OBERON 426

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

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.

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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 handbook)
	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).

THIS DEVICE SHOULD BE USED WITH AN ISOLATING TRANSFORMER TO REDUCE LEAKAGE CURRENT AT HIGH TEMPERATURES.

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.

Environmental Ratings

Operating temperature	5-50°C
Relative humidity	5-95%, non condensing

⚠ HEALTH AND SAFETY INSTRUCTIONS

1. Read this entire handbook 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.

UNPACKING AND INITIAL INSPECTION

Our Packing Department uses custom designed packaging to send out your unit, but as accidents can still happen in transit, you are advised, after unpacking the unit, to inspect it for any sign of shipping damage, and confirm that your delivery is in accordance with the packing note. If you find any damage or that part of the delivery is missing, notify us or our agent, and the carrier immediately. If the unit is damaged you should keep the packing for possible insurance assessment.

ELECTRICITY SUPPLY 

Before connecting to the electricity supply please familiarise yourself with the parts of the handbook relevant to your model.

The apparatus is provided with an approved power cord. If the plug is not suitable for your location then the plug should be removed and replaced with an appropriate plug.

Take care to ensure the old plug is disposed safely.

The cable is colour coded as follows:

COLOUR	FUNCTION
Green/yellow	Earth (Ground)
Brown	Live (line)
Blue	Neutral

Please ensure that your unit is correctly connected to the electricity supply.

THE APPARATUS MUST BE CORRECTLY EARTHED (GROUNDED)

IT IS RECOMMENDED THAT AN ISOLATING TRANSFORMER IS USED. This prevents problems with earth leakage currents at high temperatures.

⚠ CAUTIONARY NOTE

ISOTECH PRODUCTS ARE INTENDED FOR USE BY TECHNICALLY TRAINED AND COMPETENT PERSONNEL FAMILIAR WITH GOOD MEASUREMENT PRACTICES.

IT IS EXPECTED THAT PERSONNEL USING THIS EQUIPMENT WILL BE COMPETENT WITH THE MANAGEMENT OF APPARATUS WHICH MAY BE POWERED OR UNDER EXTREMES OF TEMPERATURE, AND ARE ABLE TO APPRECIATE THE HAZARDS WHICH MAY BE ASSOCIATED WITH, AND THE PRECAUTIONS TO BE TAKEN WITH, SUCH EQUIPMENT.

SPECIFICATION

Temperature Range:	450°C to 1090°C
Emissivity:	Greater than 0.995
Stability:	±0.05°C
Display resolution:	0.1°C to 1100.0
Cavity Size:	50mm diameter 300mm deep
Communications:	Supplied as standard with serial interface, PC adaptor cable and Cal Notepad
Supply:	110VAC, 1kW, 50/60Hz (230VAC Isolating Transformer available)
Dimensions:	Height - 490mm Width - 415mm Depth - 280mm
Weight:	30kgs approximately

THE OBERON FURNACE

The Oberon furnace for realisation of metal freezing points is based on the heat-pipe. It is furnished with two independent control systems, one for temperature control and the other for over-temperature protection.

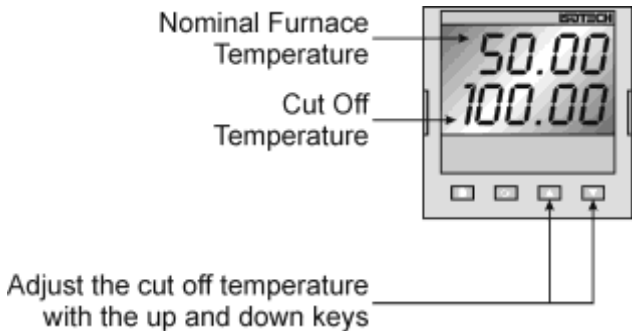
An over-temperature cut-off controller is fitted. Its purpose is to establish an upper temperature limit after which all systems power will be shut down to protect the system from the effects of possible operating controller failure.

In use:

a. The over-temperature controller should be set at a temperature approximately 50°C higher than the operating controller setting.

b. To turn on system power at start up (or as required), it is necessary to actuate the front panel off/on switch and also to depress the reset button on the front of the over-temperature protection controller.

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

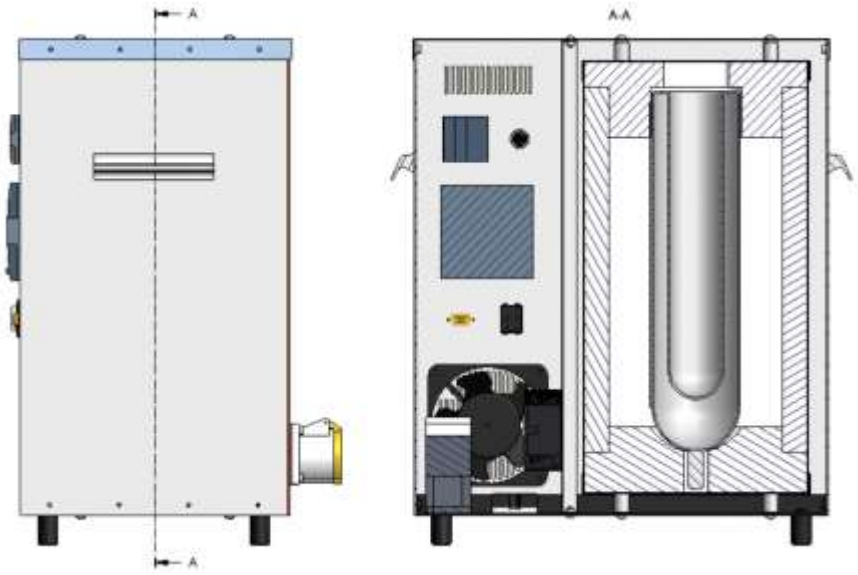


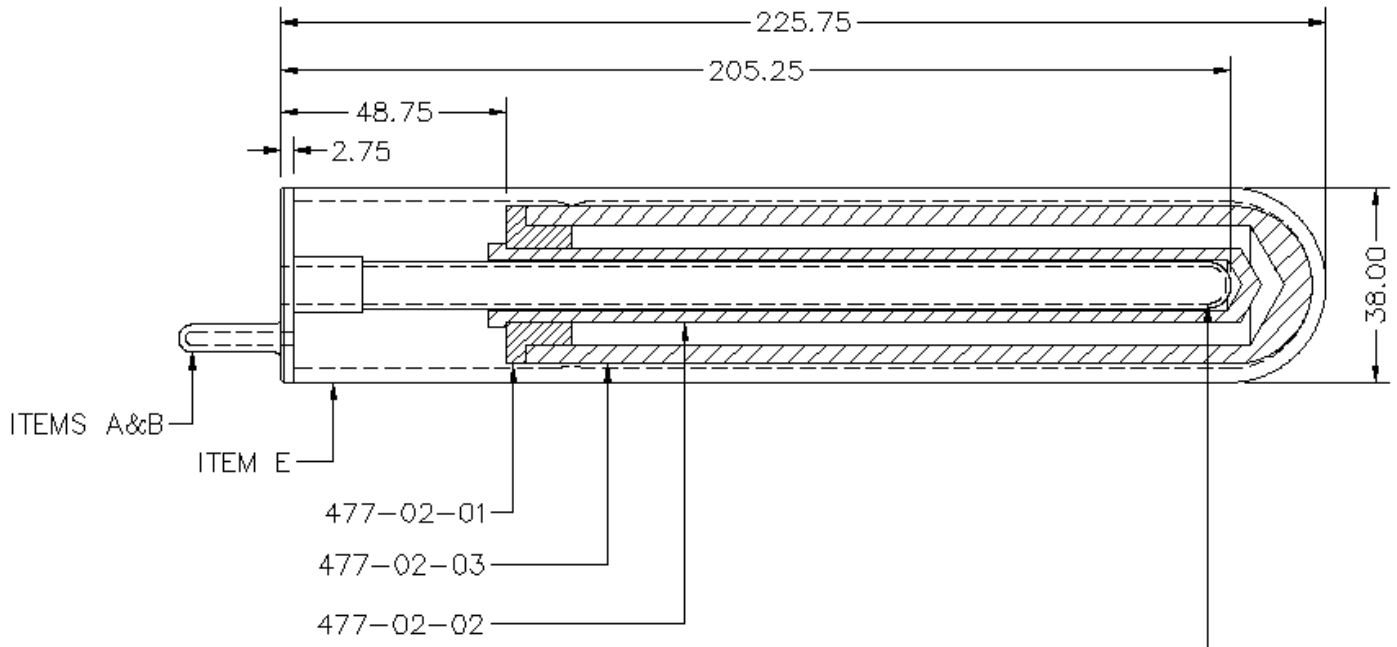
A section through the sealed type of metal freezing-point cell used in the furnace is shown on drawing 477-00-00.

Figure 1 shows a sectional view of front and side through the furnace and case (not to scale).

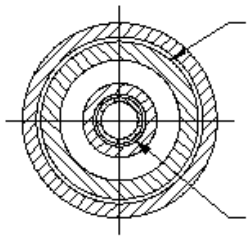
The furnace is equipped with a thermometer pre-heating tube.

Figure 1





SECTION A–A



Note 1:
When cell is placed horizontally the graphite components may touch the quartz at Surface 2 but not at Surface 1.
When slightly inclined the graphite should move along the quartz assembly.

Note 2:
Quartz tubing sized to permit only 1 to 3mm movement of the graphite Cell along the Quartz assembly.

Notes:
All dimensions are in mm.
Refer to individual drawings for Tolerances

MOD#	DESCRIPTION
–0–	PREVIOUSLY DRAWING NUMBER 978SC
–1–	THICKER TUBE ADDED BELOW FLANGE
–2–	ALTERED TO SUIT MANUFACTURE

HOLE	DESCRIPTION

GENERAL TOLERANCES
ISO DIN 2768 'C'

MATERIAL
SEE INDIVIDUAL DRAWINGS

FINISH
SEE INDIVIDUAL DRAWINGS

DO NOT SCALE TO DRAWING

THIRD ANGLE PROJECTION

ISOTHERMAL TECHNOLOGY LIMITED
PINE GROVE, SOUTHPORT, MERSEYSIDE, U.K.

–3–	ISSUE#
–2–	MOD#
18/07/06	DATE

DESCRIPTION
GENERAL ASSEMBLY
SLIM SEALED CELLS

DRAWN A.J.Land
CHECKED BY J.Tavener

DRAWING NUMBER
477–00–00

ISOTECH

INSTRUCTIONS FOR OBERON FIXED POINT CELLS

It is assumed that the user is familiar with fixed point calibration and the problems associated with handling graphite at high temperatures.

If not, steps should be taken to obtain training. The inexperienced user may inadvertently, permanently damage the cells he is using otherwise.

CELL HANDLING

In order to facilitate introduction to, and removal from, the furnace, Isotech provides, for each cell, supplementary equipment largely comprising an Inconel basket.

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 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.

Before use, the cell should be thoroughly cleansed with alcohol and dried thoroughly with tissue. Once cleaned the cell should not be handled with bare hands.

Careful cleaning of quartz glassed cells is especially important for Aluminium, Silver and copper, as devitrification of the quartz can occur very easily at these elevated temperatures.

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 a diametrically-pivoted handle if one is used. The handle will need to be maintained in a non-vertical plane while being used for removing and replacing the assembly.

CELL KIT

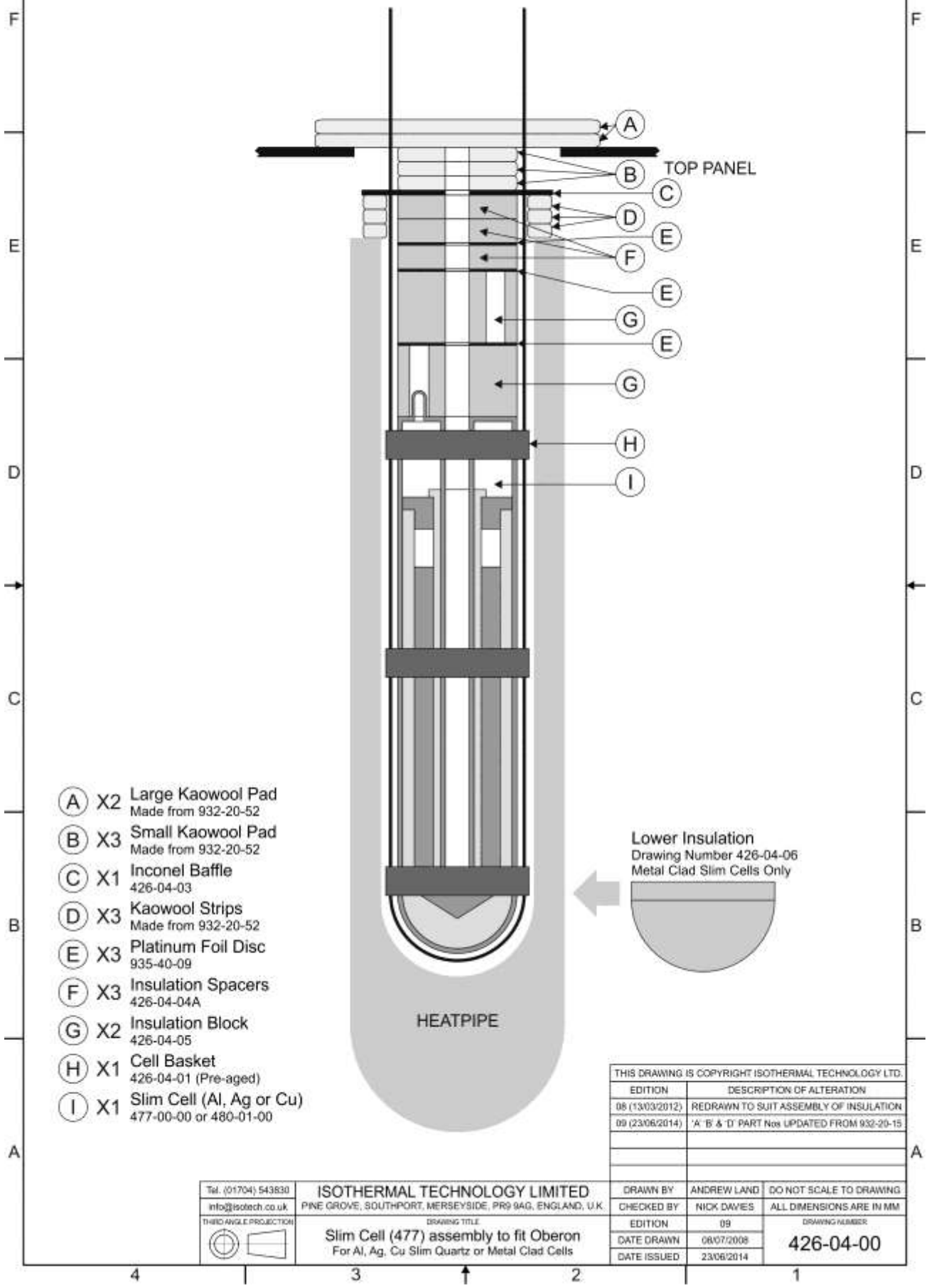
Basket, discs of ceramic insulation and platinum foil discs along with an inconel baffle.

Drawing 426-04-00 shows the recommended assembly of the cell basket and insulation discs.

It is most convenient and because of the thermal properties of the Oberon more accurate to use the melt curve of the fixed point cell.

If cells are to be used in the freeze mode, please consult the slim cell handbook for details of the techniques involved.

As a guide and help to the user a procedure follows which describes a slim cell calibration facility using Medusa and an Oberon furnace to calibrate thermometers.



(A) X2 Large Kaowool Pad
Made from 932-20-52

(B) X3 Small Kaowool Pad
Made from 932-20-52

(C) X1 Inconel Baffle
426-04-03

(D) X3 Kaowool Strips
Made from 932-20-52

(E) X3 Platinum Foil Disc
935-40-09

(F) X3 Insulation Spacers
426-04-04A

(G) X2 Insulation Block
426-04-05

(H) X1 Cell Basket
426-04-01 (Pre-aged)

(I) X1 Slim Cell (Al, Ag or Cu)
477-00-00 or 480-01-00

EDITION	DESCRIPTION OF ALTERATION
08 (13/03/2012)	REDRAWN TO SUIT ASSEMBLY OF INSULATION
09 (23/06/2014)	'A' 'B' & 'D' PART Nos UPDATED FROM 932-20-15

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THIRD ANGLE PROJECTION

DRAWING TITLE
Slim Cell (477) assembly to fit Oberon
For Al, Ag, Cu Slim Quartz or Metal Clad Cells

DRAWN BY	ANDREW LAND	DO NOT SCALE TO DRAWING
CHECKED BY	NICK DAVIES	ALL DIMENSIONS ARE IN MM
EDITION	09	DRAWING NUMBER
DATE DRAWN	08/07/2008	426-04-00
DATE ISSUED	23/06/2014	

PROCEDURE FOR THE CALIBRATION OF PRT'S USING SLIM FIXED POINT CELLS AND A TTI 2 BRIDGE (AS USED IN 1999)

SCOPE

This procedure is used to calibrate PRT's and SPRT's over the range encompassing the fixed points of In, Sn, Zn and Al.

Due to the depth of immersion available (140mm within the cell beneath the metal ingot) and the consequent problems of stem conduction, only PRT's of suitable construction can be calibrated, such as the Isotech 935-14-95 and 935-14-72 type of thermometer and of course all types of thermocouple.

The operator should be conversant with the procedures regarding the handling and use of sealed fixed point cells. The fixed points are used on their melt plateaux rather than the freeze, since this is easier to achieve and maintain.

For use of any laboratory equipment not detailed in this procedure, the appropriate manual or procedure should be consulted.

PRT CONSTRUCTION

As mentioned above PRT's should have a suitable internal construction of platinum leads such as the Isotech models 935-14-95 and 935-14-72 or 909 SPRT's. Their length should be at least 300mm long and no more than 8mm diameter. For PRT's of unknown construction immersion tests as detailed later should be carried out over the calibrated range.

EQUIPMENT

There are three Medusa furnaces two of which are dedicated to maintaining the fixed points of In, Sn and Zn. The third Medusa is used as an annealing furnace to maintain thermometers previously calibrated at Al (660.323°C), at 450°C upon withdrawal from the cell.

An Oberon heat pipe furnace is used to maintain the Aluminium fixed point. This ramps up to 450°C when switched on, dwells at 450°C, until the controller is adjusted to 2°C above the Al fixed point.

FURNACE ASSEMBLY AND HANDLING OF QUARTZ SHEATHED CELLS

The standard cells are presently stored in a suitable cell rack within the Northern Temperature Primary Laboratory. Prior to use, each quartz-sheathed cell is first cleaned with Ethanol to remove any surface contamination and so prevent devitrification of the quartz glass outer. When handling the cells direct contact with the quartz glass envelope should be avoided or rubber gloves worn to prevent this. The cell is then mounted within a slim fixed point cell inconel basket assigned to the cell in question, which contains a small wad of kaowool at the bottom to prevent mechanical damage to the cell. Similarly, there should be a small wad of quartz wool in the bottom of the re-entrant tube of the cell to minimise physical damage to the cell during insertion of PRT's. Both the inconel basket and kaowool pad should have been pre-fired at 700°C prior to use to remove any possible contamination.

In the case of the Aluminium point, the inconel basket is of a different design so that it fits into the Oberon well with a greater depth of immersion to minimise stem conduction. The basket is constructed of inconel wire held together with inconel rings. Insulating ceramics are then arranged above each cell, see drawing 426-04-00.

The cell and inconel basket are then mounted into the appropriate furnace. A monitor SPRT appropriate for the temperature to be measured is then cleaned with Ethanol and inserted into the re-entrant tube of the cell to be melted.

The temperature controller is then set to:

1.5°C above the fixed point temperature for In and Sn and
2°C above the fixed point for Zn and Al.

Following this the top of each cell is further insulated with Quartz wool on top of the furnace, to prevent excess loss of heat to the environment.

The SPRT is connected to the TTI 2 Resistance Bridge and the output in °C monitored. The output is also recorded on the computer software package "TTI 2 measurement program" which is configured to record °C in graphical format and also to datalog the results to a data file. It is advantageous to record the cell reaching the melt plateau but not essential, however at least 10 minutes of stable temperature (ie. no more than ±3mK) should be obtained before calibration of PRT's begins. The actual temperature as recorded by the SPRT should agree with the ITS-90 value assigned to that fixed point to ±10mK.

A print out of the temperature graph and appropriate section of the datalog file should be obtained and retained for records. The monitor thermometer may now be removed and a PRT for calibration (pre-warmed to approximately 50°C below the fixed point to be used) inserted into the fixed-point cell.

Once calibrations have been performed on a maximum of 3 PRT's at any one time, the SPRT must be inserted again to determine the cell temperature. The mean of the two readings from the SPRT is then used as the actual temperature of the cell and each should agree with the other to ±5mK.

CALIBRATION OF PRTS

PRT's for calibration should be inserted into each fixed-point cell, having first been pre-warmed to a temperature approximately 50°C below the fixed point to be used. This "cool" insertion helps to maintain the melt plateau for a longer period of time since it freezes a little of the cell with each insertion. The pre-warming minimises thermal shock to the cell. At present the PRT's are pre-warmed in the annealing medusa but future Medusa furnaces will have pre-warming tubes as part of their construction for this purpose.

If the PRT is of short construction it should be fan cooled at the top during calibration, especially at the Aluminium and Zinc temperatures.

The PRT is connected to the TTI 2 and "no sensor calibration" selected. The output in ohms in the statistics mode is selected and outputted to the computer as before, except resistance is now recorded on the graph. During calibration at least 20 minutes data logging with less than ±5mK for semi standards and ±2mK for SPRT's, should be obtained before the final output in mean ohms is recorded.

For In, Sn and Zn following calibration the PRT may be removed directly from the cell to ambient and cooled in a vertical position.

For PRT's being calibrated at Al these should be slowly removed from the cell and carefully placed in the annealing Medusa furnace which has been pre-heated to 450°C. These PRT's should be left for at least ½ an hour in this furnace, with their heads fan cooled, before removal to ambient temperature. This anneals the platinum element.

IMMERSION TESTS

Immersion tests for the monitor SPRT used in each fixed point cell to verify the temperature should be carried out each 3 months and the results recorded in the slim cell fixed point file on the appropriate table. The reason for this test is to verify the integrity of the system and the repeatability of the SPRT.

To do this the SPRT once stabilised on the melt plateau should be withdrawn 3cm at 1cm intervals from the cell to be tested and the °C output should not change by more than ±5mK. If any more than this is found an investigation into the cause should be conducted. When returned to the full immersion in the cell, the SPRT should read the same temperature ±0.5mK, which it did at the start of the test.

PRT's of unknown internal construction for calibration should also be tested in this manner to assess the effects of stem conduction on the results. For semi standard PRT's the change in °C should not be in excess of ±8mK and ±5mK for SPRT's. If the results are observed to be more than this a revised uncertainty for the PRT should be calculated to take this factor into account.

CALIBRATION DATA FORMAT

The ohms recorded for each (S)PRT will be coupled with the actual recorded temperature of the fixed point cell as monitored by the laboratory SPRT for that day. For example, the ITS-90 assigned fixed point temperature will not be used if the actual measured temperature of the Zn was found to be 419.525°C, the latter temperature will be used.

FOLLOWING CALIBRATION

Once calibration is complete, each Medusa furnace may be switched off but must be left connected to the mains electricity supply to allow the internal fans of the furnace to completely cool the unit. For the Oberon unit, the "set point 1" (SP1) should be manually reduced to 450°C prior to switching off (so that during subsequent use it will not ramp directly up to 660°C instead of dwelling at 450°C first) Again this unit should be left connected to the mains to allow the internal fans to continue cooling.

FAULT-FINDING AND AFTER-SALES SERVICE

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 phone or fax Isotech (or the agent from whom you purchased the apparatus). On our helpline we are anxious to serve you and will swiftly be able to help you solve your problem, or deal with a technical enquiry.

SERVICING THE OBERON TEMPERATURE FURNACE

No regular servicing is required.

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.

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.

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, quartz glass has a softening (annealing) temperature of 1050°C, some 35°C below the Copper melt point. A user should therefore not be surprised if his Copper cell begins to devitrify at these elevated temperatures.
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 cells should be returned to Isotech for a new quartz cover.

Most 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 cell and well may be cleaned with copious isopropanol and dried with a tissue. 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 melts 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.

APPENDIX 1: AIR COOLING

The Isotech Oberon Furnace has an air cooled operation to maintain a low case temperature. The air ventilation ducts should be not be obstructed at any time. Doing so will cause the case temperature to rise and may cause irreparable damage to internal components.

The Oberon uses very high efficiency insulation and will keep its temperature for long periods after powering down. After the setpoint is reduced, allow time for the heat pipe to reduce in temperature before powering down.

APPENDIX 2: NOTE ON THERMOMETER USE AT HIGH TEMPERATURE

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 annealed at 480°C for 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.

APPENDIX 3: NOTE ON HEAT-PIPE OPERATION

The principle of the operation of heat pipes is simple. The inside walls of the inner and outer tubes are covered with a fine wire mesh screen. When heated, the small amount of sodium with which the heat pipe is charged vaporises throughout the interior cavity and condenses on the walls of the cavity. Assuming that the interior is isobaric, the vapour-liquid transition at the walls is a thermal equilibrium condition which can only occur at constant temperature. The condensed liquid is returned to the bottom of the structure (for re-vaporisation) by capillary action at the mesh screen.

In order to prolong the life of the heat-pipe it is necessary to control the rate of temperature increase while the sodium is in its solid phase. The controller has been programmed automatically to ramp the heat-pipe temperature at a controlled rate until it approaches 450°C, at which temperature the sodium can be considered molten and the temperature is then allowed to rise at the full rate.