BLACK BODY FIXED POINT CELLS

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.

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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.

⚠ 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.

BLACK BODY 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.

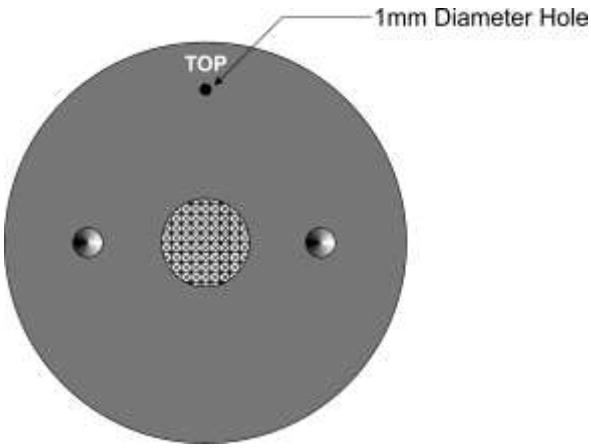
If not, steps should be taken to obtain training. The inexperienced user may inadvertently, permanently damage the cells they are using otherwise.

When these cells are manufactured they are held vertically, filled with metal shot and melted.

As the metal shot melts it begins to shape to the inside of the cell and subsequently takes up less of the inner volume. Therefore, following this first fill/melt the cell is filled and melted a second time.

IMPORTANT NOTE: **There is a 1mm diameter hole in the front face of the graphite cell, this is to ensure that the cell does not internally pressurise as the metal melts and freezes.**

During use with the cell held horizontally in a furnace, it is critical that the cell is orientated so as the 1mm hole is positioned uppermost at the top of the cell - as marked on the graphite and illustrated below.



As the cell is filled vertically, the first time you realise the cell horizontally the metal inside will have to reshape itself and so may not give a typical melt or freeze.

PRINCIPLE OF OPERATION

The most accurate calibration is performed using Fixed Points.

The Fixed Point Cell is usually a crucible made of high purity graphite containing an ingot of high purity metal. As the metal melts or freezes the temperature remains constant. A graphite cavity built into the ingot can then be used to calibrate radiation pyrometers.

The most common metals used are Indium, Tin, Zinc, Aluminium and Silver, although Gold, Copper, Antimony and Lead can also be used.

Isotech produce a range of Fixed Point Cells designed for the calibration of radiation pyrometers.

To accommodate the Cells Isotech offers apparatus called Medusa R (for Indium, Tin and Zinc) and the Oberon R (for Aluminium, Silver and Copper). Also available are specially modified larger furnaces.

Figure 1 shows the Cell dimensions whilst Figure 2 shows how the cell and accompanying graphite disc are assembled into the inconel holder. Figure 3 shows the assembly of parts inside the apparatus. The gas inlet is positioned on the side of the apparatus and should be connected using vacuum pipes, first to the flow meter and then to the gas bottle. (See 984 - gas flow and control indicator, available separately).

Typical flow, measured at room temperature would be 0.2 of a litre per minute.

Monitoring the graphite case is essential to ensure the integrity of the cell. A good way to tell what is happening to the graphite Cell is to regularly weigh it. After a weight loss of 2 to 3grammes the crucible should be replaced, but even a weight loss of 1 gram should cause concern and an investigation into the integrity of the gas supply.

Figure 1: Graphite Housing for Black Body Sources to National Standards (964-01-01)

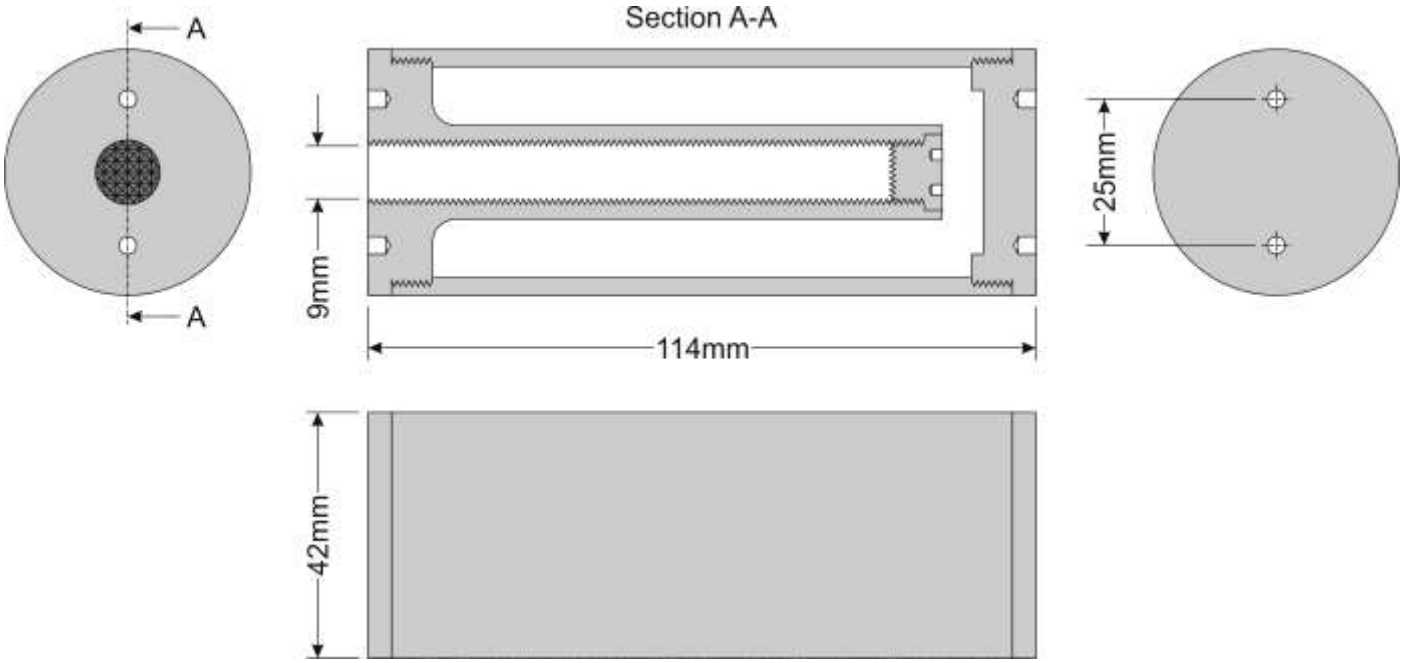
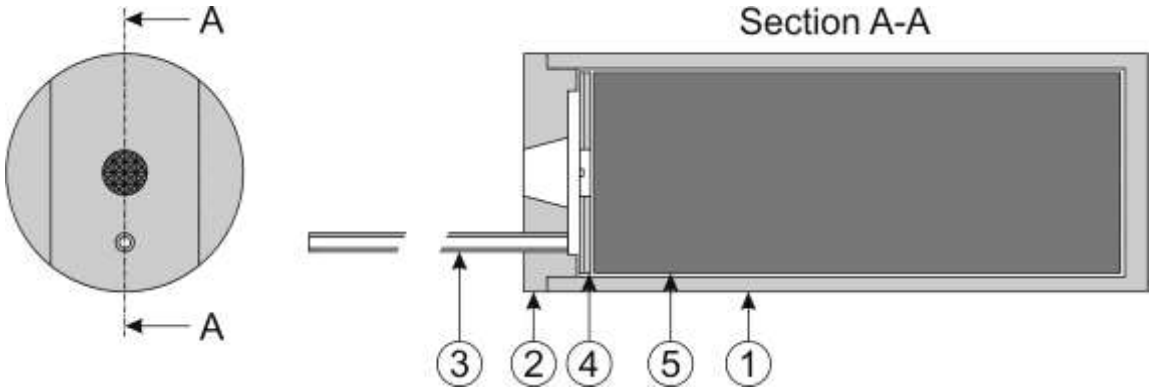
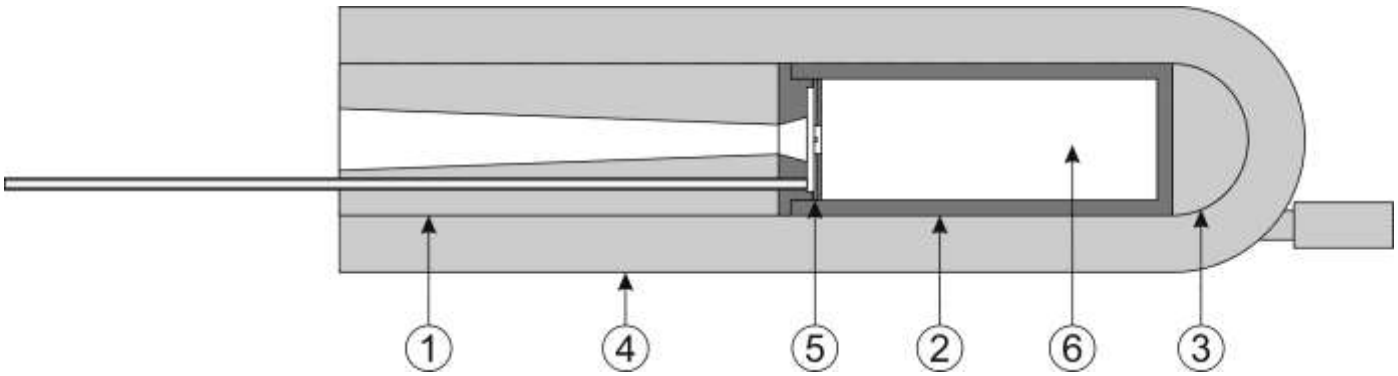


Figure 2: General Assembly (969-01-07)



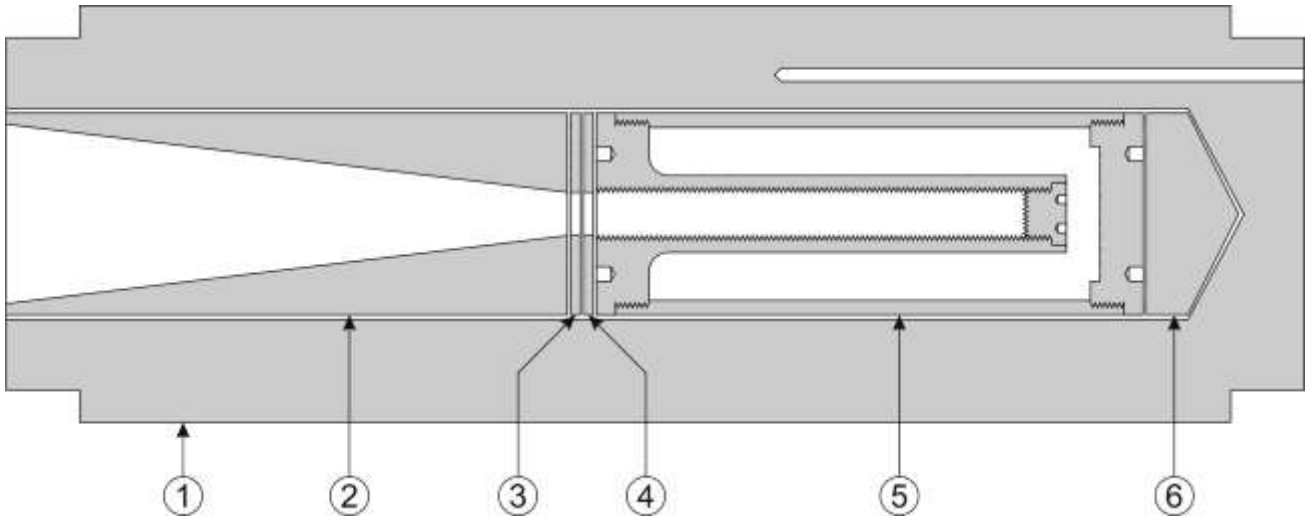
1. Inconel Housing Main - 969-01-12
2. Inconel Housing Lid – 969-01-12
3. Inconel Housing Tube – 969-01-12
4. Graphite Disc – 969-01-13
5. Fixed Point Cell

Figure 3: Black Body Assembly (969-01-00)



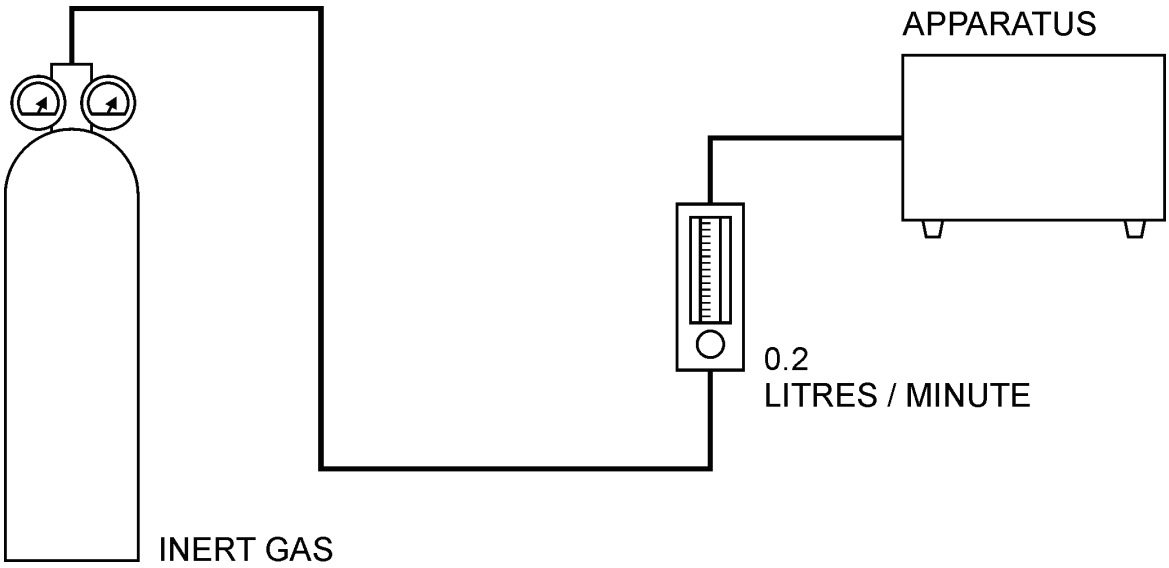
1. Upper Insulation (Blank 969-01-03 / Machined 969-01-06)
2. Inconel Housing (Drawing Number 969-01-01)
3. Domed Insulation (Drawing Number 969-01-02)
4. Heatpipe (Sodium)
5. Graphite Disc (Drawing Number 969-01-13)
6. Graphite Cell

Figure 4: General Assembly of Medusa Blackbody



1. Block (998-02-02)
2. Upper Insulation Block (998-02-05)
3. Aluminium Washer (998-02-06)
4. Graphite Disc (998-02-04)
5. Graphite Cell (998-02-01)
6. Bottom Cell Insulation (998-02-03)

Figure 5: Schematic to Outline the Gas Flow Circuit



ASSEMBLY OF FIXED POINT CELLS INSIDE THE UNIT

Before using the Fixed Point, assemble all the accompanying component parts except the cell and graphite discs, into the furnace and heat them approximately 10°C higher than the transition temperature of the fixed point being used. This will burn off any unwanted contaminants which could contaminate the cell.

The Fixed Point Cell and accompanying graphite disc are assembled into the inconel holder as per Figure 2. Then the first insulating piece is placed in the furnace throat, followed by the other parts as per Figure 3. It is important whenever handling the cell or accompanying parts, that the gloves provided are worn. This will prevent contamination of the metal within the cell.

With scientific equipment such as fixed point cells, there is no substitute for experience. These cells and apparatus are intended for use by experienced personnel in the field of fixed point calibration.

A training course with experts familiar with such measurements is recommended, see our website for details www.isotech.co.uk

The following notes are aimed at helping to point out some of the pitfalls that can be avoided.

They do not represent a comprehensive guide to the subject.

PRELIMINARY THOUGHTS ON THE USE OF FIXED POINT CELLS AS BLACK BODY SOURCES

The user new to Fixed Point Cells and intending to use them as black body sources has two sets of problems.

Firstly to understand and master the use of the Fixed Point Cell, and secondly to set up and align optics to monitor the Cells.

It is too easy, when looking for fractions of a degree to get a confusing soup of slopes, offsets and errors which are due to the measurement technique, but ascribed to the Cell performance.

Let me therefore make some observations and suggestions concerning the use of these Cells.

Firstly the Cells, graphite crucibles of very pure metal (normally 99.9999% pure), with a re-entrant Graphite cavity which will act as the source during the melt or freeze of the metal.

Because of their construction and purity, these Cells will melt and freeze without change of temperature. (less than 0.05°C).

Therefore the first step in using Fixed Point systems is to familiarise oneself with the Cell performance in isolation.

I therefore strongly recommend that new users make, or purchase a very fine wire thermocouple and introduce it into the re-entrant cavity of the Cell. Using this, the Cells performance can be monitored without the confusion of optical pyrometry.

Secondly, it is surprising but most users do not understand that the closeness of the furnace temperature to the melt/freeze temperature of the Cell dictates the length of the melt or the freeze.

Simply, heat is transferred from, or to the Cell, freezing or melting it, at a rate in direct proportion to the temperature difference between cell and its surroundings.

For good plateaus 1 to 5°C is recommended as the temperature difference.

Thirdly, slowly cells give best performance when they go slowly from their freeze to melt temperatures. So let the Furnace/Cell completely stabilise either 5°C below or 5°C above the freeze/melt temperature. Monitor both controller and monitor thermometer to ascertain when stability is reached.

Fourthly, gas flow

Graphite reacts with air to form carbon dioxide, CO_2. This reaction increases with temperature. It is a small effect and can be all-but ignored at temperatures up to and including the temperatures used for Zinc Fixed Point Cells.

However, for Aluminium and Cells above Aluminium the reaction is swift enough to be a problem, and so air needs to be excluded from around the Cell.

To this end the higher temperature Cells are housed in a housing with a rear inlet for an inert gas such as Argon or Nitrogen.

The trick is to keep enough gas flowing to prevent weight loss due to the graphite oxidising but low enough not to cool the Cell.

Gas expands according to the gas law which makes the volume proportional to the absolute temperature. We measure the flow at room temperature, and so to keep constant gas flow at the higher Cell temperatures we must reduce the flow at room temperature as we use the higher temperature Fixed Points.

e.g. 0.2 litres/minute at 20°C is .63 litres at the Aluminium freeze point and .84 litres at the Silver point.

0.2 to 0.4 litres per minute is enough at the Fixed Point temperature to prevent oxidisation.

However, regular checks of the crucibles weight are recommended especially when learning about the Cells.

SETTING UP AND USING THE CELLS

Melting and Freezing the Cells

Position the Cell into the control portion of the apparatus and connect the gas purge.

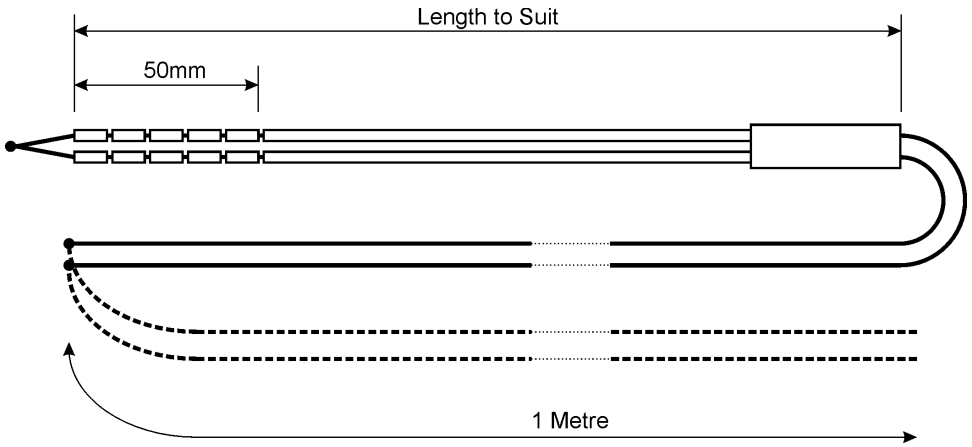
Unless you are completely familiar with Fixed Point Black Body Cell operation and their use with optical pyrometers, we recommend that during the familiarisation phase a very small, low thermal conductivity monitoring thermocouple be used to measure what is happening inside the Cell - see attached sketch.

The thermal mass of the Cell is large compared with the other masses inside the Apparatus and so the Cell must be given time to 'catch up' with the temperatures around it.

The procedure is to set the Apparatus temperature 5°C below the melt temperature. Then wait until the Cell temperature has stabilised, as indicated by the fine wire type R thermocouple.

This can be used as a calibration point by comparing the indicated temperature of the Apparatus with the output from the type R thermocouple.

Next set the apparatus temperature 5°C above the melt temperature, monitoring the cell temperature until it has passed through the melt phase, and re-established at the furnace temperature.



0.1 to 0.2mm ι type R thermocouple wire with Alumina insulators 5 x 10mm long by 0.9mm outer diameter on each wire. Then two longer insulators to make a total hot length of 200mm. The thermocouple was made continuous for a further 1 metre and insulated in kapton to two cold junctions where the Type R wire was connected to two copper multi strand Teflon insulated wires 1 metre long to load the signal to a DVM.

The temperatures of the molten Cell, and that of the apparatus are again noted.

From the above data it is possible to set a controller temperature 1°C below the freeze temperature.

Following this, the Cell is again monitored until it reaches its freeze temperature at which time a cold (room temperature) quartz rod or tube is introduced into the Cell cavity (if this is not available cold inert gas may be used as an alternative).

This is to cause solid metal to form around the black body cavity.

After two minutes the rod can be withdrawn (or the gas purge removed) and the monitor thermocouple will quickly reach the metal freeze temperature.

The temperature will then remain fixed until approximately 80% of the metal is frozen, when the Cell temperature will drop slowly to the furnaces set point.

Once the user is familiar with the method and has a feel for what is happening inside the cavity he can begin to add other components to enable him to calibrate optical pyrometers during the flat part of the freeze plateau.

There follows a conclusion of an evaluation of a silver fixed point cell at Isotech during July 1995.

The method of this evaluation is as per pages 10 and 11 of this manual.

CONCLUSION

The cell design when prepared in a similar way to other ITS-90 Fixed Point Cells is capable of giving a flat plateau to within 0.071°C or better for 2 hours 45 minutes or longer.

This is more than adequate for either thermocouples or radiation pyrometers.

Ref. Log book 26/27 July 1995.

The cell parts and Silver ingot were inspected and weighed after use and no deterioration was found.