SLIM QUARTZ AND METAL CLAD CELLS (INCORPORATING ISOCAL-6 17156M INDIUM 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.

Serial No:..

Date:..

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INTRODUCTION

In the practical world of industrial temperature measurement, a large number of laboratories who normally make comparison calibrations need 1 or 2 fixed points to monitor their Standard Platinum Resistance Thermometers, or would like to calibrate shorter thermometers than the larger cells and apparatus can accept. For this group of users Isothermal have introduced ‘slim’ cells.

What are Slim Cells?

The special requirements of immersion depth, plateau duration, etc. required for the calibration of SPRT’s may not be necessary in laboratories charged with calibrating industrial resistance thermometers, thermocouples and thermistors, but mobility and cost may be more important. “Slim cells” is a name given to another category of cell, being somewhat slimmer, slightly shorter and lower in price than the standard varieties.

Slim cells are built using the same materials, techniques and purity of metal as the larger cells, but the uncertainties associated with them are somewhat larger, not because of the cells but precisely because their properties cannot be measured with SPRT’s and transfer thermometers must be employed in qualifying them.

In consequence of their smaller size, smaller, lighter-weight apparatus (bench-top or cart-mounted furnaces) may be used to melt and freeze the metal in these cells. Sealed cells and associated apparatus, such as the Medusa and Oberon are available from Isotech.

Slim cells can be supplied in either quartz glass, or for Indium, Tin and Zinc, Isotech can safely encase the cells in a non-contaminating metallic housing. The Isocal-6 17156M Indium Cell is specially designed to fit into Isotech’s Calisto apparatus (see drawing on page 11).

INSTALLATION

If cell accessories as shown in the diagram on pages 7,8, 9,10 or 11 have been purchased from Isotech, (comprising the inconel basket, insulation spacers, foil disc, inconel baffle and kaowool pad), these should be assembled in the furnace without the fixed point cell and pre-aged for a few hours 10˚C above the fixed point cells transition temperature. This avoids possible contamination of the fixed point cell.

On arrival the cell should be inspected for damage.

One easy way to check if a cell is leaking is to plunge it into hot water. If it is leaking a stream of air bubbles will come out of the cell.

If the cell leaks it must be returned for a new glass or metallic housing.

If not, the cell can be assembled ready for installation into the apparatus.

Firstly the cell should be cleaned using a tissue and alcohol. This is especially important for the higher temperature cells of Aluminium, Silver and Copper.

Next the cell should be assembled according to the drawing on page 7, 8,9, 10 or 11 and placed into the furnace to be used for melting or freezing the cell. If a lead cell has been purchased please refer to the special assembly drawing for this.

REALISING THE FOLLOWING FIXED POINTS: INDIUM, LEAD, ZINC, AND ALUMINIUM

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.

The cell is placed in the furnace, suitable insulation and cover added and a monitoring thermometer inserted. 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 a rack and replaced by a cold rod of quartz. 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.

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).

At the temperatures of Indium, Tin and Zinc it is generally permissible to withdraw a thermometer, of type Isotech 909, directly into room temperature. At the Aluminium Point, the thermometer must be cooled slowly to 450°C. See 909 handbook.

FREEZING THE TIN CELL

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

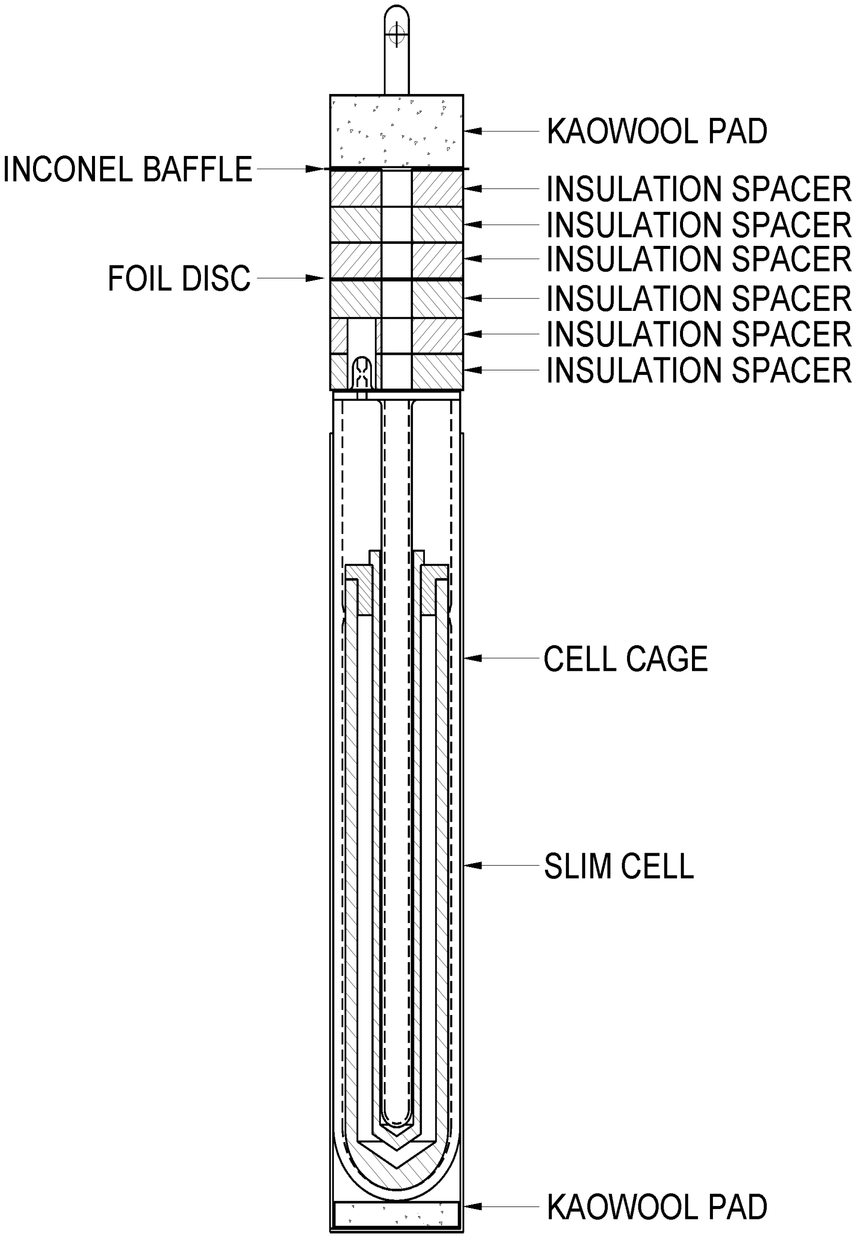
Tin can supercool as much as 10°C. If the furnace were allowed to cool to the nucleation point, it would probably not recover in time to realise the plateau.

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 cold quartz rod. 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.

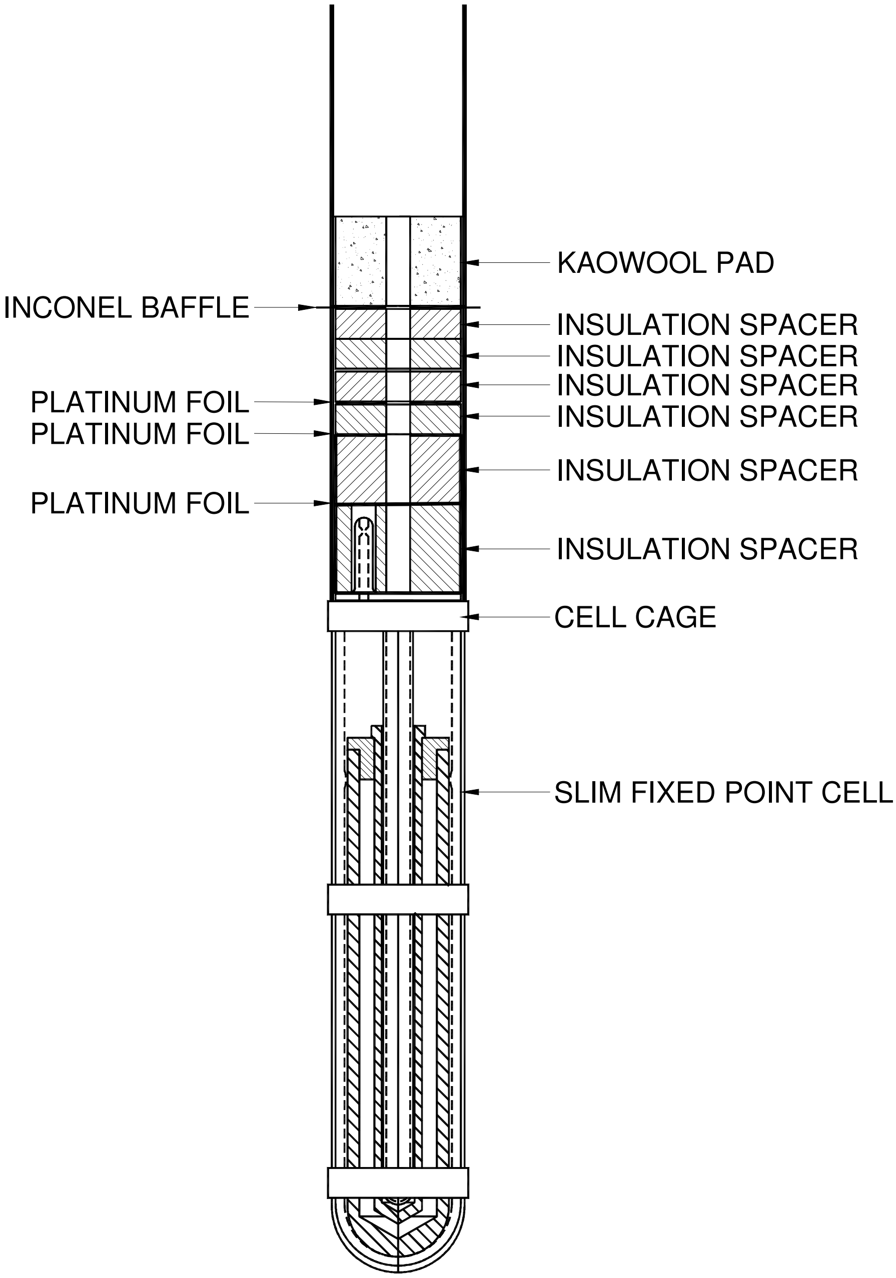
A USEFUL HINT

When first creating melts or freezes use large over or under settings - typically 3 to 5°C above or below the plateau. The result will be a shorter plateau 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 plateau temperature to increase the plateau length.

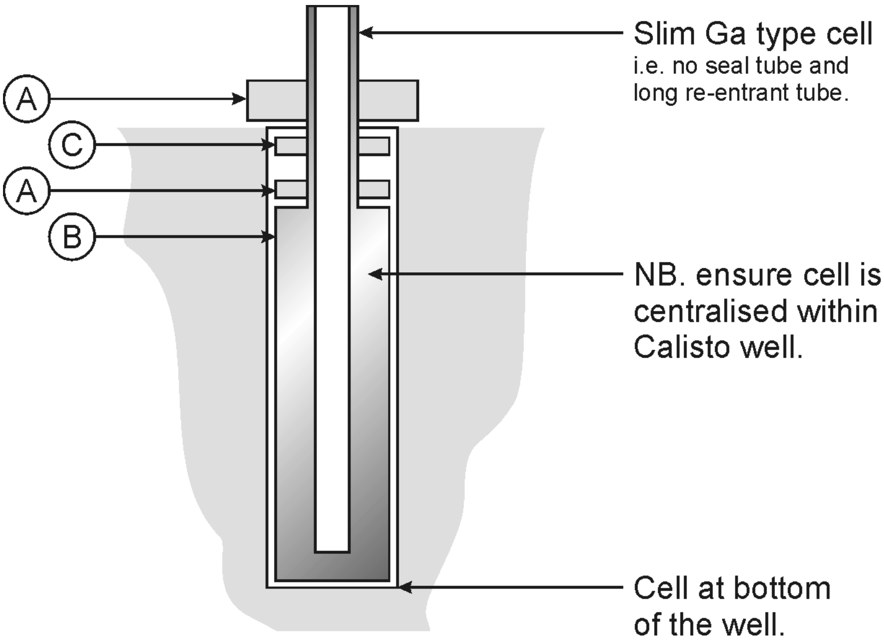
CELL BASKET ASSEMBLY FOR INDIUM, TIN AND ZINC SLIM FIXED POINT CELLS



CELL BASKET ASSEMBLY FOR QUARTZ CLAD ALUMINUM, SILVER AND COPPER FOR USE IN AN ISOTECH OBERON HEAT PIPE FURNACE



CELL BASKET ASSEMBLY FOR ISOCAL-6 17156M INDIUM CELL

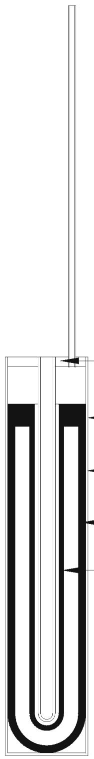


A SUPERWOOL INSULATION PART NO: 932-20-35

B ISOCAL-6 INDIUM CELL

C TOP COVER FOR CELL 953-02-10

ISOCAL-6 17156M INDIUM CELL
GENERAL ARRANGEMENT



DRG No.	DRAWING
953-06-01	STAINLESS STEEL LID & TUBE ASSEMBLY
953-06-05	GRAPHITE COVER (MOD. 477-02-01)
953-06-02	STAINLESS STEEL CONTAINER
953-06-03	GRAPHITE CRUCIBLE (MOD. 477-02-03)
953-06-04	GRAPHITE WELL (MOD. 477-02-02)

INSTALLATION OF THE SLIM CELLS INTO THE MEDUSA AND OBERON APPARATUS

Pages 7, 8, 9 and 10 of this manual show the assembly of the slim cell into the Inconel basket ready for insertion into the Medusa or Oberon apparatus. With the Medusa apparatus a small pad of kaowool is placed into the concave portion of the Medusa well for Indium and Zinc (for Lead please refer to assembly drawing). The cell assembly is then placed on top of this.

Once inside the apparatus the top of the well is insulated with a pad of kaowool.

If the user has not purchased a specially designed Inconel basket with the cell, they should bear in mind that the use of a solid housing for the cell, that sticks out of the Medusa or Oberon well, is not suitable. This would form a thermal shunt to ambient and distort the freeze and melting curves of the cell.

INSTALLATION OF ISOCAL-6 17156M INDIUM CELL INTO CALISTO APPARATUS

Place the Calisto Indium Cell directly into the Calisto well making sure it is centralised within the well.

Pack a small amount of the superwool insulation over the shoulder of the cell, then locate the top-cover over the re-entrant tube of the cell and ‘click’ into place. Place a thick pad of superwool over the top of this cover with a monitor thermometer in situ, introduce some high temperature silicone oil into the re-entrant tube ensuring the oil is level with the inner steel tube of the cell.

For operation of the Calisto apparatus please see the appropriate handbook.

THE COPPER SLIM CELL

The Copper Point 1084.62˚C is a secondary point of the ITS-90.

It requires the use of thermocouple technology to measure the melt and freeze performance.

The following hand-drawn graph was made from a series of readings taken with a calibrated type R thermocouple. On the edge of the graph is shown the uncertainty of calibration of the thermocouple which as can be seen swamps the measurement itself.

DEVITRIFICATION AND CARE OF THE COPPER CELL

All glass is a supercooled liquid and once it begins to devitrify (crystalize) the process cannot be reversed. Devitrified glass looks like frosted, or sand blasted glass.

Quartz glass which is the glass used to cover the Silver and Copper Slim 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.

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.