





User Maintenance Manual/Handbook

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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.

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#### **GUARANTEE**

©lsothermal Technology Limited

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

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# 

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               |
|-----------------------|-------------|---------------------------|
| $\triangle$           | ISO3864     | Caution (refer to manual) |
| Â                     | IEC 417     | Caution, Hot Surface      |

# 

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 lsotech 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 |
|-----------------------|--------|
|-----------------------|--------|

| non | condensing |
|-----|------------|
| ļ   | non        |

# ISOTECH

## $\triangle$ HEALTH AND SAFETY INSTRUCTIONS

- I. 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 has 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 lsotech agent for repair.
- II. Ensure materials, especially flammable materials are kept away from hot parts of the apparatus, to prevent fire risk.



## INSTRUCTIONS FOR MEDUSA R 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.



## PRINCIPLE OF OPERATION

The most accurate calibration is performed using fixed points.

The fixed point cell is usually a crucible made of graphite containing an ingot of pure 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.

lsotech produce a range of fixed point cells designed for the calibration of radiation pyrometers.

To accommodate the Cells Isotech offers two pieces of apparatus called Medusa R (for Indium, Tin and Zinc) and the Oberon R (for Aluminium, Silver and Copper).

Parts numbers for the cells are as follows: Indium 998-06-00A, Tin 998-06-00B, Zinc 998-06-00C and for the Oberon R - Aluminium 998-06-00D, Silver 998-06-00E and Copper 998-06-00F.

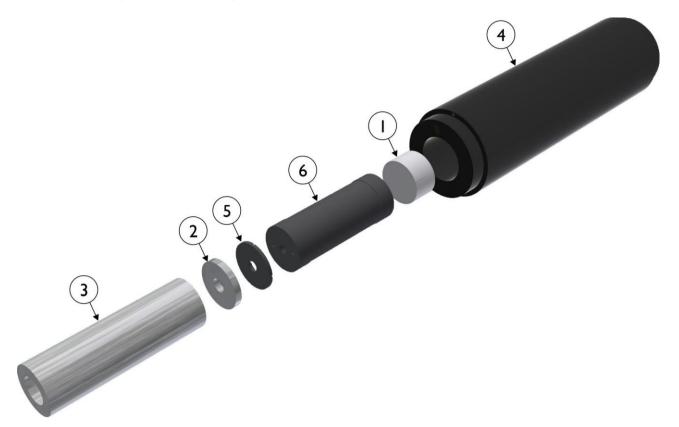
Drawing 998-02-01 shows the cell dimensions whilst drawing 998-00-00 shows the assembly of parts inside the Medusa apparatus. The Gas inlet is positioned on the side of the Medusa and should be connected using the vacuum pipes provided first to the flowmeter and then to the gas bottle (assuming that the optional extra gas flow 984-00-00 and control indicator has been purchased).

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

An easy way to tell what is happening to the graphite cell is to regularly weigh it. After a weight loss of 2 to 3 grammes the crucible should be replaced, but even a weight loss of I gram should cause concern and an investigation into the integrity of the gas supply.

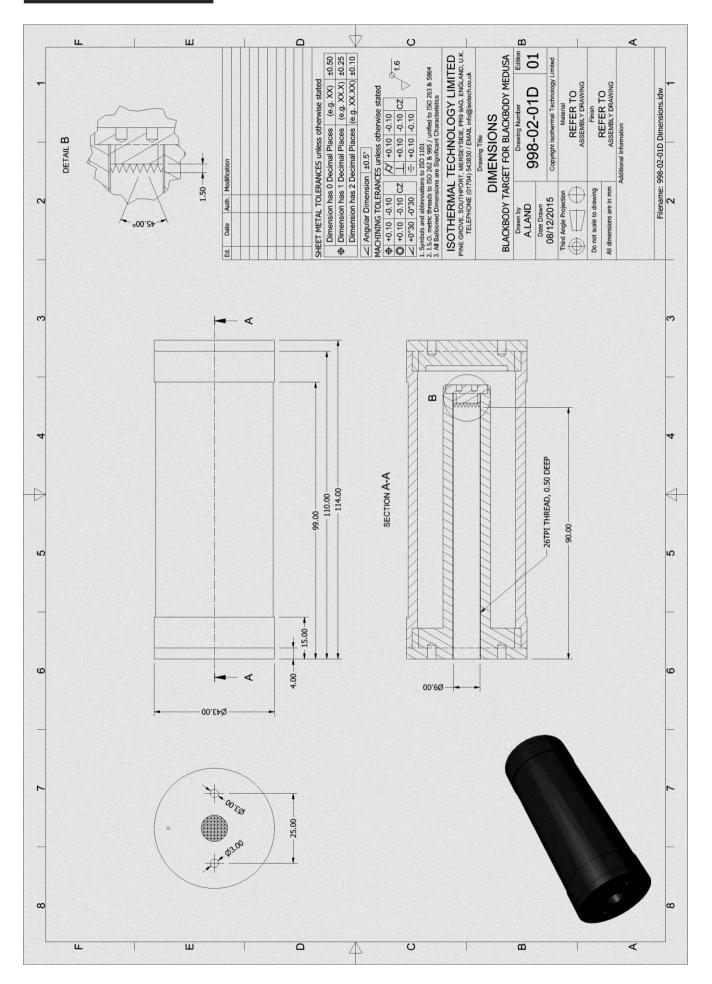


#### Medusa Blackbody General Assembly



| ltem | Part No.  | Description            |
|------|-----------|------------------------|
| I    | 998-02-03 | Bottom Cell Insulation |
| 2    | 998-02-06 | Aluminium Washer       |
| 3    | 998-02-05 | Upper Insulation Block |
| 4    | 998-02-02 | Block                  |
| 5    | 969-01-13 | Slotted Graphite Disc  |
| 6    | 998-02-01 | Graphite Cell          |

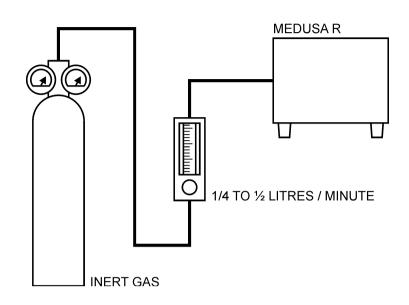




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## FLOW CHART



## ASSEMBLY OF FIXED POINT CELLS INSIDE THE MEDUSA R

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.

After lying the Medusa R on its side the first insulating piece is placed in the furnace throat, followed by the other parts as per drawing 998-00-00. The fixed point and accompanying graphite disc are assembled into the Medusa well as per drawing 998-00-00. It is important whenever handling the cell or accompanying parts that the gloves provided are worn. This will prevent contamination of the cell.

When operating state of the art products such as fixed point cells there are no simple and absolute guidelines or foolproof procedures, familiarity and know how must be gained slowly and with pains taking precision to avoid bad measurements and damage to the cells themselves.

A I or 2 day course with an expert familiar with such measurements may be costly, but cheaper than having to replace the cells.

As far as is known there is no comprehensive written information concerning the setting up and use of these fixed points.

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.

## ISOTECH

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

Note: Medusa 998 cells have a specially designed inner surface to the re-entrant tube which is extremely fragile and easily damaged, therefore if introducing a thermocouple into the cell for monitoring purposes, great care should be taken so this surface will not be damaged.

Because of their construction and purity, these cells will melt and freeze without change of temperature (less than  $0.05^{\circ}$ 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 carefully introduce it into the re-entrant cavity of the cell - see note above. 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 and 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. 0.2litres/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.



## MELTING AND FREEZING THE CELLS

Position the cell into the control portion of the Medusa R (see diagram 998-00-00) and connect the gas purge (if required).

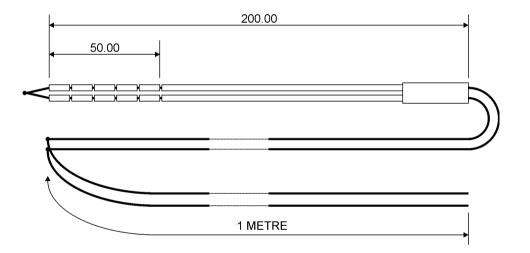
Unless you are completely familiar with fixed point black body cell operation and their use with optical pyrometers, we recommend that during the familiarisation phase a very small, low thermal conductivity monitoring thermocouple be used to measure what is happening inside the cell.

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

My procedure is to set the Medusa temperature up until I am 5°C below the melt. I 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 Medusa R with the output from the type R thermocouple.

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



0.1 to 0.2mm  $\emptyset$  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 Medusa R are again noted.

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

This done, the cell is again monitored until it reaches its freeze temperature at which time cold inert gas is introduced into the cell cavity.

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



After two minutes the gas purge can be removed and the monitor thermocouple will quickly reach the metal freeze temperature.

The temperature will then remain fixed until approximately 80% of the metals 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 an evaluation made at lsotech of a Silver 970 fixed point cell which the reader may find helpful.

## EVALUATION OF A NEW DESIGN OF 970 SILVER CELL FOR USE IN THE PEGASUS FURNACE FOR PYROMETER CALIBRATION

### INTRODUCTION

The new Black Body Silver Cell is encased in Inconel to contain and direct the flow of Argon around the cell.

(The Argon gas protects the graphite and molten Silver from air).

This report details the first tests on the cell to find the plateau length and best method of operation.

#### METHOD

The new cell assembly was placed as per the manual into the Apparatus. A gas supply of 0.2 I/m pure Argon purge was connected to the rear of the Apparatus.

The temperature as indicated on the controller was first set up in 100°C steps, allowing 3 to 5 minutes per step to 950°C. A small (0.2mm) Type R thermocouple was placed into the cavity of the Silver cell.

After stabilisation the cell temperature was recorded and compared to the controller indication, this acted as a calibration of the controller. The controller was set to 970°C to melt the cell.

The melt was plotted on a chart recorder. After 2 hours the cell was fully melted and the temperature of the Pegasus was reduced to about 1°C below the freeze temperature.

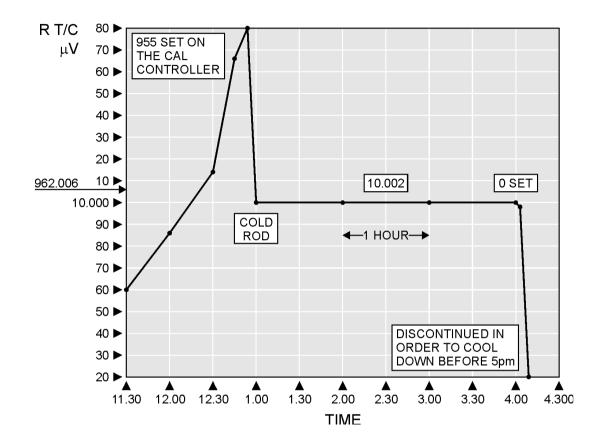
After 5 or 10 minutes two cold quartz rods were introduced into the cavity at 1 minute intervals to commence the nucleation of the Silver cell (the 970 black body cell does not have a crenulated inner re-entrant tube; consequently introduction of quartz rods to initiate the freeze does not pose a problem).

After a further 5 minutes during which the cell stabilised, the plateau remained flat (to within 1 $\mu$ V) at 10,002  $\mu$ V for 2 hours 45 minutes when the apparatus was cooled prior to the normal closing time of the factory.

(The ideal Type R value at the Silver point is  $10,003\mu$ V).



## PERFORMANCE GRAPH





## CONCLUSION

The new cell design when prepared in a similar way to other ITS-90 fixed point cells, is capable of giving a flat plateau (within  $^{1}/_{14}$ °C or better for 2<sup>3</sup>/<sub>4</sub> hours (or longer).

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

Ref. Log book 26/27 July 1995.

Cell parts and Silver were inspected and weighed after use and no deterioration was found.

**NB.** This test was performed on the second day of working with the Apparatus and new Silver cell. The first day was spent getting familiar with the Apparatus, gas flows, controller settings etc. and designing a thermocouple with no effective stem conduction.

#### Further Work

To get closer than  $^{1}/_{14}$ °C at the Silver point a new sensor assembly would be required, Pt100 4 wire connected with accuracy (or at least stability) of 0.01°C at the Silver point and no stem conduction.

If such a product can be manufactured then further tests will be performed.

I did not run the full length of the plateau, so this needs to be evaluated also.



## HINTS AND TIPS

Further information may be obtained about freeze and melt point cell operation from previous issues of Isotech's Journal of Thermometry.

The ceramics that accompany the cell are designed to give good insulation properties, and to position the cell in the zone of constant temperature. However you may reposition the cell by cutting the insulators with a normal pen knife.

Repositioning the cell will change the plateau time and flatness. Replacement, blank or specially drilled insulators are available from lsotech.

Please use the gloves provided when handling the graphite cell.

The inconel may look dirty. This is normal and will not affect the cell's performance.

Eventually the cell will lose some of its weight, due to graphite erosion.

Replacement graphite crucibles are available from lsotech.

#### PARTS LIST - INDIUM, TIN, ZINC

Graphite Crucible of Metal Top Insulation Piece Bottom Insulation Piece Graphite Disc

Aluminium Washer

Pair of Gloves

Certificates of Analysis



## USING THE PC INTERFACE

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

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



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

#### CONNECTIONS

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

#### **RS422** Connections

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



## CAL NOTEPAD

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

| 🖻 Isotech C       | NP 2006                                 |  |
|-------------------|---|--|
| <u>File H</u> elp |   |  |
| Instrument        | Heat Source Chart Log                   |  |
|                   | 700.000 -                               | Setpoint Programming Active                    |
|                   | 600.000 -                               | Scroll Minutes Temperature                     |
|                   | 500.000 -                               |  |
|                   | 400.000 -                               | ÷) 0.00  |
|                   | ≫ 300.000 -                             |  |
|                   | 200.000 -                               | T 0  |
|                   | 100.000 -                               |  |
| Running           | 0.000- <mark>1</mark><br>0 180          | Test Comms                                     |
|                   | Measurements                            | OK   |
| Start             |   |  |
|                   | Heat Source COM Port Controller Address | Controller Value Controller Setpoint 0.00 0.00 |
| Stop              | SITE Model Indicator Address            | Indicator Value Frozen PV                      |
|                   |   |  |

#### DEVELOPMENT

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



### HOW TO INSTALL CAL NOTEPAD

- I. Download the ZIP from http://www.isotech.co.uk/downloads (7.6Mb)
- 2. Extract the files to a temporary folder
- 3. Run setup.exe



- 4. Follow the prompts which will install the application, a user manual with setup information and the necessary LabVIEW run time support files.
- 5. Should you ever need to uninstall the software then use the Add/Remove Programs option from the Control Panel.

#### PROTOCOL

The instruments use the "Modbus Protocol"

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