3 ZONE METROLOGICAL FURNACE MODEL 465

User Maintenance Manual/Handbook

This manual refers to the following cells:

Tin Freeze-Point Cell
Lead Freeze-Point Cell
Zinc Freeze-Point Cell
Antimony Freeze-Point Cell
Aluminium Freeze-Point Cell
Silver Freeze-Point Cell
Gold Freeze-Point Cell
Copper Freeze-Point Cell

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GUARANTEE

This instrument has been manufactured to exacting standards and is guaranteed for twelve months against electrical break-down or mechanical failure caused through defective material or workmanship, provided the failure is not the result of misuse. In the event of failure covered by this guarantee, the instrument must be returned, carriage paid, to the supplier for examination and will be replaced or repaired at our option.

FRAGILE CERAMIC AND/OR GLASS PARTS ARE NOT COVERED BY THIS GUARANTEE

INTERFERENCE WITH OR FAILURE TO PROPERLY MAINTAIN THIS INSTRUMENT MAY INVALIDATE THIS GUARANTEE

RECOMMENDATION

The life of your ISOTECH Instrument will be prolonged if regular maintenance and cleaning to remove general dust and debris is carried out.

We recommend that this instrument to be re-calibrated annually.

ISOTHERMAL TECHNOLOGY LTD.
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The company is always willing to give technical advice and assistance where appropriate. Equally, because of the programme of continual development and improvement we reserve the right to amend or alter characteristics and design without prior notice. This publication is for information only.

CE EMC INFORMATION

This product meets the requirements of the European Directive on Electromagnetic Compatibility (EMC) 89/336/EEC as amended by EC Directive 92/31/EEC and the European Low Voltage Directive 73/25/EEC, amended by 93/68/EEC. To ensure emission compliance please ensure that any serial communications connecting leads are fully screened.

The product meets the susceptibility requirements of EN 50082-1, criterion B.

Symbol Identification	Publication	Description
	ISO3864	Caution (refer to manual)
	IEC 417	Caution, Hot Surface

⚠ ELECTRICAL SAFETY

This equipment must be correctly earthed.

This equipment is a Class I Appliance. A protective earth is used to ensure the conductive parts cannot become live in the event of a failure of the insulation.

The protective conductor of the flexible mains cable which is coloured green/yellow MUST be connected to a suitable earth.

The Blue conductor should be connected to Neutral and the Brown conductor to Live (Line).

Warning: Internal mains voltage hazard. Do not remove the panels.

There are no user serviceable parts inside. Contact your nearest Isotech agent for repair.

Voltage transients on the supply must not exceed 2.5kV.

Conductive pollution, e.g. Carbon dust, must be excluded from the apparatus. EN61010 pollution degree 2.

HEALTH AND SAFETY INSTRUCTIONS

1. Read this entire manual before use.

2. Wear appropriate protective clothing.

3. Operators of this equipment should be adequately trained in the handling of hot and cold items and liquids.

4. Do not use the apparatus for jobs other than those for which it was designed, i.e. the calibration of thermometers.

5. Do not handle the apparatus when it is hot (or cold), unless wearing the appropriate protective clothing and having the necessary training.

6. Do not drill, modify or otherwise change the shape of the apparatus.

7. Do not dismantle the apparatus.

8. Do not use the apparatus outside its recommended temperature range.

9. If cased, do not return the apparatus to its carrying case until the unit has cooled.

10. There are no user serviceable parts inside. Contact your nearest Isotech agent for repair.

11. Ensure materials, especially flammable materials are kept away from hot parts of the apparatus, to prevent fire risk.

12. Ensure adequate ventilation when using oils at high temperatures.

CERTIFICATE OF TEST

SERIAL NO:

Prior to despatch the following tests were carried out:

Insulation Test: Date:

Earth Impedance: Date:

Temperature cycle to maximum temperature: Date:

Communication's test (if applicable): Date:

Laboratory Test at 1080°C the profile was optimised

Top controller offset

Bottom controller offset

Date:

Comments or concessions:

Absolute values checked at and

Controller parameters are listed in an appendix of the handbook.

The above serial no. furnace was tested and found to comply with the specifications.

Signature:

CAUTIONARY NOTE

PRODUCTS OF ISOTHERMAL TECHNOLOGY LTD ARE INTENDED FOR TECHNICALLY TRAINED AND COMPETENT PERSONNEL FAMILIAR WITH GOOD LABORATORY PRACTICE. IT IS EXPECTED THAT PERSONNEL USING THIS EQUIPMENT WILL BE KNOWLEDGEABLE AND SKILFUL IN THE MANAGEMENT OF APPARATUS WHICH MAY BE UNDER POWER OR UNDER EXTREMES OF TEMPERATURE (MOLTEN METALS, CRYOGENIC LIQUIDS, ETC.) AND WILL APPRECIATE THE HAZARDS WHICH MAY BE ASSOCIATED WITH, AND THE PRECAUTIONS TO BE TAKEN WITH, SUCH EQUIPMENT.

THIS EQUIPMENT MUST BE EARTHED

THE FURNACE IS SUPPLIED WITH A FUSE CARRIER FITTED WITH A NEUTRAL LINK. THIS IS CLEARLY LABELLED AND IS FOR USE WITH A MAINS SYSTEM WITH A NEUTRAL LINE, SUCH AS THE UK SUPPLY. IF THE FURNACE IS TO BE USED ON A SYSTEM WHERE BOTH SUPPLY LINES ARE LIVE WITH RESPECT TO EARTH THEN THE NEUTRAL FUSE LINK SHOULD BE REPLACED WITH A FUSE. A SPARE FUSE IS SUPPLIED WITH THE FURNACE.

FUSE AND NEUTRAL LINK REPLACEMENT

HAZARDOUS VOLTAGES ARE EXPOSED WHEN THE REAR FURNACE PANEL IS REMOVED. BEFORE REMOVING THE PANEL YOU MUST ISOLATE THE FURNACE FROM THE ELECTRICAL SUPPLY.

TO REPLACE THE MAIN ELECTRICAL FUSE OR TO REPLACE THE NEUTRAL LINK BAR IT IS NECESSARY TO REMOVE THE TOP PANEL, SEE WARNING ABOVE. THE PANEL WILL BECOME FREE AFTER THE FOUR CORNER SCREWS ARE REMOVED.

THE TOP HALF OF THE FUSE CARRIER PULLS FREE FROM THE LOWER BODY.

TWO SPARE FUSES ARE SUPPLIED WITH THE FURNACE.

3 ZONE METROLOGICAL FURNACE

The 3 Zone Metrological Furnace has two independent control systems, one for temperature control and the other for over-temperature protection.

Proportional, integral and derivative adjustments for the controller (Eurotherm 818) and the means for obtaining optimum settings are described in the manufacturer's manual, which is included as part of this manual. These parameters are initially adjusted to optimal conditions for the Isotech Laboratory environment (ambient temperature of 20 ±2°C) in which the furnace was tested prior to shipment.

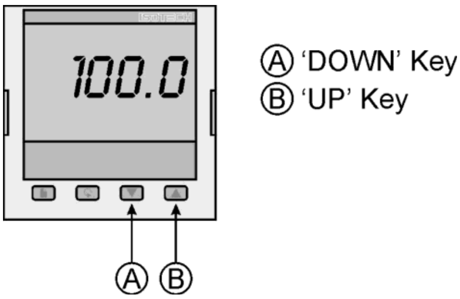
The furnace uses a Eurotherm 2116 over-temperature cut-off controller. On these models RESET button is located below the over-temperature controller. The SP value should be set approximately 50°C higher than the operating controller setting. If this limit should be exceeded the red push button switch will reset the apparatus once the furnace temperature has fallen below the SP value.

To turn on system power at start up (or as required), it is necessary to depress the red push button.

Altering the Alarm Temperature, (Setpoint)

1. Switch the unit on.

2. The controller will briefly show its software version before displaying an indication of the furnace temperature.



3. Momentarily press either the UP or DOWN key once to display the alarm setpoint. This value should be set to 50°C approximately above the operating controller temperature.

4. To alter the value press and hold the UP key to raise the value or the DOWN key to lower the value.

5. The display will return to show the nominal furnace temperature when no key is pressed for 0.5 second.

ISOTECH

COMMISSIONING:

Please refer to Figure 3 and the Test Certificate

The furnace requires a supply of 110VAC at up to 32 A.

For operation from 230VAC an isolating step down transformer should be used, a suitable model is available from Isotech.

Check the impedance between Live and Neutral and Live and Earth.

(Accept a value as low as 2MΩ on arrival since moisture may be present after transportation).

Connect the interconnecting leads between the control box and the furnace.

Connect the furnace to a suitable supply.

Connect the box to the supply - see page 10.

Read the controller handbooks to familiarise yourself with operation of the units included.

Plug in, and switch on. Press the red reset button; this will energise the apparatus. The controllers, after a self-check should indicate temperature, or offsets for the top and bottom zones.

The rate of change of temperature for the ceramic furnace liner at low temperature must be controlled, and for this reason the furnace controller has been pre-programmed to warm up slowly to its working temperature. The parameters used to affect this control are not directly accessible, for safety reasons.

Ensure the ramp light is illuminated on the main controller.

Set the main controller to 100°C and the top and bottom controllers offset to 0°C.

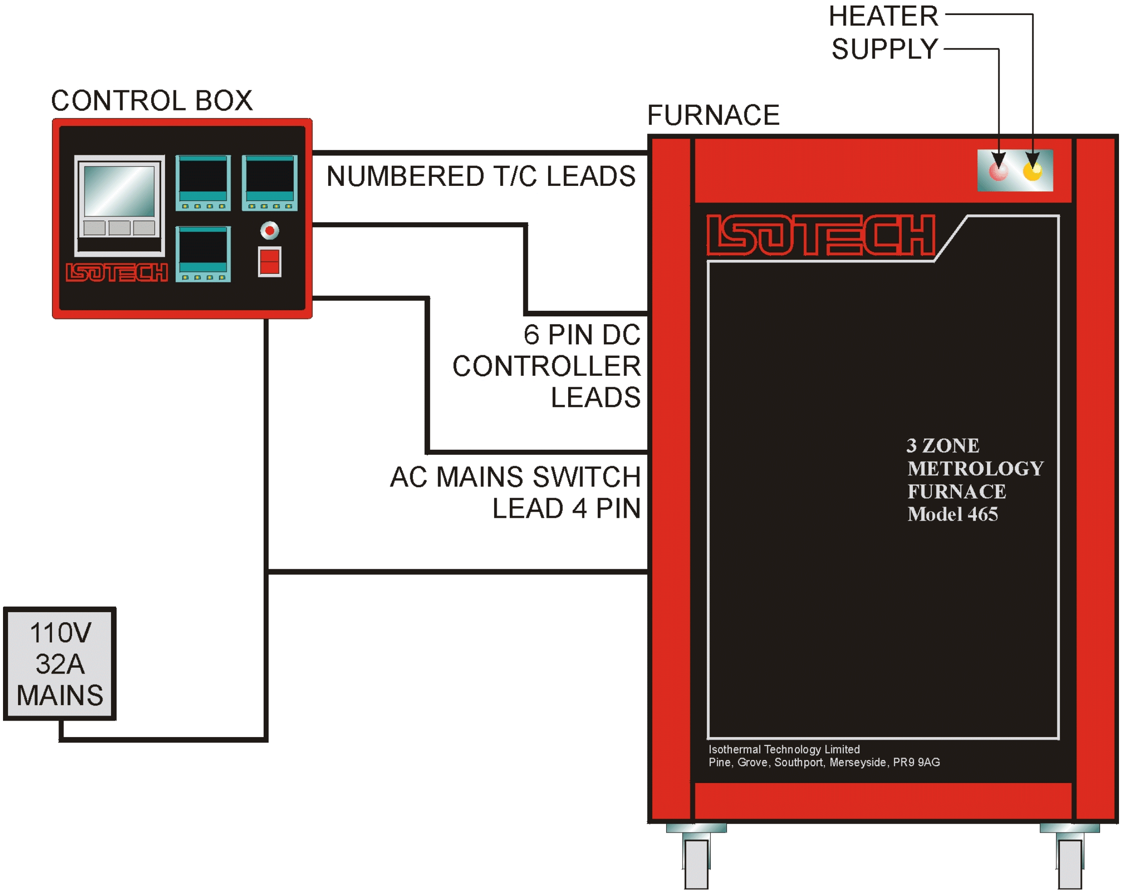
Set the over-temperature controller to 150°C.

After 3 or 4 hours the main controller will have stabilised. Reduce the over-temperature controllers setting until the relay operates and the apparatus has switched off. The apparatus is now ready for use.

All other controller functions are hidden from the operator. The values are preset and should not be changed.

At the rear are two water connections, these should be connected to a controllable supply and a flow of 1 to 2 litres of water per minute established - this prevents the top of the cabinet being over heated at high furnace temperatures. Run the water for 1 hour to check the furnace for absence of leaks (the floor underneath the cabinet should be inspected). The water flow is only required when the furnace temperature is greater than 700°C.

FURNACE CONNECTIONS



RECOMMENDED SETTING-UP PROCEDURE

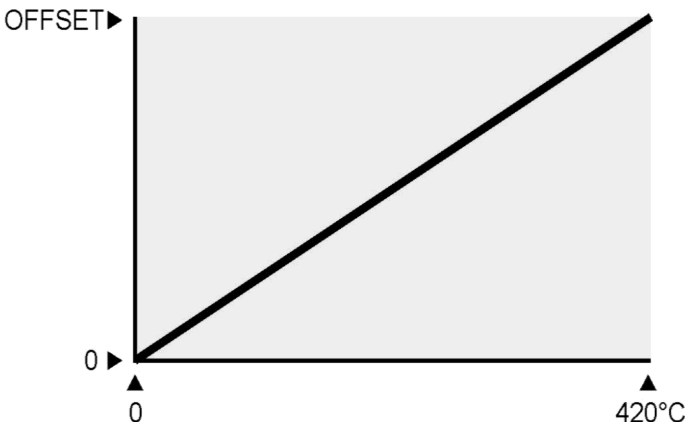
The purpose of the 3-Zone Furnace is to create an even temperature profile along that part of the furnace in which the freeze point cell will sit.

The main heater is used to set the required temperature. The top and bottom heaters are used to compensate for the end-effect heat losses.

Before leaving Isotech, the top and bottom heaters have been set to give best profile at 1083°C (the copper point), unless specified otherwise.

For lower temperatures (for example, the tin and indium points) these offsets will need to be reduced. To change the offsets follow the following procedure.

1. Open flap at the bottom of the top or bottom heater controller. Press the right hand pad until Sp appears, then press "up" or "down" keys to increase or decrease the effect.



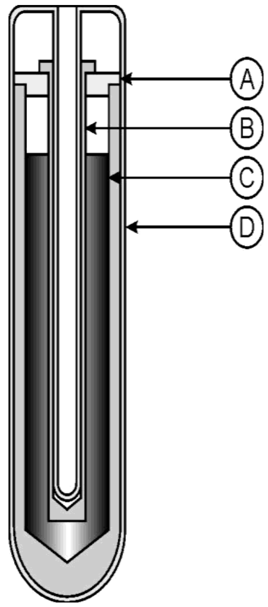
As a first approximation, the offsets may be reduced proportionally with temperature.

The settings for your particular furnace are given on the attached certificate.

If you wish, you can check your furnace offsets by putting a small thermocouple between a basket containing a cell assembly and the wall of the furnace. By moving the thermocouple up from the bottom of the well in 50mm steps, the profile of the furnace may be obtained and optimised by adjustment of the top and bottom heater offset. ±2°C over the cell is usually sufficient to give a good long plateau.

With care and patience gradients may be reduced to ±0.3°C at any desired temperature.

Figure 1: Sealed Metal Freezing Point Cell



A High-purity graphite crucible and cover
B High-purity graphite sleeve
C High-purity metal
D Fused-quartz envelope, filled to give a pressure of 1 standard atmosphere at the metal freezing temperature

Figure 2: Sealed Metal Freezing-Point Cell Body Dimensions

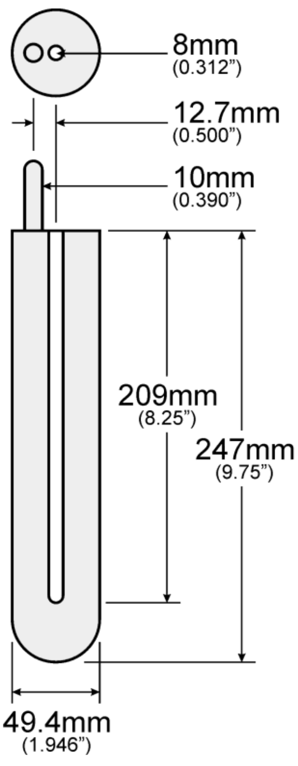
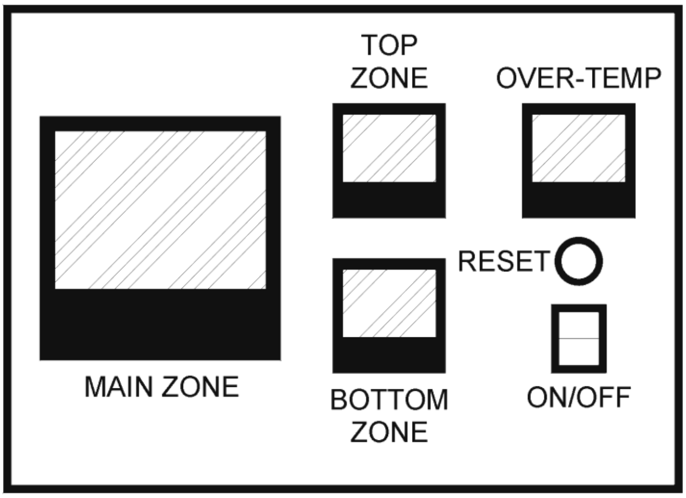


Figure 3: Controller Case

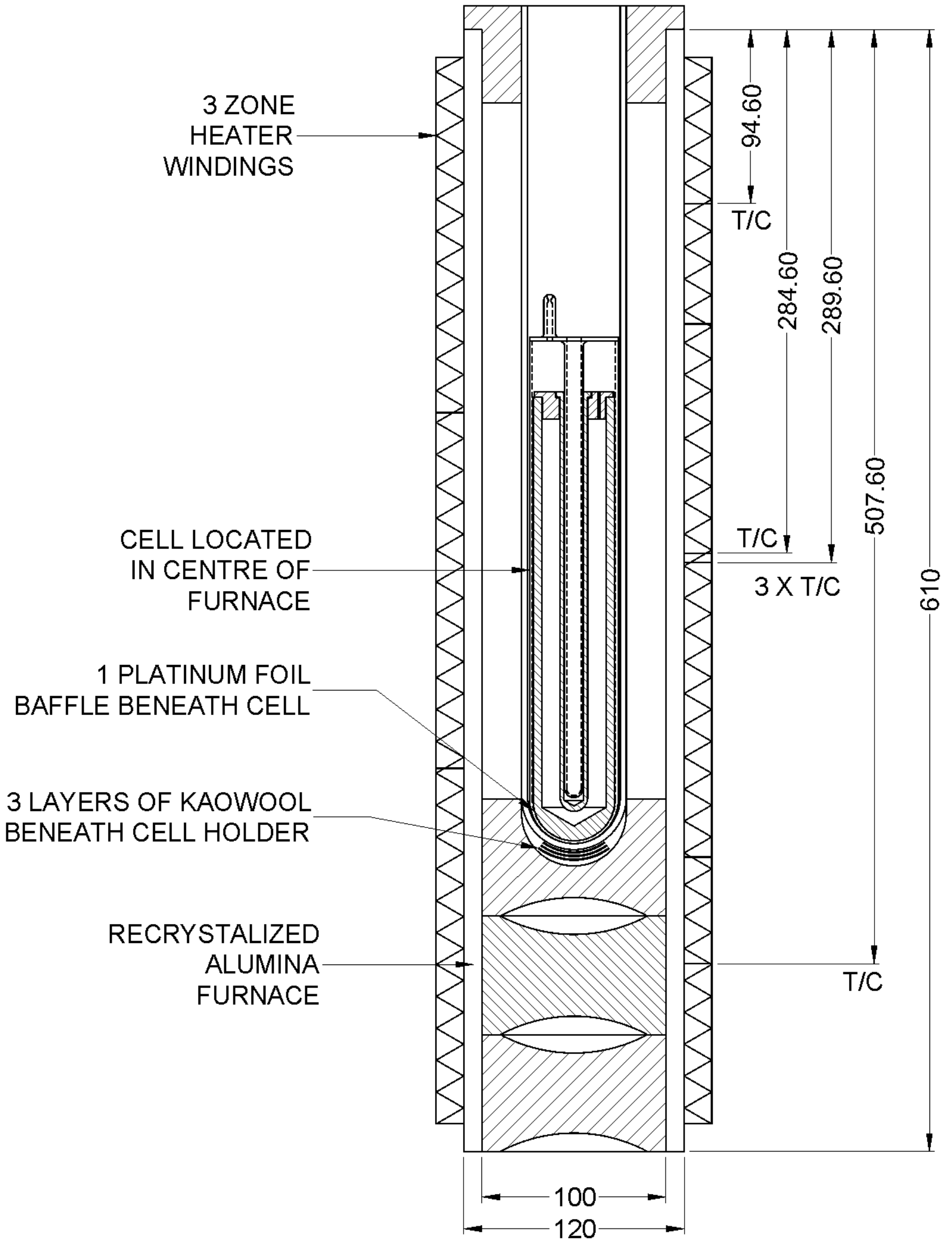


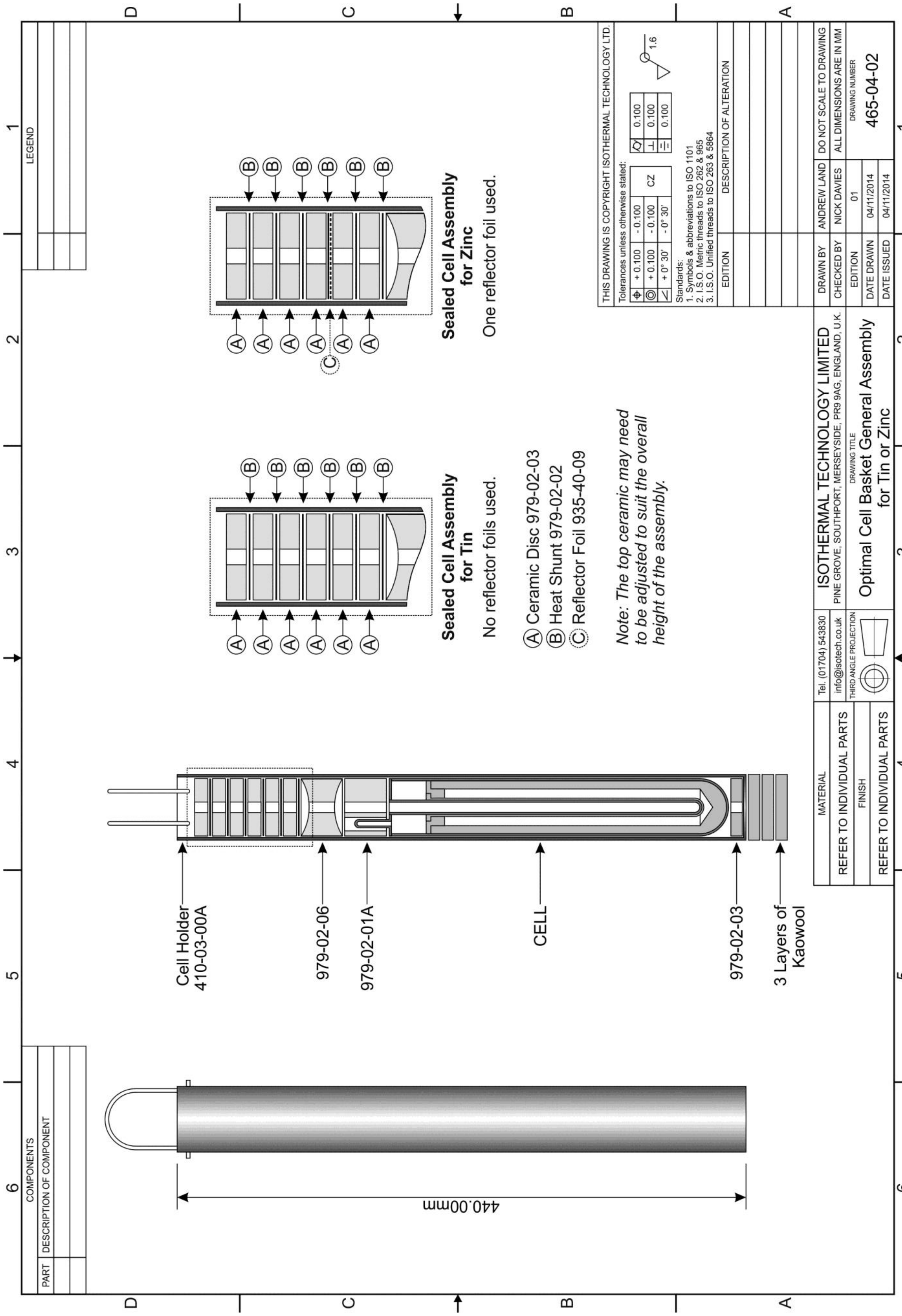
COMMUNICATIONS

Refer to the additional handbooks; the main controller has address 1, the top zone - address 2 and the bottom zone - address 3.

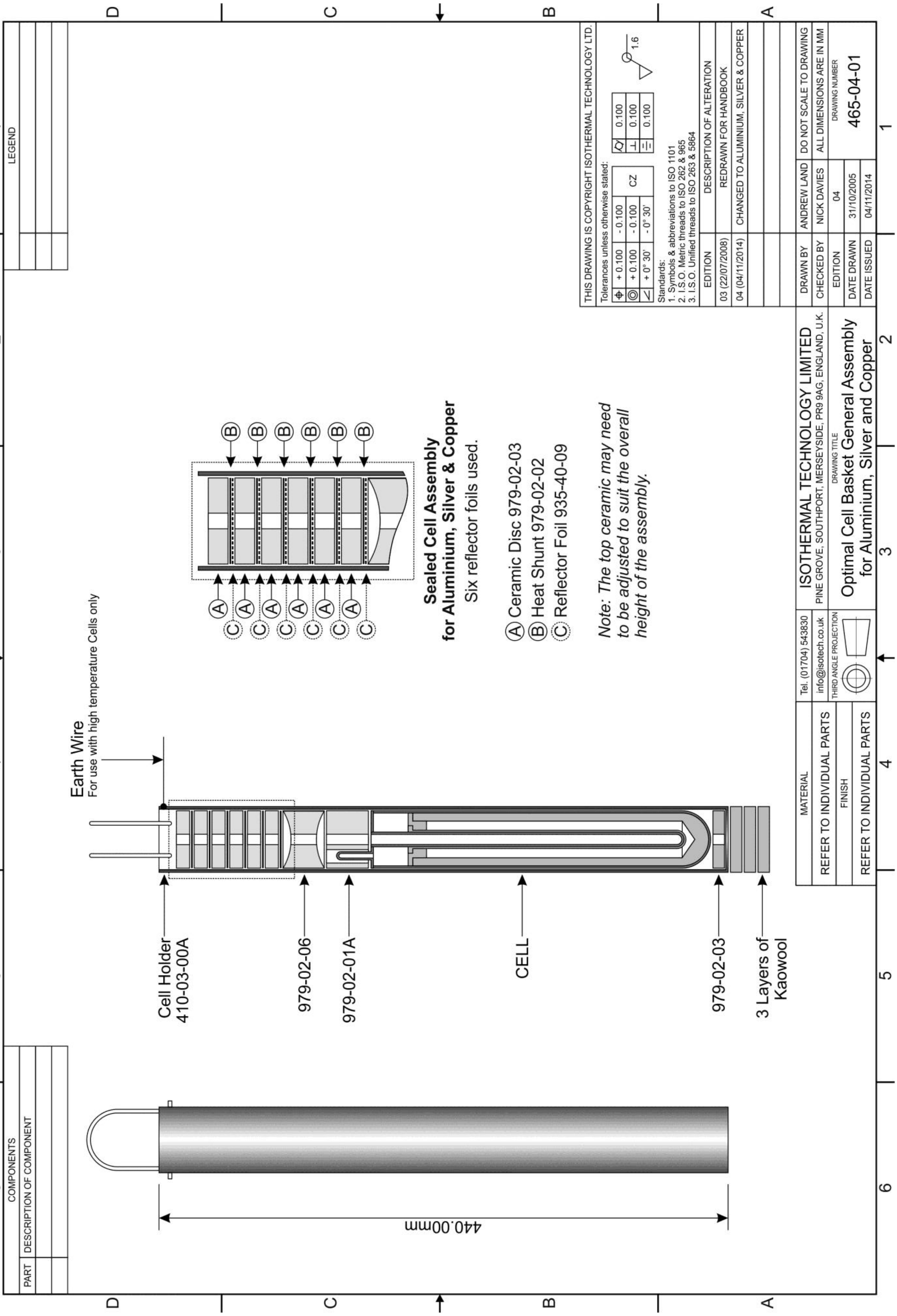
The over temperature controller does not has a communications link.

Furnace and Holder Diagram





Cell Holder 410-03-00A
979-02-06
979-02-01A
CELL
979-02-03
3 Layers of Kaowool
440.00mm
Sealed Cell Assembly for Tin
No reflector foils used.
Sealed Cell Assembly for Zinc
One reflector foil used.
A Ceramic Disc 979-02-03
B Heat Shunt 979-02-02
C Reflector Foil 935-40-09
Note: The top ceramic may need to be adjusted to suit the overall height of the assembly.
THIS DRAWING IS COPYRIGHT ISOTHERMAL TECHNOLOGY LTD.
Tolerances unless otherwise stated:
+ 0.100 - 0.100
+ 0.100 - 0.100
+ 0° 30' - 0° 30'
CZ
0.100
0.100
0.100
1.6
Standards:
1. Symbols & abbreviations to ISO 1101
2. I.S.O. Metric threads to ISO 262 & 965
3. I.S.O. Unified threads to ISO 263 & 5864
EDITION
DESCRIPTION OF ALTERATION
MATERIAL
REFER TO INDIVIDUAL PARTS
FINISH
REFER TO INDIVIDUAL PARTS
Tel. (01704) 543830
info@isotech.co.uk
THIRD ANGLE PROJECTION
ISOTHERMAL TECHNOLOGY LIMITED
PINE GROVE, SOUTHPORT, MERSEYSIDE, PR9 9AG, ENGLAND, U.K.
DRAWING TITLE
Optimal Cell Basket General Assembly for Tin or Zinc
DRAWN BY ANDREW LAND
CHECKED BY NICK DAVIES
EDITION 01
DATE DRAWN 04/11/2014
DATE ISSUED 04/11/2014
DO NOT SCALE TO DRAWING
ALL DIMENSIONS ARE IN MM
DRAWING NUMBER
465-04-02
LEGEND
COMPONENTS
PART
DESCRIPTION OF COMPONENT


Earth Wire
For use with high temperature Cells only
Cell Holder
410-03-00A
979-02-06
979-02-01A
CELL
979-02-03
3 Layers of
Kaowool
440.00mm
Sealed Cell Assembly
for Aluminium, Silver & Copper
Six reflector foils used.
A Ceramic Disc 979-02-03
B Heat Shunt 979-02-02
C Reflector Foil 935-40-09
Note: The top ceramic may need
to be adjusted to suit the overall
height of the assembly.
LEGEND
COMPONENTS
PART
DESCRIPTION OF COMPONENT
THIS DRAWING IS COPYRIGHT ISOTHERMAL TECHNOLOGY LTD.
Tolerances unless otherwise stated:
+ 0.100
- 0.100
+ 0.100
- 0.100
+ 0° 30'
- 0° 30'
CZ
0.100
0.100
0.100
1.6
Standards:
1. Symbols & abbreviations to ISO 1101
2. I.S.O. Metric threads to ISO 262 & 965
3. I.S.O. Unified threads to ISO 263 & 5864
EDITION
DESCRIPTION OF ALTERATION
03 (22/07/2008)
REDRAWN FOR HANDBOOK
04 (04/11/2014)
CHANGED TO ALUMINIUM, SILVER & COPPER
MATERIAL
REFER TO INDIVIDUAL PARTS
FINISH
REFER TO INDIVIDUAL PARTS
Tel. (01704) 543830
info@isotech.co.uk
THIRD ANGLE PROJECTION
ISOTHERMAL TECHNOLOGY LIMITED
PINE GROVE, SOUTHPORT, MERSEYSIDE, PR9 9AG, ENGLAND, U.K.
DRAWING TITLE
Optimal Cell Basket General Assembly
for Aluminium, Silver and Copper
DRAWN BY
ANDREW LAND
CHECKED BY
NICK DAVIES
EDITION
04
DATE DRAWN
31/10/2005
DATE ISSUED
04/11/2014
DO NOT SCALE TO DRAWING
ALL DIMENSIONS ARE IN MM
DRAWING NUMBER
465-04-01

REALISING THE FOLLOWING FIXED POINTS: TIN, LEAD, ZINC, ANTIMONY, ALUMINIUM, SILVER, GOLD AND COPPER

Some of these metals are characterised by a relatively short supercool (supercool is the characteristic of a freezing pure metal to remain liquid at a temperature below that at which the solid melts). The supercool of these metals can be expected to be less than 0.5°C. However typical supercool for Copper is 1.5°C to 2.5°C and Tin 10°C or more.

The cell is placed in the furnace, suitable insulation and cover added and a monitoring thermometer inserted (for Al and Ag an HTSPRT (High Temperature Standard Platinum Resistance Thermometer) and for Cu a standard thermocouple of suitable type construction). The furnace controller is set 5°C to 10°C above the expected melt temperature. The temperature rise is monitored with a bridge and/or recorder connected to the thermometer.

Following the melt arrest, the temperature of the cell will rise to the controlled temperature. The metal in the cell is now entirely in the liquid phase and may be maintained in this condition for any desired period of time, for example, to accommodate to a calibration schedule.

To freeze, the furnace controller is set below the actual freeze temperature (for pure metals, melt and freeze temperatures are theoretically identical). The suggested setting is 1°C below the freeze temperature; this is, assuredly, below the bottom of the supercool. The furnace is allowed to cool to this new setpoint temperature, taking typically 30 to 45 minutes to do so.

When the monitor indicates that the cell is at, or below, its freeze temperature, the monitor is removed to the pre-warming tube of the furnace and replaced by a rod of quartz from ambient temperature. This initiates nucleation. After 2 minutes the rod can be removed and replaced by the monitor again.

This procedure creates a radial freeze from the inside and outside walls of the cell towards the centre.

A typical melt/freeze sequence is shown in Figure 4.

If the cell is left too long in the furnace without initiating the freeze as described above, nucleation will occur and the cell will begin to freeze from the bottom of the cell upwards.

This will result in a short, imperfect, plateau and, moreover, give an incorrect value of freeze point (typically 10mK below that expected).

Depending upon factors such as furnace control and the number of thermometers successively loaded into the cell, plateau durations between an hour and many hours may be achieved. Thermometers may be preheated prior to transfer to the cell. A pocket is provided in the furnace for this purpose. It is wise to ascertain from time to time that the plateau is still in existence, by checking the cell temperature with the monitoring thermometer at intervals within the measurement sequence.

Let us suppose that Th_m is the monitoring thermometer and $Th_{1...n}$ are thermometers to be calibrated. If n=2 a suitable sequence might be

Th_m, Th_1, Th_m, Th_2, Th_m

and if n=4

Th_m, Th_1, Th_2, Th_m, Th_3, Th_4, Th_m

The Th_m measured last should be equal in indication to that of the Th_m measured first to ensure that the plateau has been present for Th_1, etc.

At the temperatures of Tin, Lead and Zinc it is generally permissible to withdraw a thermometer, of type Isotech 909, directly into room temperature. After exposure to Aluminium and Silver point temperatures, the thermometer must be cooled slowly to 450°C. Slow cooling is vital to ensure the thermometer characteristics remain unaltered.

FREEZING THE TIN CELL

Realisation of the Tin plateau is accomplished in a manner similar to those of Lead and Zinc with the following exception.

Tin can supercool as much as 10°C. If the furnace were allowed to cool to the nucleation point, it would probably not recover in time to realise the plateau.

Following melt; reduce the temperature of the furnace to a few tenths of a degree below the anticipated freeze plateau temperature. Prior to supercool, and with the thermometer still in place, withdraw the cell in its Inconel basket, from the furnace. Suspend the basket (with cell) in ambient air. Continue monitoring until the temperature begins to rise. Return the cell to the furnace, remove the monitor thermometer and replace with a quartz rod from ambient temperature. After 2 minutes remove the rod and replace the thermometer. When the monitoring thermometer has shown no change for some minutes, the plateau has been achieved.

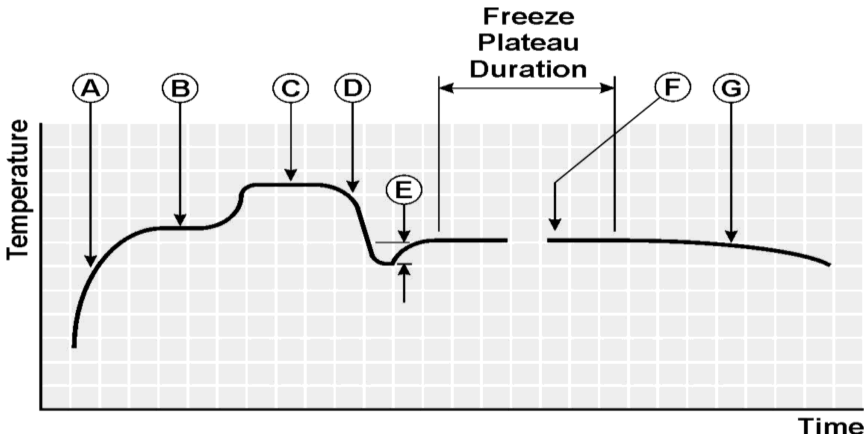
A typical melt/freeze sequence is shown in Figure 4.

A USEFUL HINT

When first creating freezes use large under settings - typically 3 to 5°C BELOW the freeze plateau. The result will be a shorter freeze time than ideal, but will engender confidence in establishing a plateau. Once familiar with the procedure using coarse settings, on subsequent exercises bring the setting of the controller closer to the known freeze temperature to increase the plateau length.

There follows later a tutorial about Fixed Points.

Figure 4: Typical Melt Freeze Sequence



A. Initial temperature rise
B. Temperature arrest during melt
C. Furnace-controlled temperature
D. Temperature drop caused by resetting furnace controller in readiness for freeze plateau realization
E. Depth of supercool (particularly pronounced for Tin)
F. Plateau showing constant temperature during freeze
G. Temperature drop at end of freeze plateau down to the set point of the furnace controller

MAINTENANCE

Unless damaged in transit, the apparatus should operate for many years without maintenance or fault.

It has been common practice in the past to list a number of possible fault modes and corrective actions. However, our experience suggests that the very low incidence of failure almost implies modes not encountered previously and therefore not easy to envisage before.

CELL HANDLING

In order to facilitate introduction to, and removal from the furnace, Isotech can provide for each cell, supplementary equipment largely comprising an Inconel basket with detachable handle.

To prevent the cell-surface becoming discoloured, it is recommended that, before using the cell, the basket and insulation be placed in the furnace and the furnace be taken to above the cell working temperature for at least 2 hours. This operation outgases the basket and insulation, which may smoke and discolour during this first temperature excursion. The cell can then be inserted into the basket in readiness for use. If removing the cell with a thermometer in its pocket (e.g. tin cell), extreme caution is necessary in applying support by means of the diametrically-pivoted handle. The handle will need to be maintained in a non-vertical plane while being used for removing and replacing the assembly.

CELL KIT

Basket, handles, discs of ceramic insulation, Inconel baffles and platinum foil discs if required.

Sketches show the recommended assembly of the cell basket (and insulation discs) in a furnace core.

<u>NOTE ON THERMOMETER USE AT HIGH TEMPERATURE</u>

A thermometer of the Standard Platinum Resistance Thermometer type, such as ITL Model 909 or 962, should never be completely withdrawn into ambient temperature when its temperature is above 500°C. Mechanical forces may be introduced which will cause strains (these can probably be annealed out) or quenched-in vacancies in the platinum lattice (these can generally not be removed by heat treatment) and consequent shifts in calibration. Thermometers can be withdrawn in 1cm length intervals, over a period of perhaps 5 minutes, until the thermometer temperature is known to be below 500°C and they may then be withdrawn rapidly. The same precaution applies to re-insertion of thermometers.

For accurate work the monitoring thermometer should be cooled slowly to 450°C then annealed at 450°C for ½ to 4 hours after each such use and its water triple point resistance measured. This is the actual water triple point measurement that should be used in calculating ratios for the determination of temperature.

SPARE PARTS

Silicon Carbide Pre-Heat Tube 19½"	935-29-20
High Temperature Kaowool Insulation	932-20-43
Middle Ceramic	465-03-10
Lower Ceramic (x2)	465-03-11
Upper Ceramic	465-03-09

SERVICING THE 3 ZONE METROLOGICAL FURNACE

No regular servicing is required.

THERMOMETRIC FIXED POINTS - A TUTORIAL

The International Temperature Scale, the scale most used in science and industry, is based on a series of fixed point temperatures. Fixed points involve two-phase or three-phase equilibria of, ideally, pure materials to which constant temperature values have been assigned by primary thermometry. Defining fixed points are chosen to be as few in number as is consistent with the need to establish satisfactory interpolation procedures.

There are secondary reference points which, also, are two-phase or three-phase equilibria of very pure materials, whose temperature values have been established by measurements made with interpolation instruments calibrated at the defining fixed points. Secondary reference points are useful for the calibration of thermometers having total ranges shorter than the interpolation ranges of the Scale. Generally, secondary points are admitted to the Scale only if the material is generally available in high purity and if sufficient reproducibility of the equilibrium temperature has been confirmed by measurements made independently by a considerable number of investigators. An average value (weighted according to individual uncertainties) is then used as the ITS temperature value.

Two-phase equilibria may be solid-liquid, liquid-vapour, or solid-vapour. From the Phase Rule of Gibbs:

$$P + F = C + 2$$

P is an integer equal to the number of phases present, C is the number of components present - for a pure material C = 1 - and F is an integer representing the number of degrees of freedom. It is evident that the temperatures of two-phase equilibria are pressure-dependent (one degree of freedom only) whereas equilibria in which all three phases are present (triple points) are characterised by unique values of temperature and pressure (zero degrees of freedom).

A necessary condition to establish a triple point is to contain the appropriate material in a sealed enclosure from which all other materials, including air, have been evacuated, leaving a space to be filled by the vapour phase at a pressure appropriate to the temperature. When the three-phase (solid, liquid, vapour) condition has been established, these parameters will settle to their unique triple-point values.

The defining fixed points above 0°C are liquid-solid equilibria of high-purity metals. Pressure-dependence is small (see the table on page 26) and thermal capacity and thermal conductivity are relatively high. Metals are generally available with a purity of 99.999% ("five-nines") or 99.9999% ("six-nines").

Figure 1, page 12, shows the design of a cell for realising the liquid-solid equilibrium of pure metal. The metal is contained in a crucible of purified graphite, with a graphite cover and a graphite re-entrant sleeve. The crucible is enclosed in an envelope of fused quartz, which extends into the sleeve interior to form the thermometer well. The cell is charged with a pure metal, purged and filled with sufficient argon (or another inert gas) to give a pressure of 101kPa (1 standard atmosphere) at the freezing temperature and then sealed. Thus it is at once protected from contamination and supplied with an inert atmosphere at the required pressure at the equilibrium temperature. A correction for the effect of change in ambient pressure on freezing point need not be made. Sealed cells of this type have shown no measurable change after 15 years of use.

In general, sealed fixed-point cells are used in vertical-tube furnaces which provide good temperature control and sufficient cell immersion to make axial temperature gradients, in the measurement zone, negligible. With the cell in the furnace, the controller is first set about 5°C above the anticipated value corresponding to the melting temperature of the metal in the cell. The onset of melting is indicated by a cessation of temperature rise because of the latent heat required to produce the phase change. This melt plateau can last for a considerable period of time. When melting is complete, the cell temperature will rise to the furnace temperature.

The furnace temperature is then reduced to a value slightly below the melt temperature. The temperature falls until the first solid nucleus of metal is formed, at which stage the temperature drop is arrested. With both liquid and solid metal present in the cell, a constant temperature is maintained by the latent heat released by the freezing metal. The controller temperature setting will cause the rate of heat egress from the cell to be relatively low, thus generating a freeze plateau that can usually be maintained for a number of hours, during which time thermometers may be calibrated.

A variation on this is the establishment of the triple point of mercury. Since this temperature is below normal ambient, the apparatus in which the point is realised must provide refrigeration as well as controlled heat. A separate manual describes the use of this apparatus.

Another variation is the realisation of the melting point of gallium. This metal is used on the melt plateau rather than on the freeze plateau. A separate manual describes the use of the apparatus for realising this fixed point.

There are, unfortunately, no convenient metal freeze points or triple points at the cryogenic end of the Scale. The defining point applicable to long stem thermometers at the low end of their useful range is the triple point of argon. In practice, the difficulties of setting up conditions to facilitate this measurement can conveniently be circumvented by carrying out the alternative procedure of comparison calibration, in which the thermometer is compared, in an environment of boiling nitrogen, to a similar thermometer which possesses a calibration traceable to national standards. A separate manual describes the nitrogen boiling point apparatus.

The temperature at which the change of phase occurs at atmospheric pressure is specific to the upper, exposed, surface of the metal. However, it is not feasible (because of the temperature gradient in this locality of the thermometer well) to obtain an accurate measurement under this condition. The closest approach to temperature uniformity demands insertion of the thermometer to the foot of the well with the consequence that the change-of-phase temperature measured is influenced by the static pressure head of the column of metal above the effective level of the thermometer sensing element.

Corrections that are used to enable measured phase-change temperatures to be converted to values that would pertain at 1 standard atmosphere pressure, for the various metals (and for mercury and water at their triple points), are given in the table on page 26. For a given column height (of the order of 200mm for Isotech sealed freeze point cells), the correction will be proportional to metal density and to a coefficient expressing the sensitivity to pressure of the phase-change temperature. The sign of this coefficient will depend on whether the metal contracts or expands on freezing.

DEFINING FIXED POINTS AND RELATED DATA

FIXED POINT	ITS 90 TEMPERATURE		PRESSURE COEFFICIENT		ITL CELL DESIGNATION	SUITABLE APPARATUS
	°C	K	mK/bar	mK/m HEAD OF SUBSTANCE		
ARGON TP	-189.3442	83.8058			NOT AVAILABLE FROM ITL	
MERCURY TP	-38.8344	234.3156	+5.4	+7.1	ITL-M-17724	ITL-M-17725
WATER TP	0.01	273.16	-7.5	-0.73	ITL 811	ITL-M-18233
GALLIUM MP	29.7646	302.9146	-2.0	-1.2	ITL-M-17401	ITL-M-17402A/B
INDIUM FP	156.5985	429.7485	+4.9	+3.3	ITL-M-17668	ITL-M-17701, 17702W, ITL-M-17703, 17707
TIN FP	231.928	505.078	+3.3	+2.2	ITL-M-17669	ITL-M-17701, 17702W, ITL-M-17703, 17707
ZINC FP	419.527	692.677	+4.3	+2.7	ITL-M-17671	ITL-M-17701/2P*/3
ALUMINIUM FP	660.323	933.473	+7.0	+1.6	ITL-M-17672	ITL-M-17702P/17702S**, ITL-M-17703, 17705, 17706, 465
SILVER FP	961.78	1234.93	+6.0	+5.4	ITL-M-17673	ITL-M-17702P/17702S**, 465
COPPER FP	1084.62	1357.77	+3.3	+2.6	ITL-M-17674	ITL 17702S, 465

NOTES:

1. TP = Triple Point, MP = Melting Point, FP = Freezing Point
2. Pressure corrections that apply to triple point and to other sealed-cell measurements are determined solely by the pressure head of material in the cell; variability of atmospheric pressure has no effect on the measurements.

* Furnace with potassium heat-pipe for zinc freezing point.

** Furnace with either potassium or sodium heat-pipe at aluminium and silver freezing points.

GENERAL NOTE ON ISOTECH METAL FREEZE POINT CELLS

Isotech freeze point cells contain metal that is 99.9999+% pure, except that aluminium cells may be filled with metal not less than 99.999% pure, depending upon the availability of aluminium in suitable physical form.

The metal is contained in crucibles of high-purity graphite. After machining the graphite, any residual metal oxides are removed by exposure to fluorine at a very high temperature. Graphite, even of high density, cannot be guaranteed to be non-microporous. Some cells, in preparation or after use, will be seen to exude droplets or spicules of the contained metal on to the outer surface of the graphite crucible; some may show a film of metal. This is considered not to be a defect of the cell; it does not reduce its useful life nor change its equilibrium plateau temperature.

The cell is a fragile device. Although it is as rugged as is consistent with its materials and purpose, it must still be regarded as a kilogram, or more, of mass, loosely contained in a frangible shell. Cells should never be inverted, although they may be slowly turned to the horizontal and laid on their sides. Transporting cells by common carrier is not recommended and, as furnished, they must be hand-carried. A broken cell cannot, in general, be repaired, although a cell which is broken but sufficiently intact to contain its metal can be used for some time if contamination is avoided.

Each cell can be supplied with an Inconel container or basket 400mm (16") in length, in which the cell should be placed to facilitate removal from the furnace. The basket has two diametrically-opposite holes near its upper end in which a wire handle of suitable material (for example, 14 SWG Nichrome) may be temporarily attached, for use in the 3 Zone Furnace Model 465, the cell holder is inconel. (For use in the 465 furnace the inconel basket for Aluminium, Silver and Copper has an earth lead attached which must be connected during use to the connector provided on the furnace) see diagram. Once the cell is in the basket there may be no future need to remove it. It is urged that the basket always be used with tin cells, because the recommended practice includes removal of the cell from the furnace as part of the freeze cycle.

PRECAUTIONS TO PREVENT DEVITRIFICATION OF QUARTZ ENVELOPES

The crucibles (containing the metal) of Isotech sealed fixed point cells are encased in an envelope of pure fused quartz, whose purpose is to avoid contamination of the enclosed metal, by foreign metal ions or oxygen. To this end, it contains an inert gas whose pressure is 1 standard atmosphere at the metal freezing temperature.

Fused quartz is vitreous in nature but, like other glasses, can be stimulated to crystallise (devitrify) by external influences at high temperatures. The crystalline form is recognisable as a localised cloudy or milky appearance. Devitrification is progressive and irreversible.

Quartz glass which is the glass used to cover the Silver and Copper Cells has an annealing (softening) temperature of 1050°C. Some 35°C below the Copper Melt Point.

A user should not therefore be surprised if his Copper Cell begins to devitrify at these elevated temperatures.

A devitrified cell can no longer be assumed to be gas-tight. It may leak its enclosed gas and atmospheric air may leak into it. The pressure at the freeze point may, as a consequence, be incorrect and, more seriously, contamination may occur.

Silver and especially Copper Cells should be regularly checked by immersing them in clean hot water to make sure there are no leaks.

If a leak is detected the cell should be returned to Isotech for a new Quartz cover.

Sealed quartz cells can be used for thousands of hours without devitrification if precautions are taken to ensure that the outside surface is scrupulously clean before raising them to temperature. Any surface dirt, a water spot or a single fingerprint is a potential seed for devitrification. Before exposing to high temperature the exterior of the cell should be cleaned with a suitable alcohol such as Ethanol and then thoroughly wiped dry with clean tissue. (Similarly SPRT's to be inserted into the cell's re-entrant tube should be previously cleaned in this way).

It is advisable to handle cells with clean cloth gloves.

The precaution applies particularly to cells for use at temperatures in excess of 500°C, although Isotech advises that all cells be carefully cleaned before use.

GENERAL COMMENT

The use of freeze-point cells embodies one of nature's simplest and most predictable phenomena. However, the technique (requiring association of cells with other equipment) involves subtlety and operator sensitivity. Before relying upon measurements made in them, the operator should perform enough freezes to become familiar with the cell, furnace, control, monitoring thermometer and readout (as a system) to ensure that the plateau is clearly identifiable and sufficiently consistent.

ADDITIONAL SERVICES AND INFORMATION

Isotech operates one of the world's most comprehensive UKAS supervised Laboratories.

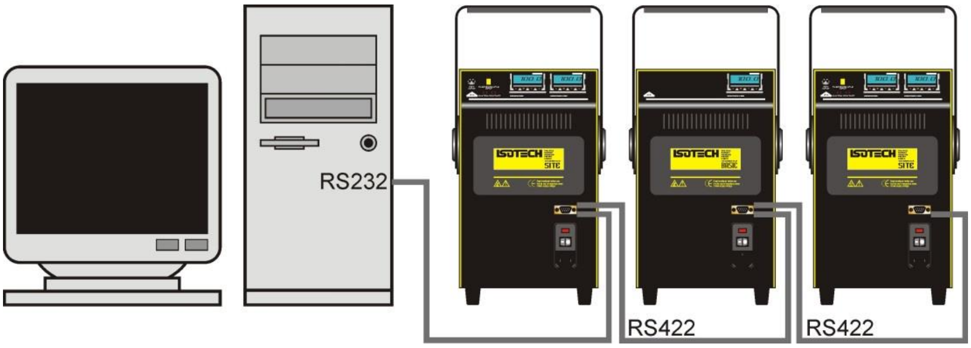
Training is available to customers at an agreed daily rate.

The Isothermal Journal of Thermometry is a must for all Laboratory Managers and is available for purchase.

USING THE PC INTERFACE

The Plus models include an RS422 PC interface and a special converter cable that allows use with the standard RS232 port. When using the bath with an RS232 port it is essential that this converter cable is used. Replacement cables are available from Isotech, part number ISO-232-432.   A further lead is available as an option, Part Number ISO-422-422 lead which permits up to 5 instruments to be daisy chained together.

The benefit of this approach is that a number of calibration baths may be connected together in a "daisy chain" configuration - and then linked to a single RS232, see diagram.



Note: The RS 422 standard specifies a maximum lead length of 1200M (4000ft). A true RS422 port will be required to realise such lead lengths. The Isotech conversion leads are suitable for maximum combined lead lengths of 10M that is adequate for most applications.

Connections

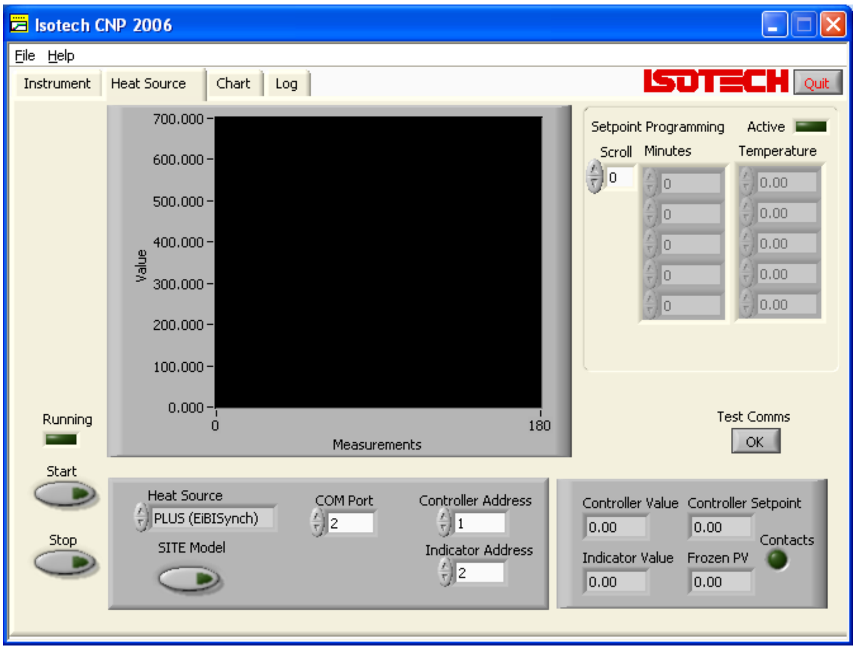
For RS232 use simply connect the Isotech cable, a 9 to 25 pin converter is included to suit PCs with a 25 pin serial converter.

RS422 Connections

Pin	Connection
4	Tx+ A
5	Tx- B
8	Rx+ A
9	Rx- B
1	Common

CAL NOTEPAD

Cal Notepad can be used can be used to log and display values from the Dry Blocks and an optional temperature indicator such as the milliK or TTI-10. The software requires Windows 9X, XP, a minimum of 5Mb of free hard drive space and free serial ports for the instruments to be connected.



DEVELOPMENT

Cal NotePad was developed by Isothermal Technology using LabVIEW from National Instruments. The license details are shown on the download page and in the Cal Notepad manual.

ISOTECH

HOW TO INSTALL CAL NOTEPAD

1. Download the ZIP from http://www.isotech.co.uk/downloads (7.6Mb)

2. Extract the files to a temporary folder

3. Run setup.exe

4. Follow the prompts which will install the application, a user manual with setup information and the necessary LabVIEW run time support files.

5. Should you ever need to uninstall the software then use the Add/Remove Programs option from the Control Panel.

PROTOCOL

The instruments use the "Modbus Protocol"

If required, e.g. for writing custom software the technical details are available from our Document Library at http://www.isotech.co.uk