

Substituting dilute magnetic alloys employing the Kondo effect can achieve relatively large negative thermopowers at low-temperatures. But for magnetic-field work their response is strongly alloy concentration critical. For the magnetic-field environment the type-E Ni-Cr-alloy/Cu-Ni-alloy thermocouple is recommended, but this has a small thermoelectric power, $S(4.2\text{K}) \sim 3\mu\text{V/K}$ rendering it an insensitive low-temperature thermometer. In general, as the thermopower depends on $\partial\xi/\partial\varepsilon$ and $\partial\lambda/\partial\varepsilon$ which have magnetic-field dependence, the form of $\Theta(T)$ in a magnetic-field is always difficult to interpret, thus thermocouples are not ideal thermometers for use in magnetic-fields.

Mercury measurement circuit in thermocouple mode

The sense voltage from the thermocouple is low-pass filtered, then buffered against a high stability reference voltage using low-noise, low-drift, ultra high precision amplifiers (MAX4238). Then the signal is filtered again and passed to a high-resolution 24-bit ADC (AD7192) and measured in differential mode with chop enabled. The sensor measurement circuit is laid out using 6 pcb layers including ground planes and guard tracks around all the sensitive signal tracks. In addition screening cans are fitted to ensure low-noise measurements. Steps have been taken in the layout, component selection and circuit design to minimise errors due to pick-up or current leakage.

The “Calibrate” button on the GUI “Temperature Sensor Details” (Figure 3) screen can be used to calibrate the ADC’s internal gains and offsets against the precision voltage source and on-board 0.01% precision resistors.

Thermocouple connection

To improve the stability of the measurement, it is useful to bias the thermocouple above the ground plane of the iTC. To do this connect pin3 (Sense –ve) of the 9-way D-type connector to pins 4 and 5 of the 9-way D-type connector so that these pins are common (Figure 1). Biasing the thermocouple in this way will not cause error currents to flow in the thermocouple even if the equipotential isothermal junction is grounded as the iTC measurement circuit is fully isolated.

To reduce noise and increase accuracy the connector should have a hood fitted which includes a cable gland to minimise any air current flowing around the thermocouple connection pins. For thermocouple devices it is preferable for this hood to be plastic rather than metal which allows the connection pins to closer match the iTC’s internal reference temperature (see below).

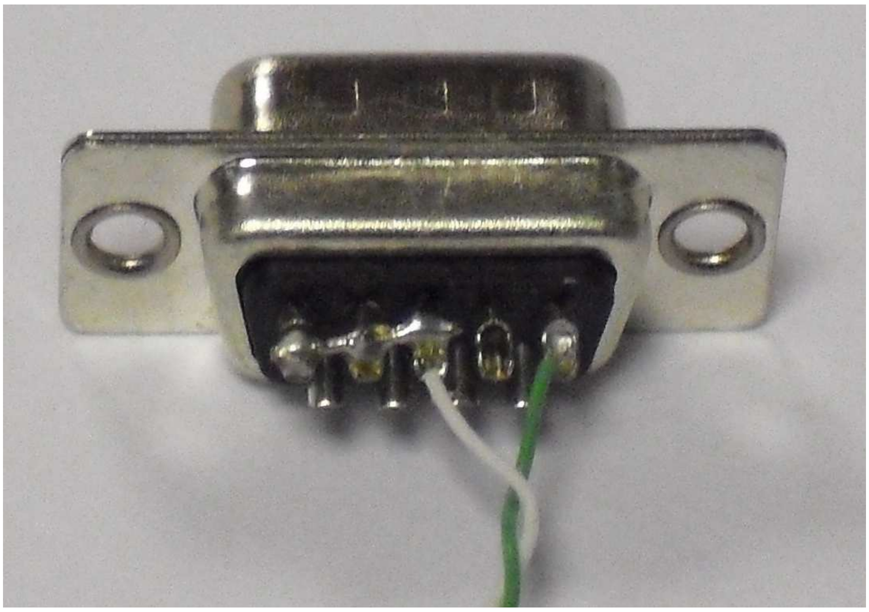
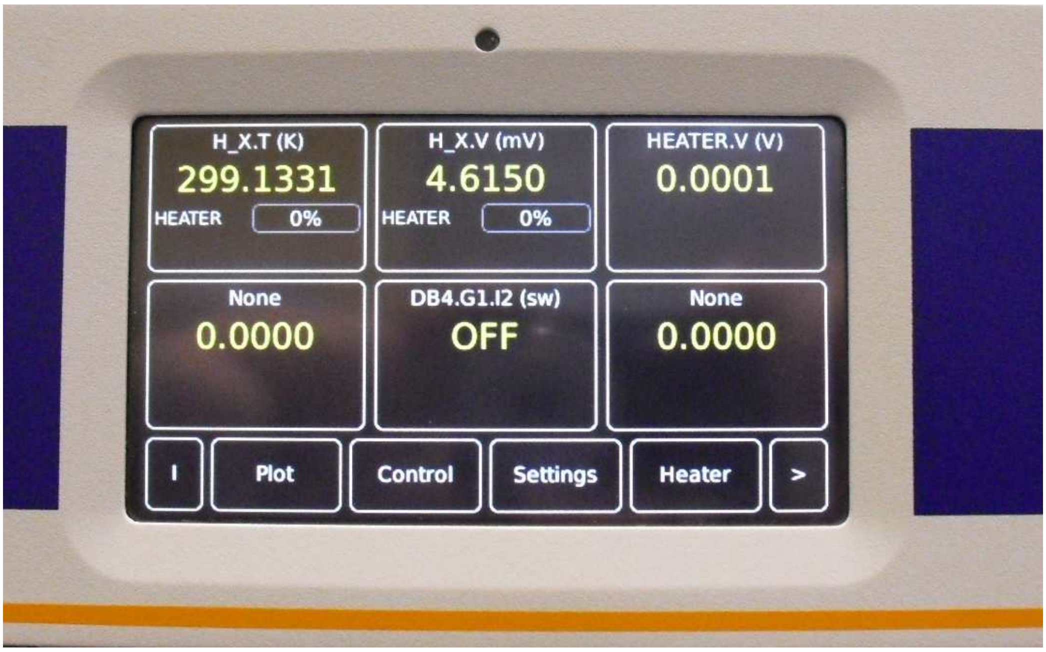


Figure 1. A 9-way D-type connector wired with a K-type thermocouple for use with a Mercury iTC. Note the link between pins 3, 4 and 5 used to bias the thermocouple above the ground plane for greater stability.

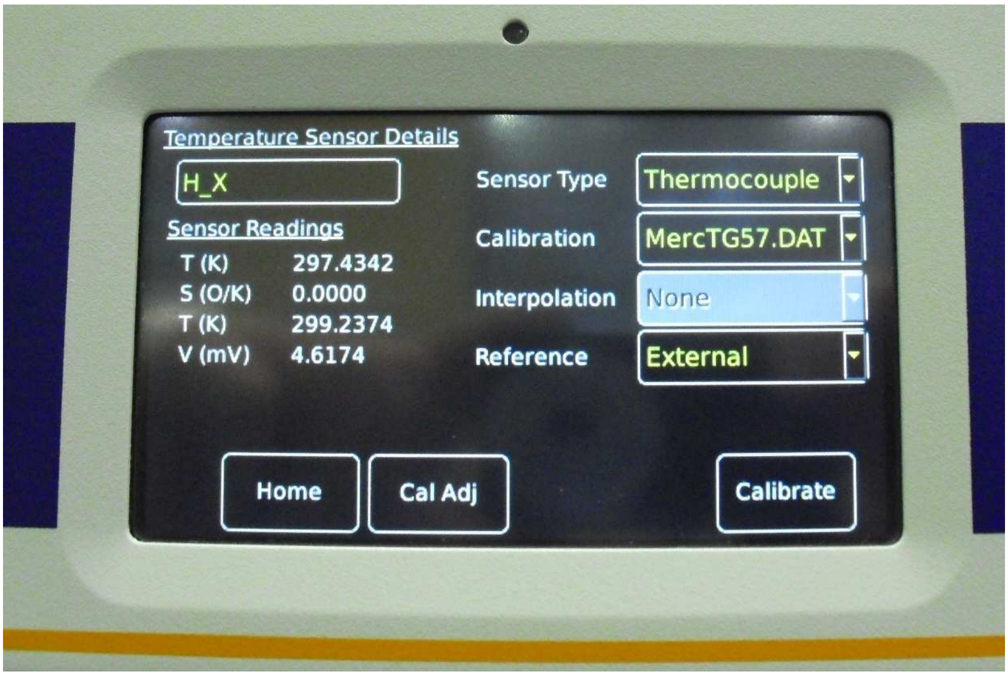
Configuring for thermocouples

For best results the iTC Cryosys (main application) firmware should be 1.0.8.17 (1.0.9 release) or later. First set up a home screen widget (Figure 2) to read the temperature device (see the mercury iTC manual for details). In the “Temperature Sensor Details” screen (Figure 3), set the “Sensor Type” to be “Thermocouple”. Set the “Calibration” to one of the Mercxxxx.dat files as these files have a higher point density than the previous ITC503 files. So, for example, use the MercTG57-2.dat if an AuFe-Chromel thermocouple is being used with a LN_2 reference junction (see below). The thermocouple does not need any excitation as it generates its own signal.



H_X.T (K) 299.1331 HEATER 0%
H_X.V (mV) 4.6150 HEATER 0%
HEATER.V (V) 0.0001
None 0.0000
DB4.G1.I2 (sw) OFF
None 0.0000

Figure 2. Mercury iTC Home screen. The top-left widget has been configured to show sample heat exchanger (H_X) temperature measured by a AuFe-Chromel (0.07% Fe) thermocouple using a LN_2 reference. The top-centre widget has been configured to show the thermocouple voltage as measured. The top-right widget has been configured to show the heater voltage. The bottom-centre widget has been configured to show the status of the heater interlock.



Temperature Sensor Details

H_X

Sensor Readings

T (K) 297.4342
S (O/K) 0.0000
T (K) 299.2374
V (mV) 4.6174

Sensor Type: Thermocouple
Calibration: MercTG57.DAT
Interpolation: None
Reference: External

Figure 3. Mercury iTC "Temperature Sensor Details" screen (Cryosys v 1.0.8.17). The "Reference" has been set to "External" as a LN_2 bath reference is being used and the internal ADT7310 is not (see below). Under "Sensor Readings" the top "T(K)" value is the internal reference temperature which is always shown for thermocouples.

Reference Junction Compensation

The Mercury iTC configuration options allows for the use of an internal or external reference junction to be used. Each Mercury iTC temperature sensor circuit has a built in temperature reference chip (ADT7310) adjacent to the sensor 9-way D-type connector. The real-time temperature reported by this chip is converted to a voltage by an inverse function of the thermocouple calibration file in use, and this voltage is added to the thermocouple voltage measured at the 9-way D-type connector. This compensated voltage is then converted to temperature via the selected calibration file.

The accuracy of using the internal reference is limited by 3 factors: -

1. The ADT7310 is specified with an accuracy of 0.5˚C.
2. Pins 1 and 3 in the 9-way D-type may differ in temperature by 0.5˚C or more.
3. The Mercury iTC assumes the internal reference chip is 2.0˚C warmer than pins 1 and 3 in the 9-way D-type. From tests this is generally correct within the accuracy of the ADT7310 chip but under some circumstances (e.g. multiple heater cards installed and running at a high output) there maybe an error in this assumption.

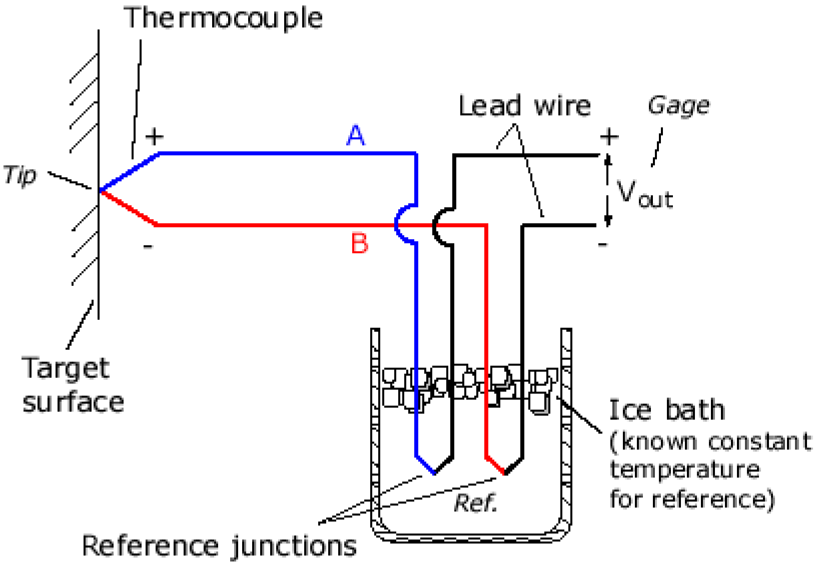


Figure 4. An example of an externally referenced thermocouple. In this case and ice bath is used to maintain the temperature of the reference junctions. A suitable calibration file with a zero volt ice point must be used (see Figure 5).

Consequently, it is recommended that external referenced thermocouples are used for high accuracy thermometry (Figure 4). In particular, for accurate cryogenic thermometry using thermocouples, cryogenic reference junctions should be used, the most common example being a liquid Nitrogen(LN_2) bath. A more convenient method of providing the compensation voltage might be from a high stability solid-state electronic source if one is available.

It's more important to use low-temperature reference junctions for cryogenic applications because the thermocouple sensitivity drops off at low temperatures (Figure 5) which has the effect of amplifying any errors that might be present in the room temperature reference. This effect can be reduced by adding dilute magnetic alloys, as discussed above, so in Oxford cryostats using thermocouples a AuFe-Chromel (0.07% Fe) thermocouple is typically used with a LN_2 bath for the reference junctions (Figure 6).

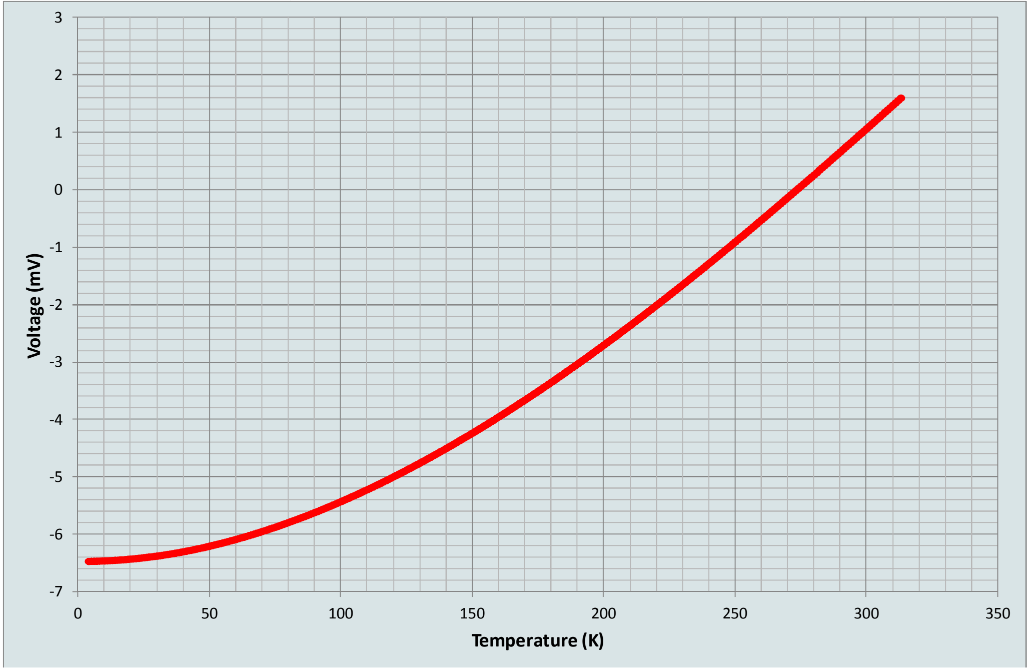


Figure 5. An example dataset for a thermocouple. This plot is for a type-K (Chromel-Alumel) thermocouple showing the Seebeck voltage in millivolts and the temperature in degrees Kelvin. Note that this dataset has been offset for an ice point reference (zero signal volts corresponds to 273.15K) and how the sensitivity drops off at low-temperatures.

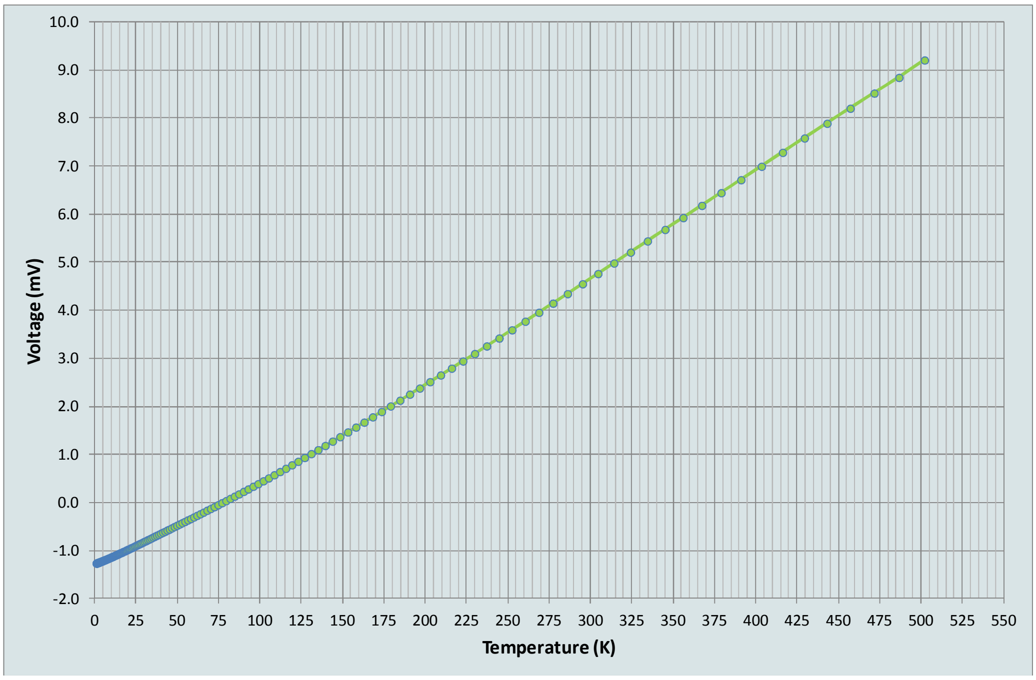


Figure 6. This plot is for a AuFe-Chromel thermocouple (K-type modified with 0.07% Fe added to the Au) showing the Seebeck voltage in millivolts and the temperature in degrees Kelvin. Note that this dataset has been offset for a LN_2 point reference (zero signal volts corresponds to 77.35K) and how the sensitivity does not drop off as much at low-temperatures. This is preferred for cryogenic applications.

Heater control Interlocks and reference compensation

If a thermocouple is being used to control a heater in a PID control loop and the thermocouple uses a cryogenic reference junction, it is important to consider what happens if the reference junction cold bath runs out of cryogen. If this occurs the reference junction temperature will be warming up. As described above, the thermocouple voltage will be a function of the temperature *difference* between the reference junction and the measurement junction. So as the reference junction warms up the measured voltage will decrease. As the Mercury iTC must assume the reference junction temperature is fixed, a decrease in measured voltage will be interpreted as a decrease in measuring junction temperature. If the thermocouple is controlling a heater at a temperature set-point, then a perceived reduction in measuring junction temperature will cause the heater output power to be increased. As the reference junction warms up further the system will keep applying more heat to the cryostat.

To prevent the system being over heated, the Oxford cryostats using cryogenically reference thermocouples use an interlock switch. This is a semiconductor device with a preset switching threshold which is inserted in the reference cryogen bath with the thermocouple reference junction. In the Mercury iTC an additional plug-and-play Auxiliary Card should be included in the system to operate this interlock switch. Input line 2 of the Auxiliary board is a digital signal which changes state depending on whether the voltage at the input pin has exceeded the defined threshold (typically 1.8V) which it will if the semiconductor device warms up above about 85K. The state of the I/O line (Figure 2 bottom-centre widget) is then used as an interlock for the heater. With this arrangement the heater is locked off if the LN_2 reference bath runs out, thus protecting the cryostat from being over-heated.