DUAL FURNACE
FOR MODELS
M17705, M17706 & M17707

User Maintenance Manual/Handbook

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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.
PINE GROVE, SOUTHPORT
PR9 9AG, ENGLAND

TEL: +44 (0) 1704 543830/544611
FAX: +44 (0)1704) 544799

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)
⚠ (hot surface)	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.

DUAL FURNACE CERTIFICATE OF TEST

Serial No:

Prior to despatch the following tests were carried out.

Insulation Test	MΩ	Date
Earth Impedance	Ω	Date
Temperature cycle to maximum temperature		Date
Communications test (if applicable)		Date

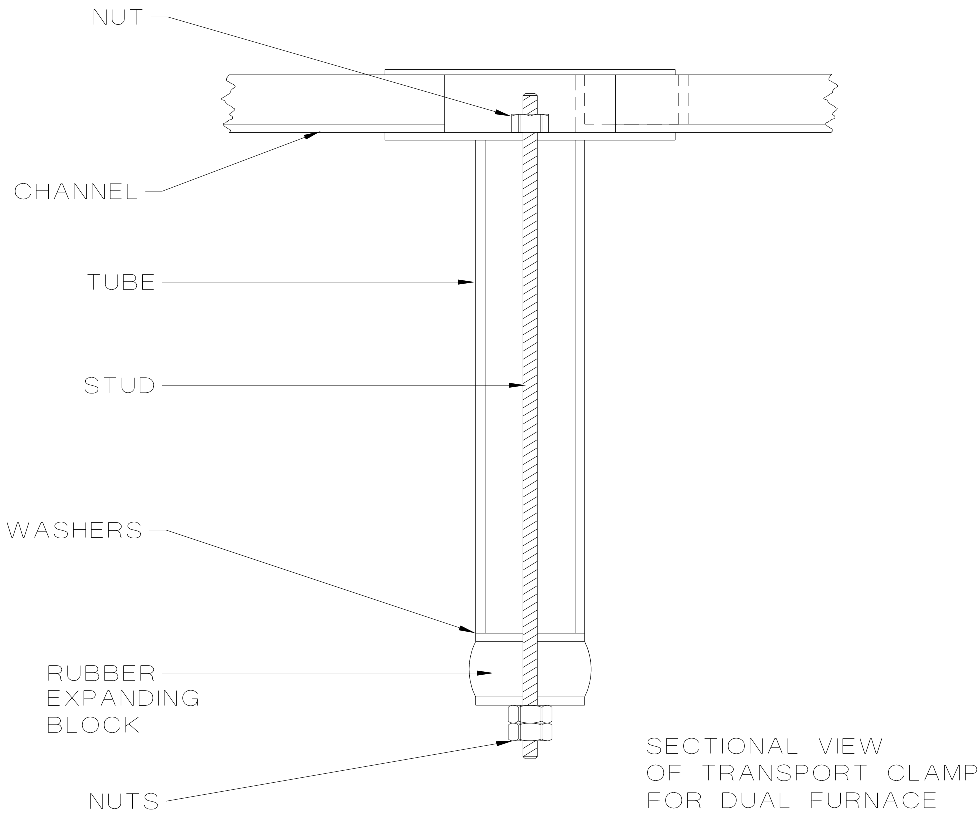
Laboratory test at °C

Comments or concessions

Controller parameters are listed in an appendix of the manual

The above Serial No. Furnace was tested as above and found to comply with the specifications.

UNPACKING



EVERY EFFORT HAS BEEN MADE TO PACKAGE THIS UNIT FOR TRANSPORT AND TO ENSURE ITS GOOD CONDITION ON ARRIVAL AT ITS DESTINATION.

BEFORE COMMISSIONING IT IS NECESSARY TO REMOVE THE FURNACE CORE TRANSIT CLAMP. TO AVOID DAMAGE PLEASE FOLLOW INSTRUCTIONS:

1. Slacken the central nut using the required tube spanner; the insertion of a screwdriver through the tube spanner will prevent the central stud turning.

2. Remove the 3 screws securing the transit clamp to the top of the unit. Slacken each screw by degrees equally.

3. Gently lift the transit clamp vertically; the attached tube and rubber securing block will thereby be extracted from the furnace core.

4. Normal commissioning procedure may now be followed.

5. Keep the transit clamp and use it if the furnace is ever transported.

SAFETY

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 rear panel; see warning above. The panel will become free after the four corner screws are removed.

The fuse holders will be seen at the lower right hand side of the cabinet. The black fuse holder will always contain a fuse. The white fuse holder will leave Isotech with a neutral shorting bar. As explained elsewhere, if your local supply does not have a neutral line but has both lines live with respect to earth this link should be replaced with a fuse and the holder labelled to this effect.

The top half of the fuse carrier pulls free from the lower body.

Two spare fuses are supplied with the furnace; they are type A2 and have a rating of 10A for the low and medium temperature furnace and 32A for the high temperature furnace.

INTRODUCTION

The Dual Furnace range of calibration apparatus comprises 3 models, all working on the same principle and having the following features in common:

1. A rack for storage, at around room temperature, of up to 4 thermometers. Lined with ceramic board, the rack will not be damaged if very hot thermometers are placed in it.

2. A furnace of diameter 50mm and depth 400mm which can be used to pre-warm or anneal up to 4 thermometers.

3. A pre-calibration pocket to install a single thermometer immediately prior to calibration or to house the monitor thermometer between periods of use.

4. A heat-pipe furnace to create optimal conditions for an appropriate range of fixed point cells. The use of freeze-point cells within the heat pipe is described in the manual which accompanies Isotech cells.

All 4 features are built into one cabinet, making the dual furnace a self-contained calibration facility.

Both component furnaces of the dual furnace can be fitted with communications interfaces for automated use.

THE 3 DUAL FURNACES

MODEL 17707

Contains a water heat pipe, giving an operational temperature range from 125°C to 250°C and thus making it an ideal system for realisation of Indium and Tin freeze points.

Its annealing furnace has a temperature range from near ambient up to 700°C.

MODEL 17706

Contains a potassium heat pipe with a temperature range from 400°C to 1000°C, thus encompassing Zinc, Aluminium and Silver freeze points.

Its annealing furnace has a temperature range from near ambient to 950°C.

MODEL 17705

Contains a sodium heat pipe with a temperature range from 450°C to 1100°C, thus constituting a facility for Aluminium, Silver, Gold and Copper freeze point measurements.

Its annealing furnace has a temperature range from near ambient to 950°C.

OPERATIONAL CONVENIENCE

Until the advent of the dual furnace, the handling of thermometers was treated as a separate problem, or ignored.

With the dual furnace, thermometers can be stored, pre-warmed, calibrated and annealed in one comprehensive facility, without introducing contamination or strain.

The two furnaces in each dual furnace have independent temperature controllers and independent adjustable over-temperature cut-outs for added security. These features are shown in the wiring diagram.

ACKNOWLEDGEMENT

The Isotech dual furnace owes its origin to Dr. P. Marcarino of Italy. His research and development solved many of the problems of contamination and handling of high temperature thermometers.

COMMISSIONING

Connect the step down transformer to a suitable 230Vac supply.

Before connecting the unit to the mains power supply, check the impedance between live and earth, and neutral and earth (values as low as 2MΩ on arrival are acceptable since moisture may be present after transportation).

Familiarise yourself with controller operation; manuals are supplied with all equipment.

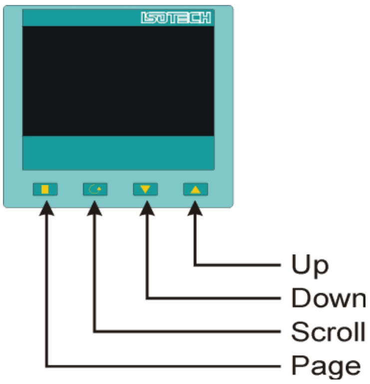
Plug in and switch on the dual furnace (figure 1) and operate the reset button. This will energise the annealing furnace controller. Adjust the set point to 100°C (450 and 500°C for potassium 17706 and Sodium 17705 models) and the over-temperature controller set point to 50°C above the controller set point. After 1 or 2 hours the annealing furnace will stabilise. Adjust its over-temperature set point downwards until the circuit relay trips, switching off the annealing furnace.

Later models of the furnace use a Eurotherm over-temperature cut-off controller. On these models the 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 AL1 value.

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

To adjust SP press the "Scroll" until the controller indicates "SP".

Press the UP or DOWN keys to modify the SP value.



NOTE; the annealing/pre-heating furnace controller has been pre-set to produce a slow change in temperature, in line with the heating and cooling rates recommended for annealing of thermometers. Do not over-ride the control settings.

The recrystallised alumina liner has a closed end and thermometers may be placed directly into the liner for annealing.

Switch on the main furnace (heat pipe) and press the reset button. This will energise the heater.

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

As soon as the minimum temperature has been reached, the initial restriction on the rate of approach to set point is removed.

The remainder of the instructions differ for the three models; reference should be made to the page clearly marked with the appropriate dual furnace model.

MODEL 17707 WATER HEAT-PIPE FURNACE

FINAL PART OF THE COMMISSIONING INSTRUCTIONS

The heat up rate of the furnace is limited to ensure long life of the heat pipe.

Impose a heat pipe set point of 150°C and set the over-temperature controller to 200°C.

Allow 1 to 2 hours for the furnace to stabilise and then reduce the over-temperature cut-out setting until the relay trips, thus switching off the furnace.

The 17707 furnace is now ready for use.

NEXT GENERATION CALIBRATION APPARATUS FOR INDIUM AND TIN FIXED POINTS

INTRODUCTION

Before calibrating a standard platinum resistance thermometer, it is usual to check its water triple point resistance value and then to anneal the thermometer. The annealing temperature varies typically between 400 and 700°C.

DESCRIPTION

The new calibration apparatus developed by Isotech not only allows you to create and maintain the ITS 90 freezing points of Indium and Tin using a water heat-pipe, but also provides a second furnace for annealing thermometers at temperatures up to 700°C.

Complementary features of the apparatus are a pre-heating tube, maintained at the cell temperature, and a rack for storage of up to 4 thermometers at approximately ambient temperature.

METHOD OF OPERATION

1. By adjusting the set-point to a few degrees above the appropriate melting point, the cell, (tin or indium) is melted in the essentially temperature-gradient-free water-filled heat-pipe. When the cell's contents are completely melted, the heat-pipe temperature is re-adjusted to a level 0.5°C below the freeze temperature of the cell. When this set-point has been reached, a cold rod is introduced into the re-entrant tube of the cell to initiate the freeze, giving a plateau that can be maintained for between 12 and 24 hours. A modified technique is appropriate for tin, in the form of an applied thermal shock to the whole cell to counteract the tendency for a slow, large undercool to occur.

2. The thermometers are removed from their rack and, assuming they have already been annealed and are stable, are placed successively in the pre-warming tube for 5 minutes and then transferred to the cell for calibration. After 20 to 30 minutes the thermometer can be removed slowly from the cell and, after cooling to room temperature, the R_{TPW}-value can be checked.

SPECIFICATION

Dimensions of the cabinet containing the apparatus are:

Width:	600mm
Depth:	560mm
Height:	960mm (excluding thermometer storage rack)
Control:	The heat-pipe and the annealing furnace are controlled independently to a resolution of 0.1°C.
Communications:	RS422
Temperature range:	120°C to 250°C.
Over-temperature protection:	Provided on each furnace and operated by an independent sensor.
Supply:	2kW, 220-240V, 50/60Hz. (110-120V to special order)
Performance:	Essentially temperature-gradient-free heat-pipes provide the ideal apparatus for fixed point calibration and give a virtually perfect freezing profile within the fixed point cell. The addition of the second furnace enables thermometers to be stabilised before calibration, by annealing out strain and removing any 3D oxides. A comprehensive manual accompanies each apparatus.

Reproducibility of the temperatures of freezing indium or tin is typically ±0.1mK with Isotech fixed point cells.

HOW TO ORDER

Ask for Model 17707. We need to know your voltage and whether you require communications. A temperature equalising block is an available option (for comparison calibration purposes).

PLEASE NOTE: You may wish to purchase cells and thermometers with this apparatus. Both are available from Isotech. Next Generation Apparatus is also available for Zinc, Aluminium Silver and Copper fixed points. Please ask for data sheets 17705 and 17706.

THE POTASSIUM HEAT-PIPE FURNACE, MODEL 17706

FINAL PART OF THE COMMISSIONING INSTRUCTIONS

The initial slow temperature ramp is maintained until the temperature reaches 400°C.

Impose a heat-pipe set-point of 420°C and set the over-temperature controller to 450°C. Allow 1 to 2 hours for the furnace to stabilise and then reduce the over-temperature cut-out setting until the relay trips, thus switching off the furnace.

Allow the furnace to cool and isolate it from the electricity supply.

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 17706 furnace is now ready for use.

NEXT GENERATION CALIBRATION APPARATUS FOR ZINC, ALUMINIUM AND SILVER FIXED POINTS

INTRODUCTION

Using high temperature thermometers presents many problems due to strain and contamination, which can very easily be introduced into the thermometer during thermal cycling.

Limited understanding of the relevant mechanisms has resulted in a dearth of published information and of available apparatus for the safe treatment of these sophisticated devices.

DESCRIPTION

Sufficient information now exists at Isotech to allow introduction of a new apparatus, specifically designed not only to realise and maintain the ITS 90 fixed points of Zinc, Aluminium and Silver, but also to pre- and post-condition the thermometers to be calibrated. To this end a second furnace has been incorporated, which, because of its design, will permit conditioning to be carried out safely and without introducing contaminants into thermometers.

Complementary features of the apparatus are a pre-calibration tube (held at the temperature of the fixed point) made of a unique and gas tight material, together with a rack for storage of up to 4 thermometers at approximately ambient temperature.

METHOD OF OPERATION

1. By adjusting set-point to a few degrees above the appropriate melting point, the cell (zinc, aluminium or silver) is melted in an essentially temperature gradient free potassium-filled heat-pipe. When melting is complete, the heat-pipe temperature is readjusted to a level 0.5°C below the freeze temperature of the cell. When this set-point has been reached a cold rod is introduced into the re-entrant tube of the cell to initiate the freeze, giving a plateau that can be maintained for between 12 and 24 hours.

2. The thermometers are removed from their storage rack and placed in the pre-conditioning furnace. The furnace is slowly heated to 400°C (if the cell is Zinc), 650°C (for Aluminium) or 900°C (for Silver).

   During conditioning, thermometers are protected from contamination by a slow air flux around them.

   CAUTION: it is essential, when thermometers are inserted into furnaces, to keep their heads cool. This can be accomplished by blowing air, at ambient temperature, across them by means of a free-standing fan.

3. Thermometers are transferred individually to the cell for 20 to 30 minutes, for calibration, and then returned to the post-conditioning furnace.

4. When all the thermometers have been calibrated, the post-annealing furnace is slowly cooled to 450°C after which they can safely be exposed to room temperature and, thereafter, measured at the triple point of water.

   Throughout the time the thermometers are above 450°C the temperature changes are slow enough to prevent strain, and the slow flow of air prevents contamination at high temperatures.

SPECIFICATION

Dimensions of the cabinet containing the apparatus are:

Width:	600mm
Depth:	560mm
Height:	960mm (excluding thermometer storage rack)
Control:	The heat-pipe and the thermometer furnace are controlled independently to a resolution of 0.1°C.
Communications:	RS422
Over-temperature Protection:	Provided on each furnace and operated by an independent sensor.
Temperature range:	400 to 1000°C.
Power:	3kW, 110V, 50/60Hz
Performance:	Essentially temperature gradient-free heat-pipes provide the ideal apparatus for fixed point calibration and give a virtually perfect freezing profile within the fixed point cell. The addition of the second furnace, with its air-flow, enables thermometers to be easily and safely calibrated. A comprehensive manual accompanies each delivery.

THE SODIUM HEAT-PIPE FURNACE, MODEL 17705

FINAL PART OF THE COMMISSIONING INSTRUCTIONS

The initial slow temperature ramp is maintained until the temperature reaches 450°C. Impose a heat-pipe set-point of 500°C and set the over-temperature controller to 550°C. Allow 1 to 2 hours for the furnace to stabilise and then reduce the over-temperature cut-out setting until the relay trips and thus switching off the furnace.

Allow the furnace to cool and isolate it from the electricity supply.

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 overheated 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 17705 furnace is now ready for use.

N.B. The sodium heat-pipe incorporated in Model 17705 has a life of about 1000 hours at 1100°C, limited by the Inconel containment.

It should endure operation for at least 10,000 hours at 1000°C.

NEXT GENERATION CALIBRATION APPARATUS FOR ALUMINIUM, SILVER AND COPPER FIXED POINTS

INTRODUCTION

Using high temperature thermometers presents many problems due to strain and contamination which can very easily be induced into the thermometer during temperature cycling.

Limited understanding of the relevant mechanisms has resulted in a dearth of published information and of available apparatus for the safe treatment of these sophisticated devices.

DESCRIPTION

Sufficient information now exists at Isotech to allow introduction of a new apparatus, specifically designed not only to realise and maintain the ITS 90 fixed points of Aluminium, Silver and Copper, but (housed in the same facility) incorporating a second furnace which, because of its design, can be used safely and without contamination, to pre-heat and anneal the thermometers for calibration.

Complementary features of the apparatus are a pre-calibration tube (held at the temperature of the fixed point) made of a unique and gas tight material, together with a rack for storage of up to 4 thermometers at approximately ambient temperature.

METHOD OF OPERATION

CAUTION: it is essential, when thermometers are inserted into furnaces, to keep their heads cool. This can be accomplished by blowing air, at ambient temperature, across them by means of a free-standing fan.

1. By adjusting the set-point to a few degrees above the appropriate melting point, the cell (aluminium, silver or copper) is melted in an essentially temperature-gradient-free sodium-filled heat-pipe. When melting is complete, the heat-pipe temperature is re-adjusted to a level 0.5°C to 1°C below the freeze temperature of the cell. When this temperature has been reached a cold rod is introduced into the re-entrant tube of the cell to initiate the freeze, giving a plateau that can be maintained for between 12 and 24 hours.

2. The thermometers are removed from their storage rack and placed in the pre-heating furnace. The furnace is slowly heated to 600°C (if the cell is Aluminium) and 900 or 950°C (if the cell is Silver or Copper).

   During conditioning, thermometers are protected from contamination by a slow air flux around them.

3. Thermometers are transferred individually to the cell for 20 to 30 minutes, for calibration and then returned to the annealing furnace.

4. When all the thermometers have been calibrated and have been returned to the annealing furnace, they are slowly cooled to 450°C at which stage they can safely be exposed to room temperature and, when cool, measured at the triple point of water. Throughout the time the thermometers are above 450°C temperature changes are slow, which prevents strain and the flow of air prevents contamination at high temperatures.

SPECIFICATION

Dimensions of the cabinet containing the apparatus are:

Width:	600mm
Depth:	560mm
Height:	960mm (excluding thermometer storage rack)
Control:	The heat-pipe and the thermometer furnace are controlled independently to a resolution of 0.1°C.
Communications:	RS422
Over-temperature Protection:	Provided on each furnace and operated by an independent sensor.
Temperature range:	450 to 1100°C.
Power:	3kW, 220-240V, 50/60Hz (110-120V to special order)
Performance:	Essentially temperature-gradient-free heat-pipes provide the ideal apparatus for fixed point calibration and give a virtually perfect freezing profile within the fixed point cell. The addition of the second furnace with its air-flow enables thermometers to be easily and safely calibrated. A comprehensive manual accompanies each delivery.

ELIMINATING CONTAMINATION DURING THE CALIBRATION OF HIGH TEMPERATURE THERMOMETERS

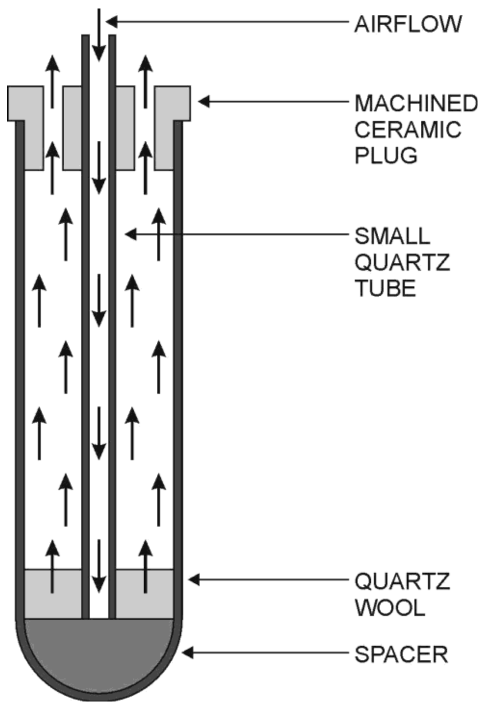
It is quite feasible to heat a high temperature thermometer to 1000°C and return it to ambient temperature without altering the water triple point resistance value by more than 0.5mK equivalent.

Instructions in the Isotech 962 and 96178 thermometer manuals explain how to handle and keep clean the thermometers. It is imperative that procedures are followed because contaminants can penetrate and pass through physically-intact quartz sheaths at temperatures above 800°C.

To prevent this happening, a secondary containment has been provided, in kit form that can be fitted to dual furnace Models 17705 and 17706.

The completed assembly is depicted below:

ANNEALING COMPONENT OF THE DUAL FURNACE SHOWING AIR FLOW



Air percolates through the quartz wool, oxidising any pollutants and rendering them harmless and unable to penetrate the quartz sheath of the thermometer.

The combination of air-flow and positioning ensures that thermometers will not become contaminated at high temperature.

The dual furnace also has a pre-calibration tube made of silicon carbide, which justifies its high cost by remaining impervious to all gases that would poison the high temperature thermometer. The pre-calibration tube can be used not only to store each thermometer immediately prior to calibration but also to house the standard thermometer used to monitor the cell's plateau at various stages in a calibration schedule.

Optional equipment includes an equalising block that fits in the heat-pipe so that the furnace can be used as a gradient-free comparison facility. Please note that the temperature-equalising block is for use up to 700°C only. Above 700°C the sensors to be calibrated may be immersed directly into the heat-pipe without using an equalising block.

IMPORTANT NOTE: Since the calibration of sensors at high temperatures can be fraught with difficulties, if any doubt exists, please consult Isotech for advice before embarking on any exercise in which there is a risk of contamination of the sensors you are trying to calibrate.

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-hand.

Therefore, we now prefer to work differently. With international communications so good these days, if anything goes wrong with the apparatus or you need any other after sales service, just contact Isotech or the agent from whom you purchased the apparatus. On our help-line we are anxious to serve you and will swiftly be able to help you solve your problem, or deal with a technical enquiry.

SPARE PARTS

Chopped Bulk Fibre (kg)	932-20-18
32A Fuse A1	935-12-48
A2 Black Fuse Holder	935-11-09
45 amp Opto Relay	935-21-19
110V Contactor	935-21-18
110V Rocker Switch	935-27-02B
N Thermocouple	935-14-42
White Fuse Holder	935-11-10
Inconel Cell Basket	410-03-00A
Ceramic Bricks	935-29-02
Silicon Carbide Tube (Sodium/Potassium)	935-29-20
Pre-heat Tube (Water)	420-03-22

CELL HANDLING

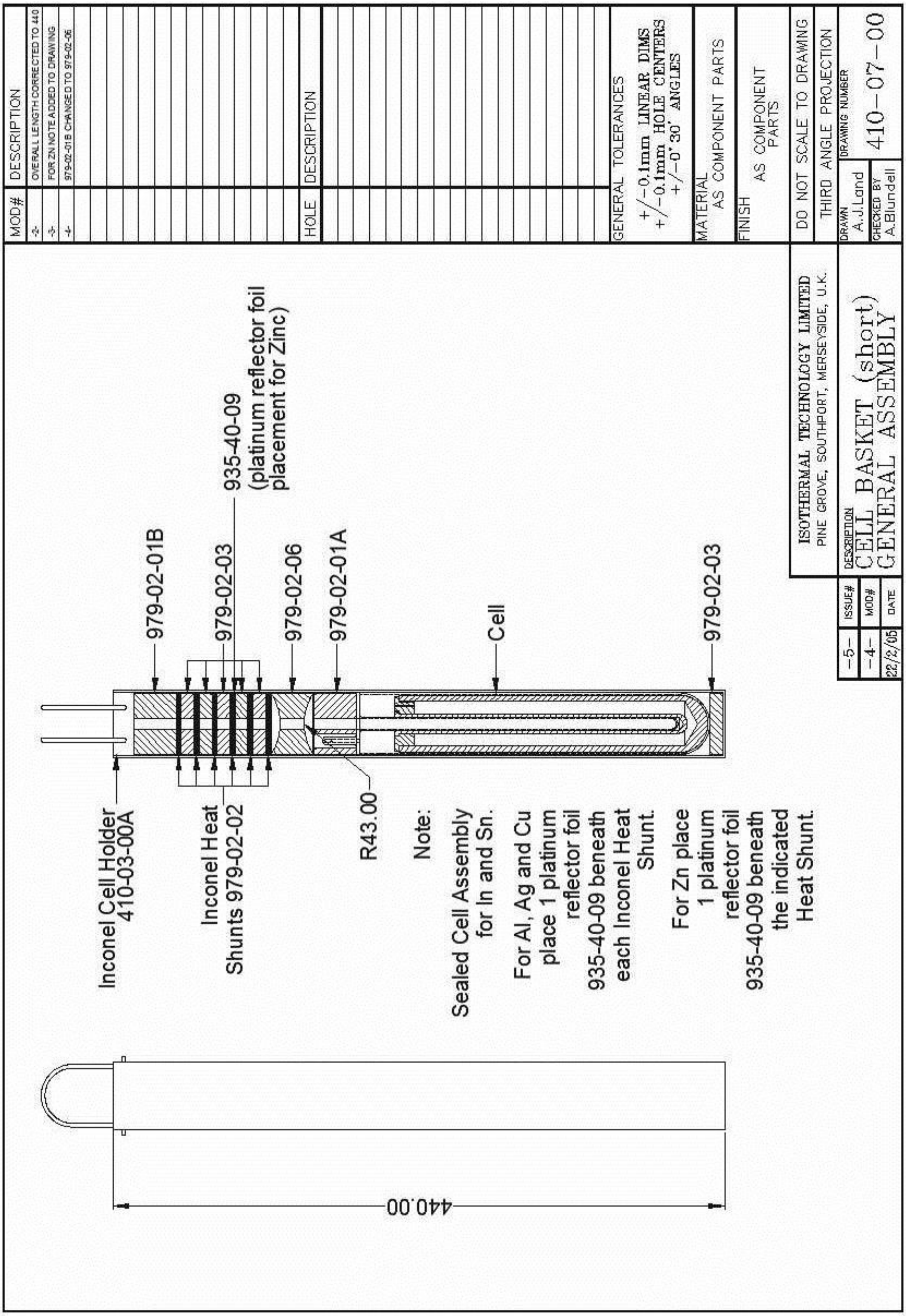
In order to facilitate introduction to, and removal from the furnace, Isotech can provide for each cell, supplementary equipment largely comprising an Inconel basket with detachable handle.

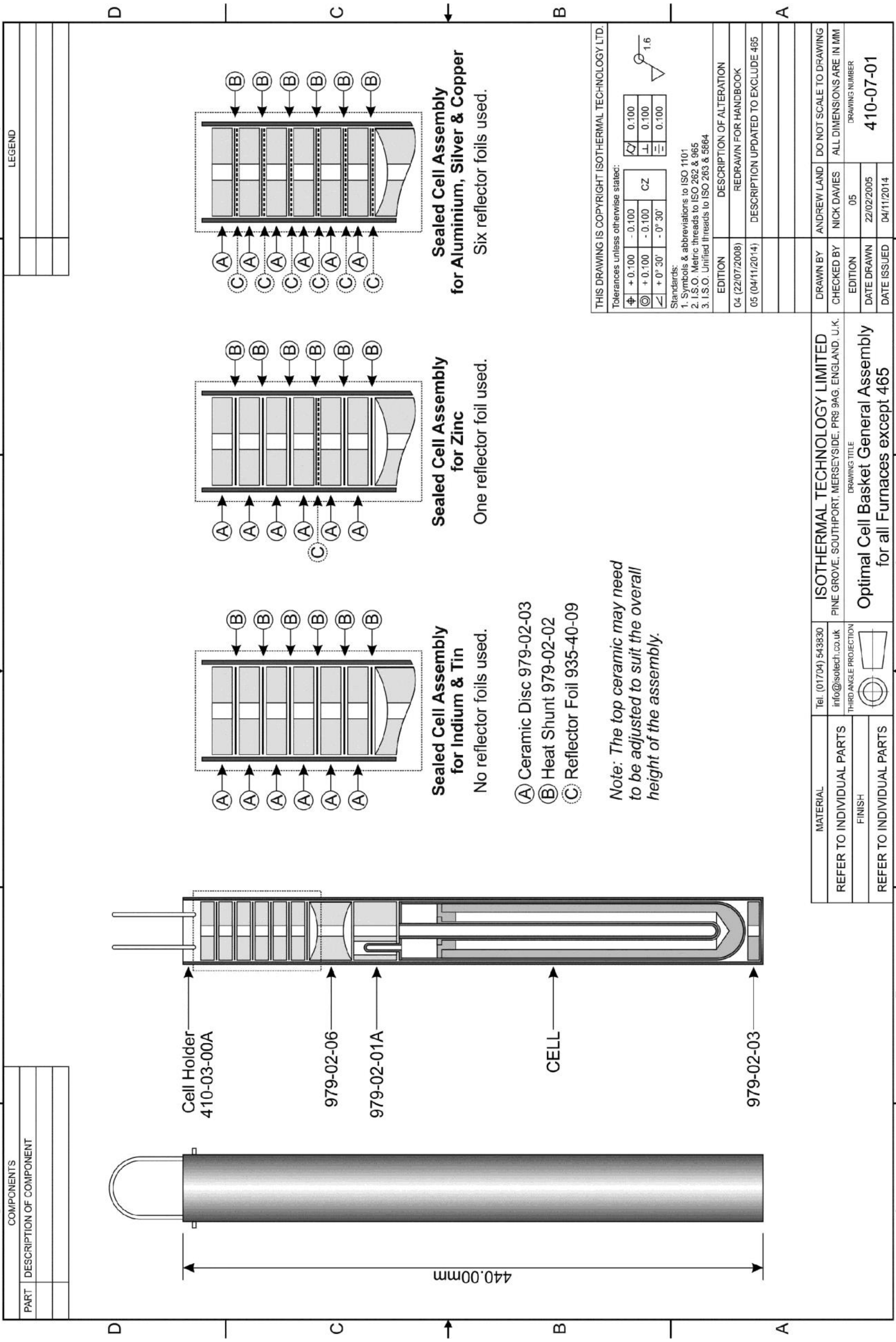
To prevent the cell-surface becoming discoloured, it is recommended that, before using the cell, the basket and insulation be placed in the furnace and the furnace be taken to above the cell working temperature for at least 2 hours. This operation outgases the basket and insulation, which may smoke and 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.

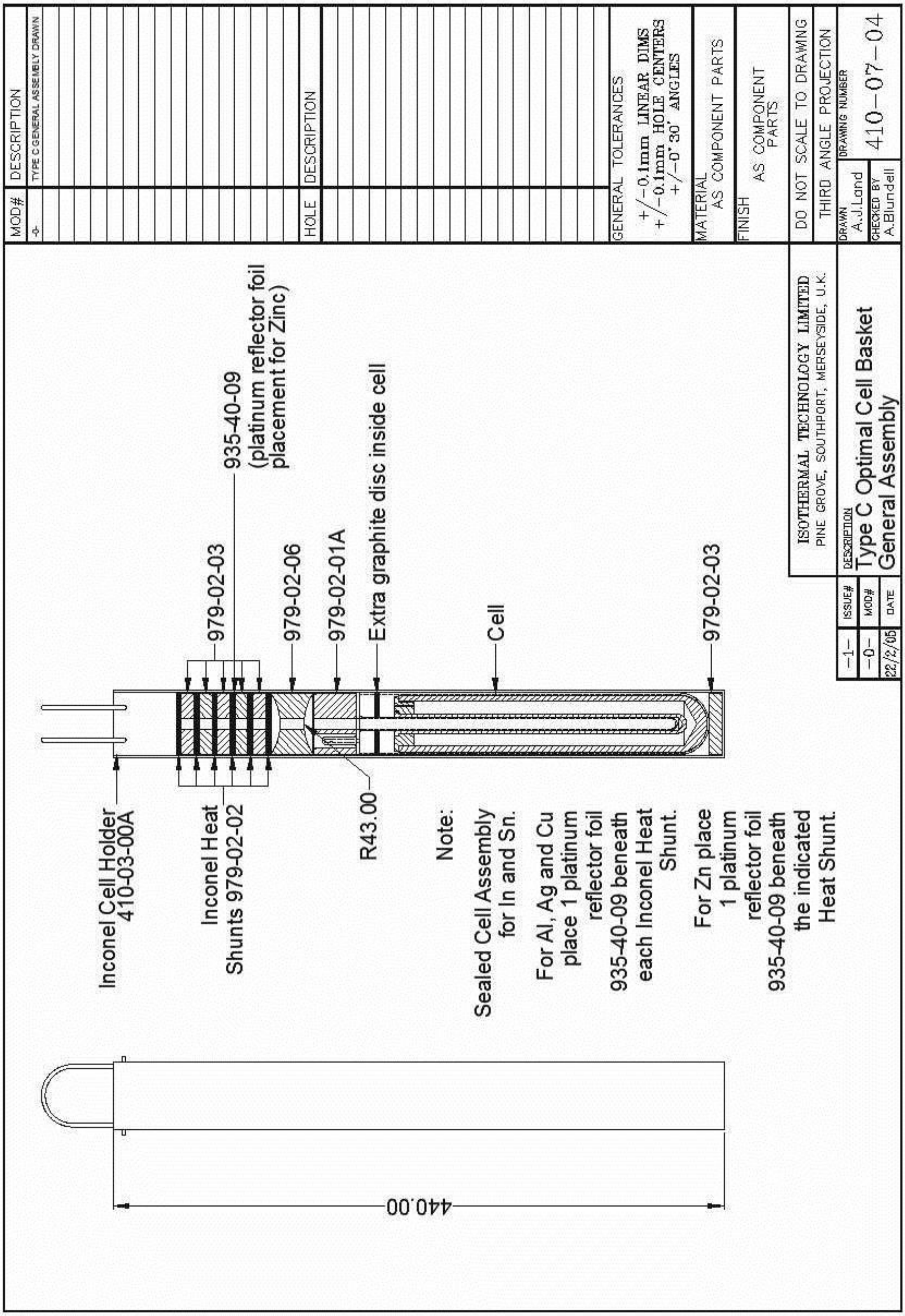
CELL KIT

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.


MOD#
DESCRIPTION
-2- OVERALL LENGTH CORRECTED TO 440
-3- FOR ZN NOTE ADDED TO DRAWING
-4- 979-02-01B CHANGED TO 979-02-06
HOLE
DESCRIPTION
GENERAL TOLERANCES
+/-0.1mm LINEAR DIMS
+/-0.1mm HOLE CENTERS
+/-0' 30' ANGLES
MATERIAL
AS COMPONENT PARTS
FINISH
AS COMPONENT PARTS
DO NOT SCALE TO DRAWING
THIRD ANGLE PROJECTION
DRAWN A.J.Land
CHECKED BY A.Blundell
DRAWING NUMBER 410-07-00
ISOTHERMAL TECHNOLOGY LIMITED
PINE GROVE, SOUTHPORT, MERSEYSIDE, U.K.
DESCRIPTION
CELL BASKET (short)
GENERAL ASSEMBLY
-5- ISSUE#
-4- MOD#
22/2/05 DATE
979-02-01B
979-02-03
935-40-09
(platinum reflector foil placement for Zinc)
979-02-06
979-02-01A
Cell
979-02-03
Inconel Cell Holder 410-03-00A
Inconel Heat Shunts 979-02-02
R43.00
Note:
Sealed Cell Assembly for In and Sn.
For Al, Ag and Cu place 1 platinum reflector foil 935-40-09 beneath each Inconel Heat Shunt.
For Zn place 1 platinum reflector foil 935-40-09 beneath the indicated Heat Shunt.
440.00


Sealed Cell Assembly for Indium & Tin
No reflector foils used.
Sealed Cell Assembly for Zinc
One reflector foil used.
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.
Cell Holder 410-03-00A
979-02-06
979-02-01A
CELL
979-02-03
440.00mm
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
04 (22/07/2008)
REDRAWN FOR HANDBOOK
05 (04/11/2014)
DESCRIPTION UPDATED TO EXCLUDE 465
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 all Furnaces except 465
DRAWN BY
ANDREW LAND
CHECKED BY
NICK DAVIES
EDITION
05
DATE DRAWN
22/02/2005
DATE ISSUED
04/11/2014
DO NOT SCALE TO DRAWING
ALL DIMENSIONS ARE IN MM
DRAWING NUMBER
410-07-01



440.00
Inconel Cell Holder 410-03-00A
Inconel Heat Shunts 979-02-02
R43.00
Note:
Sealed Cell Assembly for In and Sn.
For Al, Ag and Cu place 1 platinum reflector foil 935-40-09 beneath each Inconel Heat Shunt.
For Zn place 1 platinum reflector foil 935-40-09 beneath the indicated Heat Shunt.
979-02-03
935-40-09 (platinum reflector foil placement for Zinc)
979-02-06
979-02-01A
Extra graphite disc inside cell
Cell
979-02-03
MOD# DESCRIPTION
-0- TYPE C GENERAL ASSEMBLY DRAWN
HOLE DESCRIPTION
GENERAL TOLERANCES
+/-0.1mm LINEAR DIMS
+/-0.1mm HOLE CENTERS
+/-0°30' ANGLES
MATERIAL AS COMPONENT PARTS
FINISH AS COMPONENT PARTS
DO NOT SCALE TO DRAWING
THIRD ANGLE PROJECTION
DRAWN A.J.Land
CHECKED BY A.Blundell
DRAWING NUMBER 410-07-04
ISOTHERMAL TECHNOLOGY LIMITED
PINE GROVE, SOUTHPORT, MERSEYSIDE, U.K.
DESCRIPTION Type C Optimal Cell Basket General Assembly
ISSUE# -1-
MOD# -0-
DATE 22/2/05

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 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 2 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. 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 FOR CELL OPERATION
	°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	A11/50 or B11/50	ITL-M-18233
GALLIUM MP	29.7646	302.9146	-2.0	-1.2	ITL-M-17401	ITL-M-17402A
INDIUM FP	156.5985	429.7485	+4.9	+3.3	ITL-M-17668	ITL-M-17701/3/4/7
TIN FP	231.928	505.078	+3.3	+2.2	ITL-M-17669	ITL-M-17701/3/4/7
ZINC FP	419.527	692.677	+4.3	+2.7	ITL-M-17671	ITL-M-17701/2*/3/6
ALUMINIUM FP	660.323	933.473	+7.0	+1.6	ITL-M-17672	ITL-M-17702**/5/6
SILVER FP	961.78	1234.93	+6.0	+5.4	ITL-M-17673	ITL-M-17702**/5/6

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-microsporous. 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. 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 melt 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 on subscription.

FIGURE 1; SECTION THROUGH DUAL FURNACE

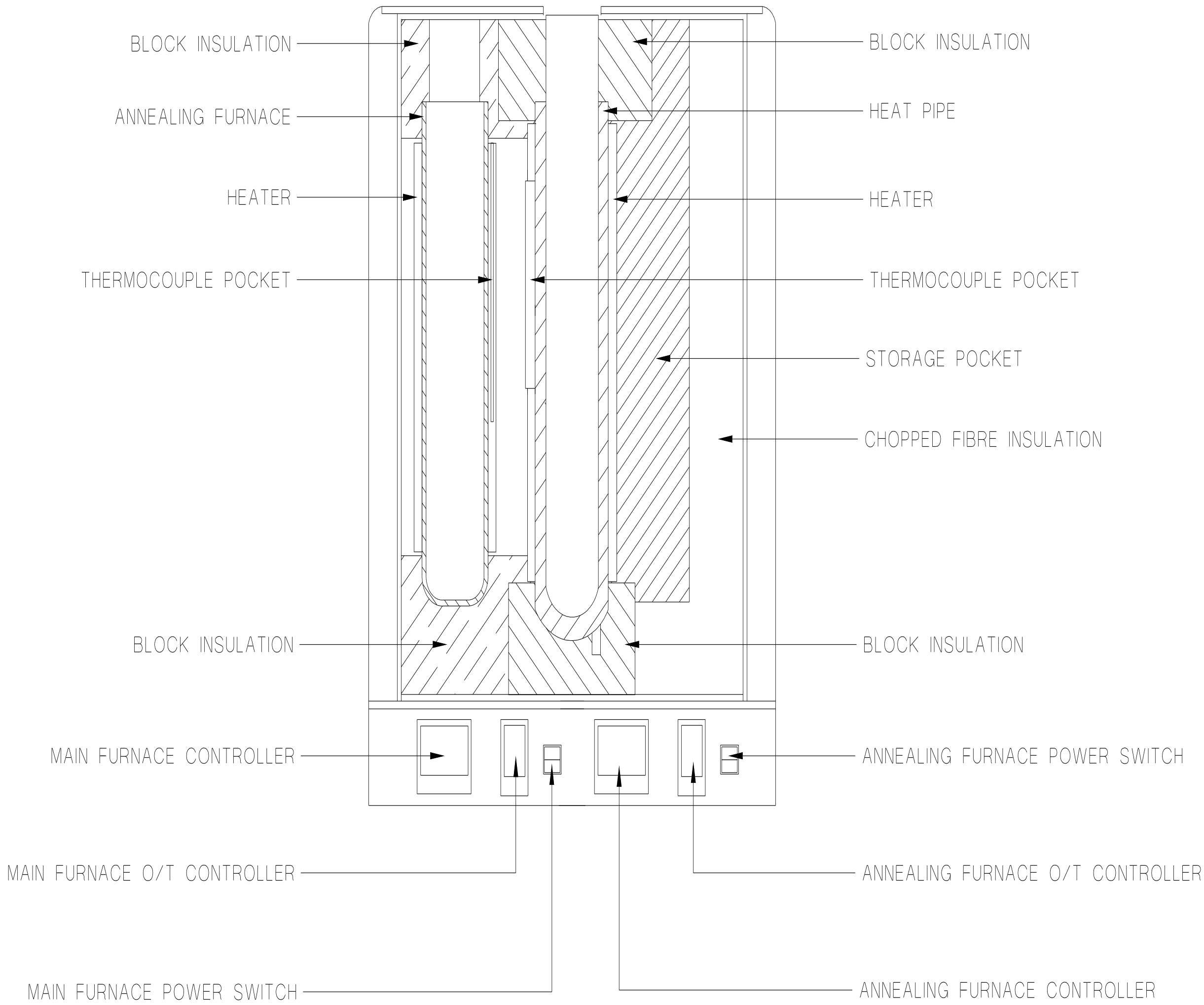
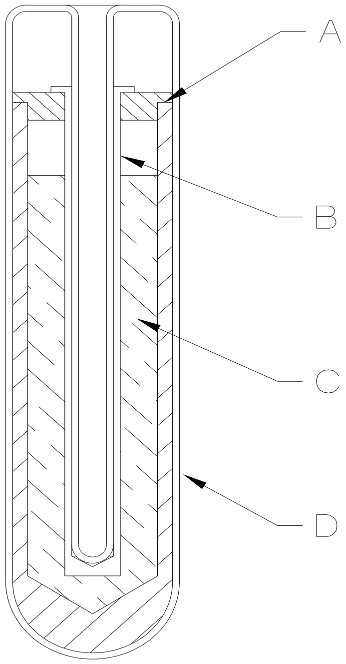


FIGURE 2; SEALED METAL FREEZING POINT CELL



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 3; SEALED METAL FREEZING POINT CELL BODY DIMENSIONS