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# **PHITEC BTC 130**

## **Operating Manual Volume 1**

### **Hardware Details**

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## SAFETY NOTES

The PHI-TEC BTC is an apparatus that allows you to screen re-chargeable batteries for thermal runaway. It is intended for use in a ventilated area away from personnel. However, any system that examines exothermic behaviour has some inherent hazards. **It is the responsibility of the user to ensure that any risk is minimised and that all testing is performed safely. Moreover, it is the user's responsibility to carry out a full hazard identification and risk assessment of the system and its intended use.**

Note the heating oven can become very hot during testing. Before touching the system, use the software and mimic diagram to check that the temperatures have cooled to near ambient.

Following cooling, there may still be hazardous decomposition products present in which case the equipment may need to be suitably vented.

The system is not designed for testing explosives or taking samples to violent decomposition. The BTC containment vessel is not designed as a pressure vessel it is however strong and should a rapid decomposition occur that may develop pressure then a suitably rated relief valve is fitted to the unit.

It is possible that some projectiles and or hazardous fumes /gases may be developed in the event of battery decomposition. For this reason, the PHI-TEC BTC must only be used in a well-ventilated area away from personnel.

# 1. PHI-TEC BTC OVERVIEW

## 1.1 Introduction

The PHI-TEC BTC is an adiabatic calorimeter, which can be used for screening batteries and other samples and simulating the way in which exothermic self-heating and battery decomposition may occur. The sample sits at the heart of the calorimeter which is surrounded by metal guard heaters. The BTC guard heaters can track temperatures up to  $35^{\circ}\text{C min}^{-1}$  and can reach temperatures of up to  $\sim 450^{\circ}\text{C}$ . (Note however that some seal materials used in the BTC may degrade at such high temperatures and generally it is recommended that testing is restricted to temperatures of not more than  $350^{\circ}\text{C}$ .)

The guard heaters assembly is housed in a containment vessel. This containment vessel is not designed as a pressure vessel but rather to contain projectiles and fumes in the event of battery decomposition.

The BTC is designed for use in a well-ventilated area. Any exhausts e.g. from a vent valve, and potentially from the pressure relief valve should be piped to a safe location. Typically these are directed towards the rear of the apparatus and certainly not towards the user or other people in the laboratory. The external surfaces of the equipment may get hot to the touch when in operation and should be sited away from people and other apparatus particularly flammable materials. The equipment should be sited in a suitable location that allows for some manual handling of the equipment between tests. A good level of background lighting should be provided for the users safety and comfort.



**The PHI-TEC BTC130**

### **1.1.1 Range of Experiments**

The PHI-TEC BTC calorimeter can be used to perform a variety of experiments for the investigation of thermal stability of batteries. These include ramped screening experiments, heat-wait-search or heat soak operation and adiabatic tracking. The following data may be obtained :

- Exotherm onset temperatures.
- Determination of kinetic parameters for Arrhenius equation. (i.e. Activation energy, frequency factor, order of reaction).
- Enthalpy of runaway reaction.
- Investigation of the effects of external heat or cold sources

Further special tests can be performed, such as:

- Specific heat capacity determination
- Adiabatic charging/discharging (additional equipment required)
- Battery shorting (additional equipment required)
- Sub-ambient temperature testing (additional equipment required)
- Adiabatic testing of liquid chemistry (additional equipment required)

## **1.2 Summary of Design Features**

### **1.2.1 Equipment Size and space requirements**

#### Main BTC assembly

Nominal width: 600 mm

Nominal depth: 600 mm

Nominal height: 800 mm

Maximum Internal height: 200 mm

Internal diameter: 130 mm

Nominal Power rating: 10kW (depending on accessories)

The electronics and PSU are housed underneath the unit and are connected to the main unit via fitted cables. A 3m cable connects the electronics to the PC although longer connection cable can be supplied upon request.

Additional space should be allowed for optional devices such as a multi-temperature measuring device, programmable battery charging or programmable load devices, serial communications interface box (ATOP) etc.

### **1.2.2 Equipment power requirement**

The BTC runs of single phase mains power supply. E.g. 230-240 V. The number of mains sockets and current rating of the mains supply required will depend on whether optional devices are ordered with the system. (Typically a 30A mains supply will be more than adequate for the maximum load for most configurations). In reality the power consumption is a tiny fraction of this and most items can run off a multi-way extension strip. It is however recommended that each of the two BTC power supply units runs off its own mains socket rather than an extension strip. The system can run off a lower voltage rated (e.g.110V), single phase supply but the power will be reduced significantly and this may affect the maximum temperature and the maximum tracking rate achievable with the unit.

The base system will require mains sockets for the following.

1. PC base unit
2. PC Monitor
3. Electronics rack for the BTC
4. 1<sup>st</sup> Power Supply Unit (for BTC heaters)

Additional mains power sockets will be required for optional devices such as, ATOP box, a multi-temperature measuring device, battery charging devices etc.

### **1.2.3 Containment Vessel**

Operator safety is assured by carrying out all reactions in a strong steel containment vessel. Note that this is not a pressure vessel but is designed to retain fragments and fumes should a sample decompose.

### **1.2.4 Temperature Measurement**

In the standard configuration two fast-response, high temperature, 1mm diameter, inconel sheathed type K thermocouple measures the sample temperature. This is placed in direct contact with the sample and is normally secured with high temperature glass tape or securing wire. This thermocouple should be located such that it is not in direct contact with the sample heater.

Data acquisition is achieved via an A/D converter and amplifier that give a typical range of 0 to 500°C (with a resolution of 0.1°C). The heater temperatures are also measured by inconel sheathed 1mm diameter type K thermocouples.

### **1.2.5 Pressure Measurement**

Pressure is measured by an absolute transducer. The normal range for this transducer is 0 to 300bar, with an accuracy of up to  $\pm 0.15\%$ .

### **1.2.6 Adiabatic Temperature Control**

Zone (guard) heaters, which completely surround the sample, are used to provide adiabatic sample conditions. These are independently monitored and powered by a proportional-integral-differential (PID) controller to track the sample temperature.

### **1.2.7 Exotherm Detection: Sensitivity**

The instrument will detect exothermic activity down to self-heat rates of approximately 0.02-0.03°C min<sup>-1</sup>.

### **1.2.8 Exotherm Tracking Rate**

The guard heaters can track the sample temperature to rates of up to 35°C/min.

### **1.2.9 Data Acquisition**

Temperature and pressure data are logged on a computer. Relevant information is displayed on monitor during an experiment, either numerically or graphically. Data can be stored on disk at rates of up to 10 sets per second. The data save rate can be user defined to allow greater save rates at high self-heat rates. This allows detailed analysis of fast periods of an experiment, without creating excessively large data files.



### **1.2.10 Heater Control**

The design of the heating units and their temperature control is a key in the thermal performance of BTC. Independently controlled heaters are employed, the temperature of each being measured by a two type K, fast response thermocouples. Energy is supplied by a programmable power supply unit (PSU).

## 2. EQUIPMENT ASSEMBLY

This section describes the major hardware units of the PHI-TEC BTC Calorimeter and gives details of how to set up the hardware for running experiments.

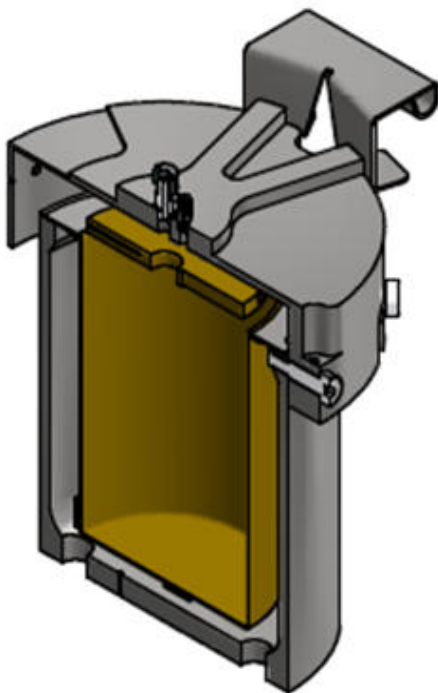
### 2.1. Main Unit

The lid of the vessel contains several ports/connections. These allow the connection of the instrumentation and feed lines to the cell:

- Pressure transducer
- Can thermocouple
- Ball valve
- Feed line to test cell

Further ports are located on the side of the lid for the top heater and thermocouple. Additional pipework e.g. to a solenoid valve or relief device can be connected to the main feed line.

A separate optional lid is available to which a automatic nail penetration unit can be mounted.



**BTC 130 section – with standard lid**



**BTC130 with Nail Penetration Option**

## 2.2 Heaters

Three main types of heaters are employed in the PHI-TEC BTC calorimeter, the sample power (can) heater, the sample calibration heater and the guard heaters.

### 2.2.1 Sample Power Heater

Function of this heater is to help to heat up the sample to the required temperature faster. This is a flexible resistance wire heater with high temperature braided mineral insulation that is wrapped tightly around the sample. This is connected via means of a termination block. The heater element can be fixed in place using high temperature glass tape or securing wire. Note alternative resistance heater elements may be used but these should have a resistance greater than 12 Ohms.

### 2.2.3 Sample Calibration Heater

This heater is used for MCp determination. It is a flat, Kapton laminated heater of about 1" square surface area. The heater should be sandwiched between two or more samples in order to eliminate heat loss. It can be fixed in place using Kapton tape. Voltage output of the MCp PSU is 9V.

### 2.2.4 Guard Heaters

The guard heaters completely surround the test cell. These are used to maintain an adiabatic environment for the sample by maintaining the surrounding temperature equal to that of the sample. The guard heaters comprise of separate heater elements for the top, side and bottom, each of which are independently controlled using a PID control algorithm. The heaters consist of two separate assemblies:

- **Top Heater Assembly:** This consists of a heater disc suspended from the lid assembly. Two cartridge heater elements are inserted into the top heater disc. One K-type thermocouple is used to provide temperature measurement.
- **Side & Bottom Heater Assembly:** Comprises of cylindrical anodised aluminium drum. The side heater consists of a tape heater wrapped around the sides of the drum. The bottom heater consists of two cartridge heaters. These elements are insulated from the main body using Superwool insulation. One Type K thermocouple is held against the drum for each element.

On closing the containment vessel, the two heater assemblies form together to produce a near closed cylindrical heating chamber. Superwool insulation is placed between the two parts to minimise heat losses.

## 2.3 Instrumentation

### 2.3.1 Pressure Transducer

- **An absolute pressure transducer:** to measure the pressure. The transducer used as standard has a range 0 - 300 bar and 0.15% accuracy.

### 2.3.2 Thermocouples

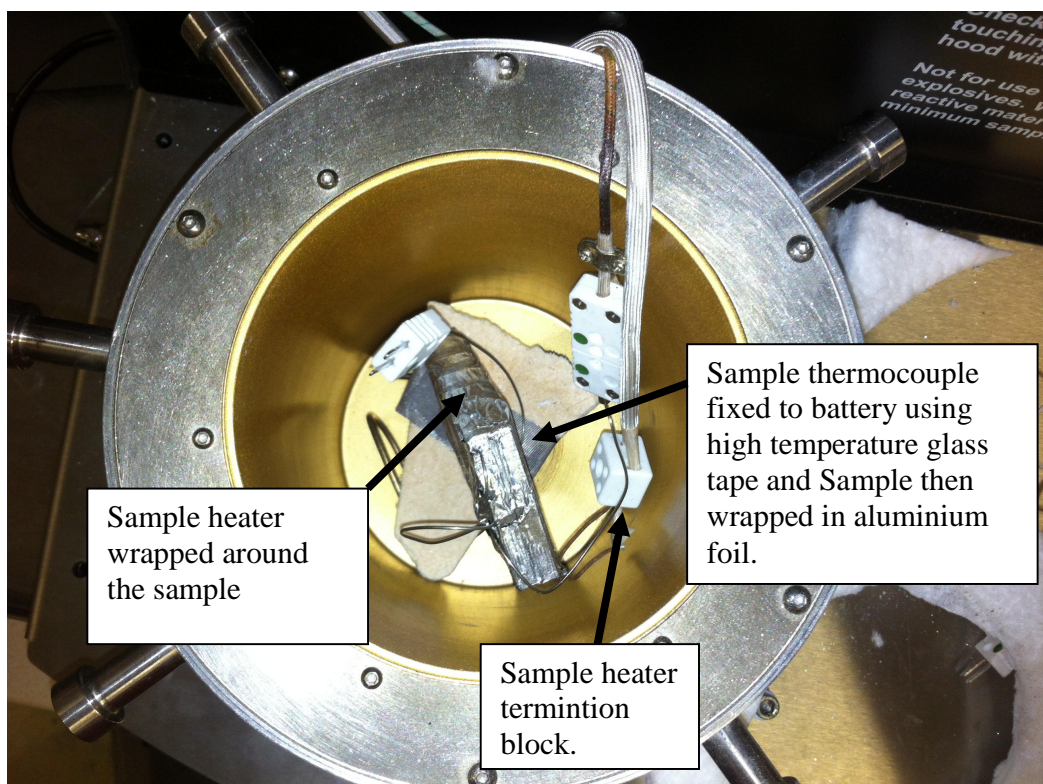
Eight Type K thermocouples are normally used. One thermocouple is used to measure the sample temperature. One thermocouple is used to measure the temperature of each of the guard heaters.

## 2.4 Setting up for a Test

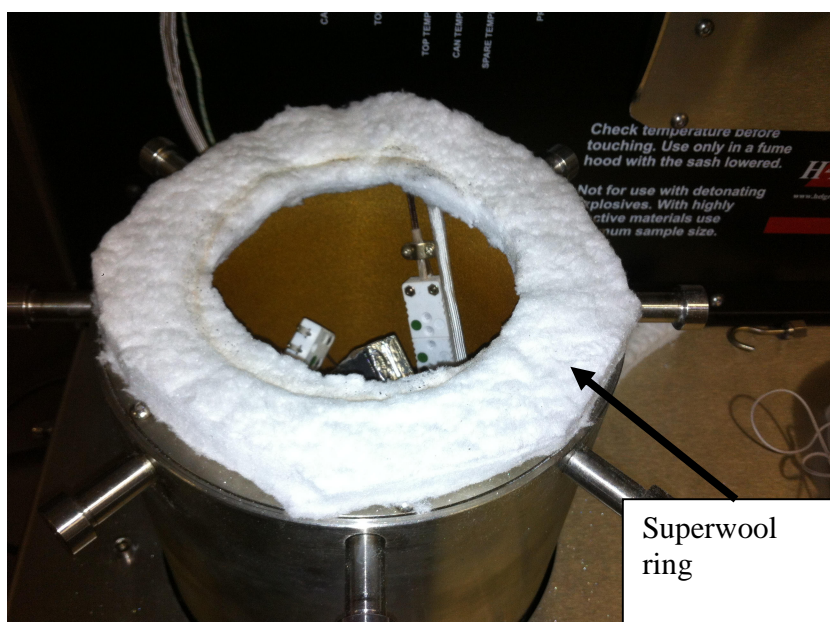
The equipment is supplied with the heaters installed. Should these need to be replaced the advice should be sought from HEL Ltd. A protective stainless steel drip tray can sit inside the side/bottom heater drum to collect any residues that may drip or leak from the sample. This can be removed/replaced to allow cleaning of this tray. The main PHI-TEC BTC unit should be opened up ready to receive the sample. A support bracket is provided for the lid to rest on while the apparatus is open. The heaters should not be powered at this stage and the unit should be cool.

### 2.4.1 Loading a Sample

Before placing the sample inside the calorimeter it is often preferable to tightly wrap the sample with the sample heater. This should be secured in place using high temperature glass tape or suitable fixing wire. Sufficient 'free end' lengths of the heater element should be allowed to connect to the sample heater termination block inside the calorimeter. The heater should be wrapped in such a manner that there is still sufficient space to affix the sample thermocouple without it being in direct contact with the sample heater. At this point one k-type thermocouple is attached to the sample. After the attachment of thermocouple and sample heater, sample must be carefully wrapped in an aluminium foil in order to minimise radiative heating of the sample by the guard heaters. A sticky aluminium foil is recommended. The sample is placed in the middle of the calorimeter. It should be held off the base of the calorimeter using an appropriate support so that it is not in direct contact with the guard heater. tripod laboratory stand or even something as simple as an upturned beaker may be adequate.



A superwool ring should then be placed on top of the oven before placing the lid down. This ring will reduce heat loss from the oven. The ring dimensions are 130mm(ID) x 190mm(OD)



### 2.4.2 Checklist for Test Set Up

Below is a checklist for the running of a typical experiment. This is useful to ensure that all preparations have been properly made, and avoid abortive experiments:

- (1) **Connect instrumentation:** Attach all of the instrumentation.
- (2) **Check guard heater connections:** Ensure that all of the guard heater power leads and thermocouple connections are properly made.
- (3) **Load sample** (see section above). Check the sample and guard thermocouple signals on the computer show acceptable values. Ensure that no wires/cables or seal ring will be trapped or damaged when closing the lid. Close and use the lid handle to lock the lid in place.
- (4) **Check power supply is on.** If necessary re-start the software to ensure the over-temperature trip is cancelled on the front of the PSU.
- (5) **Select, configure and start experimental plan**

## **2.5 ELECTRONICS UNIT**

The electronics unit provides the following basic functions:

- Amplification of pressure, temperature and other analogue signals
- Provides power supplies
- Controls all heaters, solenoid valves etc.
- Provides the link between sensors and the computer (via interface cards).

The electronics unit is powered from a single phase mains supply. The power lead is plugged into the front of the unit. The sensor and control cables (3m standard length) are also plugged into the front of this unit and connect the electronics to the PHI-TEC BTC main unit hardware.

## **2.5 POWER SUPPLY UNIT**

The power supply unit provides the power to the guard heater elements. It is powered from a single phase mains supply. As this requires a high current it is recommended that it is plugged directly into the mains supply socket and not via a multi-way extension strip or adapter. A communication cable connects the electronics to the power supply unit and power leads from the power supply are connected to the PHI-TEC BTC main unit hardware.

## **2.6 COMPUTER AND INTERFACE CARDS**

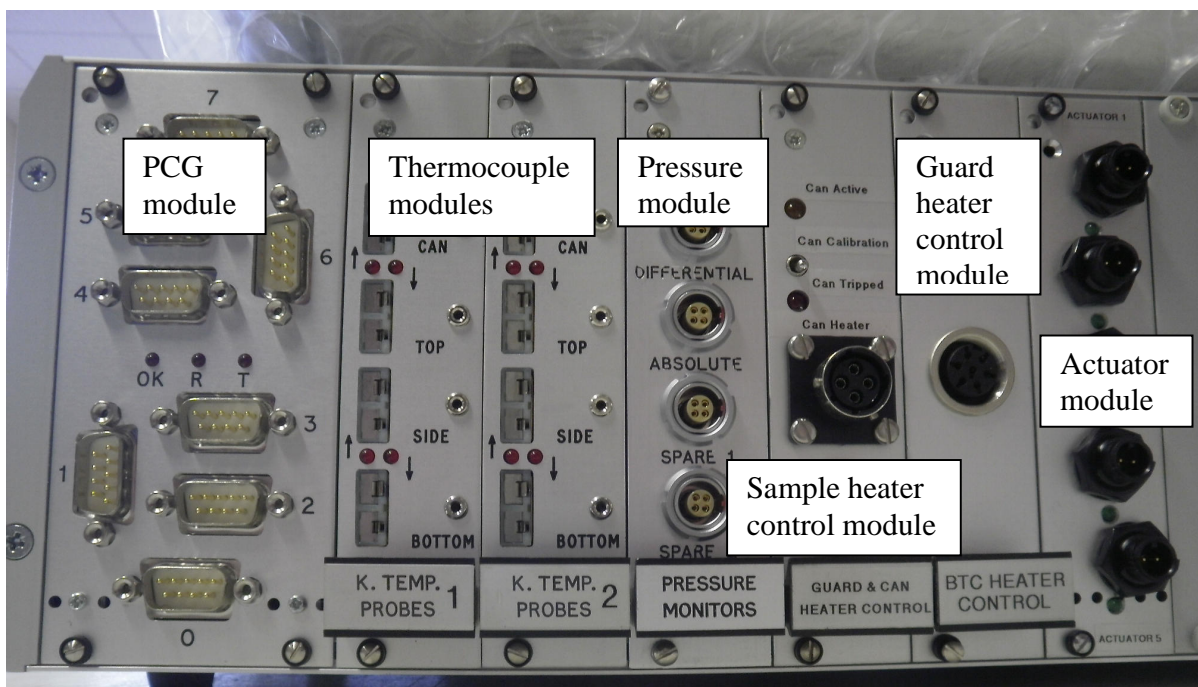
In the basic version of the equipment, one interface card, PCG2, is installed in one of the spare slots in the electronics rack. A connecting cable links the card to the PC. This card provides the means of communications between the hardware and the software. If a number of optional serial devices are supplied then the PCG2 card and these serial devices may be connected by a serial interface box (such as an ATOP) box which is in turn connected to the PC via an Ethernet cable through the LAN port on the PC.



### 3. ELECTRONICS

#### 3.1 ELECTRONICS RACK

The electronics is housed in a 42HP rack. It provides all the necessary signal conditioning for the transducers (e.g. temperature, pressure etc) and power for the motor drives solenoids and sample heater. Power for the guard heaters is provided from a separate power supply unit (PSU), this is controlled from the winISO software via the mains electronics rack. The following section describe the various modules used in the rack. The mains power input is to the right hand side on the front of the rack (not shown). This input is fused. The mains switch is lit when the rack is powered



Electronics rack-front view showing the modules

##### 3.1.1 PCG2 Interface Module

This interface card communicates between the computer (PC) and the rack. Normally the serial communication (PCG) cable is plugged into port 0 on this module.

##### 3.1.2 Thermocouple Modules

These two modules provides signal conditioning for up to eight type K thermocouples. Each card presents the interface with four absolute temperatures, selectable by jumpers on the board.



### **3.1.3 Pressure Module**

This module provides conditioning for four piezo-resistive strain gauge type pressure transducers. The output range and sensitivity depends on the range of the transducer used. The module can also accept signals from other strain gauges as required. Normally the pressure transducer will be plugged into the socket marked “absolute”.

### **3.1.4 Can Heater Control and Power Measurement Module**

This module provides the control signal to drive the calibration/sample heater. This uses a pulse width modulated 24V dc power supply. i.e. the mark space ratio of the switching signal determines the mean power supplied to the heater element. The maximum output current is approximately 2A depending upon element impedance (which must be  $\geq 12\Omega$ ), giving a potential output capacity of about 48 watts.

There is a front panel light to indicate whether the calibration heater is on or off and a toggle switch (spring biased off), which gives full power when pressed. This switch is used for calibration of the Calibration Heater Power Measurement module only.

This module also monitors the voltage and current supplied to the sample (calibration) heater element and outputs two analog signals, which are true r.m.s (Root Mean Square) averages of them. The voltage is measured using a four-wire technique to eliminate errors due to cable impedances.

### **3.1.5 Guard Heater Control and Power Measurement Module**

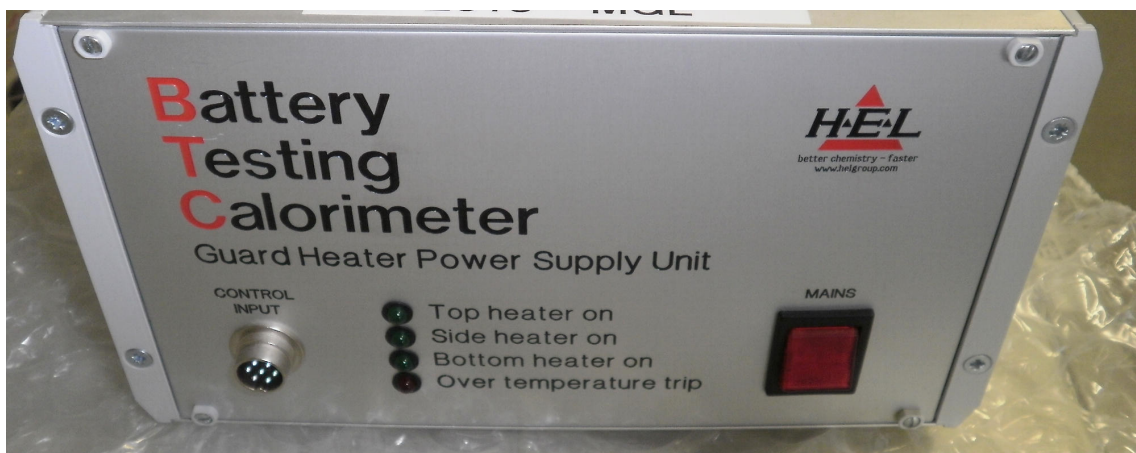
This module is used to allow for three 0-10V analogue control voltages and one digital output to be fed to the BTC Guard Heater Power Supply unit.

### **3.1.6 Actuator Module**

This module provides control over five uncommitted relays, which are connected to a 24V dc supply when activated. In the BTC actuators are used for gas cooling and vent solenoid valves. The actuator output sockets are located on the front panel.

### 3.2 POWER SUPPLY UNIT (PSU)

The unit supplies the power to the guard heater elements on the PHI-TEC BTC. The mains power lead supply is plugged into the back of the PSU as are the power leads out from the PSU to the PHI-TEC BTC hardware. The control signal lead from the electronics rack plugs into the front of the power supply unit (Control input socket). When power is being supplied to the PHI-TEC guard heaters LED's for the individual elements will light on the front of the unit. An over temperature trip indicator is also positioned on the front of the PSU. This will light should any of the temperature sensors become unplugged, they fail open circuit or if the temperature actually exceeds the hardware limits. When lit the power is turned off to all of the guard heaters.



Power Supply Unit Front view



Power Supply Unit Rear View

A 13A fuse is fitted to mains power inlet and 6.3A or 4A fuses are fitted to power outputs as indicated on the rear view of the PSU.

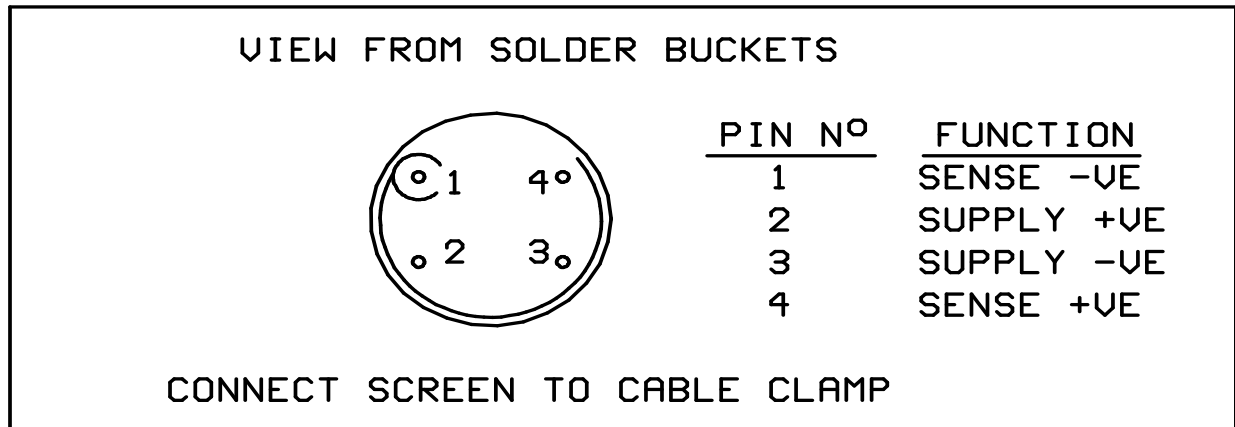
### 3.2 CONNECTORS & CABLES

#### 3.2.1 Thermocouple Temperature Sensors

Thermocouple temperature sensors for use with the electronics must be type K and must be fitted with miniature type K thermocouple plugs (pins).

#### 3.2.2 Pressure Transducers

Pressure transducers to be connected to the electronics require a 4 way Lemo 1B series plug fitting. Manufacturers code number FGA.1B.304.CLAD52. The pin connections are shown below:



#### 3.2.3 Can Heater/Sample Heater

The can/sample heater connects to the front of the electronics via a four pin male QM multi-pole connector wired as follows:

PIN NUMBER	FUNCTION
A	SUPPLY +ive
B	SUPPLY -ive
C	SENSE -ive
D	SENSE +ive

The sense connections are for the power measurement (see section 4.2.8).

### 3.2.4 Actuator Connectors

The actuators are connected to the front panel of the electronics rack. The pin connections are shown below.

PIN NUMBER	FUNCTION
1	+ 24V SUPPLY
2	GROUND

### 3.2.5 Guard Heater Control Connector

The guard heater control module is connected to the guard heater power supply unit by a 2-metre cable with connections as follows:

#### Lemo Plug FGG.2B306.CLAD62

Pin 1 (An 0V)	Red
Pin 2 (Top DAC)	Blue
Pin 3 (Side DAC)	Green
Pin 4 (Bottom DAC)	Yellow
Pin 5 (Dig 0V)	White
Pin 6 (Quench Heater)	Black

Screen connected at both ends.

#### Lemo socket FGJ.2B.306.CLLD62

Skt 1
Skt 2
Skt 3
Skt 4
Skt 5
Skt 6

## 4. TROUBLE-SHOOTING

### 4.1 GENERAL

#### 4.1.1 Fuses

The most likely cause of problems from the electronics hardware is from fuses blowing. Each of the heater power outlets from the power supply unit is separately fused, the fuses being located on the rear panel. To change a fuse, first disconnect the unit from the mains supply and then unscrew the fuse from its holder. **Only fit a fuse of the correct rating**, as labelled next to the fuse holder.

#### 4.1.2 Diagnostics Tools

Several diagnostic tools are available for use by service engineers. The user may be asked to run these tools under guidance from HEL staff or agents. The most

sophisticated of these tools is DIAGPROJ an executable diagnostics program in the WinISO directory.

## **4.2 FAULT FINDING**

### **4.2.1 Power Supply Indicators Fail To Light**

If the power supply indicators fail to come on and the mains switch fails to illuminate then check the mains plug fuse and the mains inlet fuse at the rear of the rack. If both mains input fuses are intact and the front panel LED's do not illuminate contact your HEL agent. Note all thermocouples must be connected and in working order otherwise the over temperature trip will be activate as indicated by the illumination of the over temperature trip light. If this is the case please ensure all thermocouples are correctly connected then re-start the WinISO software to reset this safety trip.

### **4.2.2 Temperature Probes**

Temperature measurement faults almost always are due to a fault with a thermocouple usually due to chemical attack or physical damage. An open circuit on the thermocouple will give a high saturated temperature reading whereas an electrical short will give a low saturated temperature reading. Such faults are normally readily apparent. Should a guard heater thermocouple fail it will need to be replaced and will involve removing the guard heater set from the calorimeter to do this. Ideally all the guard heater thermocouples should be replaced at the same time using matched thermocouples. Calibrations should be checked after replacing thermocouples to ensure performance of the BTC has not been compromised or altered. The thermocouples used on the BTC are insulated from the sheath and thus should give an open circuit between the pins and the sheath even when tested with an electrical resistance meter on the coarsest setting. If not they should be replaced. This type of fault may be less obvious causing irregular behaviour only under certain conditions. A common cause of this type of fault is moisture ingress into the insulation inside the thermocouple sheath or connector resulting in a partial electrical short. The cables and connectors should be examined for obvious damage before replacing thermocouples.

### **4.2.3 Sample Heater**

Heater faults almost always take the form of broken or corroded wiring. Measuring the resistances between the connector pins will indicate the presence of such a fault. The sample heater will have a nominal resistance (greater than 12 Ohms, less than 100 Ohms depending upon the length of resistance wire used). Repeated bending and rebending of the wire may cause fatigue and fracture of the resistance wire. Heavy contamination of the mineral insulation around the wire or bare wires may result in an electrical short.

#### **4.2.4 Guard Heaters**

Guard heater faults are rare unless they have suffered physical or corrosion damage due to missile damage or leak of materials following a sample decomposition. There should be very high resistance (essentially an open circuit) between the element and the aluminium onto which they are fixed or the main frame of the BTC. The BTC may still work with a partial electrical short from the element but will typically be slower to respond and operate erratically. Other problems such as a guard heater not controlling at the set point temperature are typically due to a thermocouple problem or incorrect set up of the operating software.

#### **4.3 WATCHDOG FUNCTION**

The PCG card contains circuitry to detect the healthy functioning of the computer and software. Its action is discussed in section 5. A number of hardware and software features have been incorporated in the design which will alert the user to potential problems and render the reactor safe if an operator is not present.

## **5. SAFETY FEATURES**

### **5.1 ELECTRONICS WATCHDOG CIRCUITRY**

Watchdog circuitry is used on the PCG Card such that if it fails to receive a signal for a period of approximately 2-10 seconds then all analog and digital outputs are set into predetermined safe states. The analog outputs used to control the heaters are switched off. Normally all of the digital outputs are set to be off.

In addition to this there is a hardware safety trip on the temperature readings. Should a thermocouple be unplugged or fail open circuit an over temperature trip as indicated by an over temperature trip light on the front of the power supply unit will prevent the guard heaters being powered.

### **5.2 SOFTWARE RELATED SAFETY FEATURES**

The software automatically implements many safety features whenever predefined 'hardware safety limits' are reached. Many of the actions are taken automatically although options for manual overrides may be available.

A number of safety conditions are also pre-set in the experimental plans. These will shut down an experiment and activate a set of pre-programmed actions. These are not modifiable by the user. These safety limits will not exceed hardware specifications.

The user can specify maximum operating temperatures and pressures for the experiment although these in turn will be limited by the pre-set plan and hardware safety conditions. When these maximum operating conditions are exceeded the test will typically terminate to the cool down step in which the user can also specify if gas cooling (and in effect inerting) is activated.

## **6. INSTRUMENT CALIBRATION**

Annual servicing and calibration of the equipment by a HEL service engineer is recommended.

Additional calibration of the equipment may be required if, the equipment has been damaged and replacement parts have been fitted, or if the equipment has been relocated or significant changes have occurred in the environment.

The user has the facility to calibrate the equipment provided suitable equipment is available. The correct procedure for calibration of the equipment is given in volume 2. For a full calibration the user will need a Type K thermocouple simulator, an appropriate electronic pressure calibration gauge, pressure supply and connection assembly, a sample heater "break out" box, calibrated multi-meter and connection leads. It is important that the electronics have been switched on and allowed to warm up before performing a calibration.