

LOW TEMPERATURE FURNACE MODEL ITL-M-17701

User Maintenance Manual/Handbook

**Isothermal Technology Limited, Pine Grove, Southport, PR9 9AG, England
Tel: +44 (0)1704 543830 Fax: +44 (0)1704 544799 Internet: www.isotech.co.uk E-mail: info@isotech.co.uk**

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.

GUARANTEE

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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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Isothermal Technology Limited
Pine Grove, Southport, PR9 9AG, England
Telephone: +44 (0)1704 543830/544611 Fax: +44 (0)1704) 544799
Email: info@isotech.co.uk / Website: www.isotech.co.uk



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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)
	IEC 417	Caution, Hot Surface

ELECTRICAL SAFETY

This equipment must be correctly earthed.

This equipment is a Class 1 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.

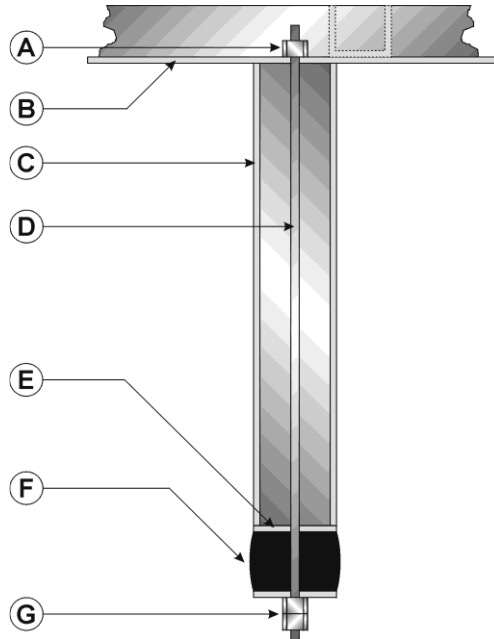
Conductive pollution, e.g. Carbon dust, must be excluded from the apparatus. EN61010 pollution degree 2.

HEALTH AND SAFETY INSTRUCTIONS

1. Read all of 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.

UNPACKING

SECTIONAL VIEW OF TRANSPORT CLAMP



- A NUT
- B CHANNEL
- C TUBE
- D STUD
- E WASHERS
- F RUBBER EXPANDING BLOCK
- G NUTS

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

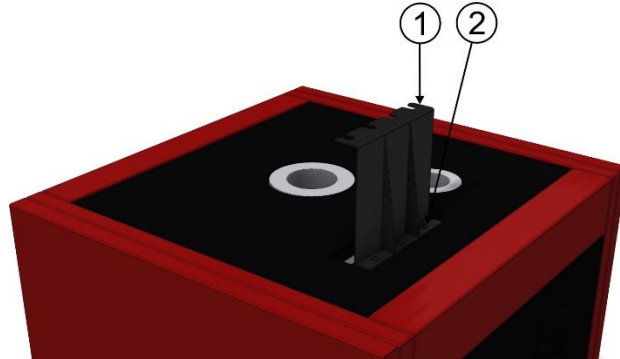
BEFORE COMMISSIONING IT IS NECESSARY TO REMOVE THE FURNACE CORE TRANSIT CLAMP. TO AVOID DAMAGE PLEASE FOLLOW INSTRUCTIONS:

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 equally by degrees and then completely remove the screws.
3. Gently lift the transit clamp vertically; a central tube which has secured the furnace core will then slide out. The 3 screws should then be replaced in the top of the unit
4. Normal commissioning procedure may now be followed.
5. Keep the transit clamp and use it if the furnace is ever transported.

INSTALLATION OF THE THERMOMETER SUPPORT (REMOVED FOR TRANSIT)

This furnace is supplied with the thermometer support for the safe storage of up to four thermometers, but removed for transit purposes. Please see the below image for a guide to the installation of the support to the furnace.

1. Thermometer support
2. Screw locations



Please screw the thermometer support to the furnace in the way shown above using the screws supplied. Tighten sufficiently.

Please take care when inserting or removing thermometers from the pocket below the support.

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.

ELECTRICAL INSTALLATION

THE FURNACES HAVE BEEN DESIGNED FOR CONVENIENT USE AND MAY BE PLUGGED TO A STANDARD THREE-PIN 13-AMPERE SOCKET. THEY ARE SUPPLIED SUITABLE FOR THE UK MAINS WHICH HAS LIVE, NEUTRAL AND EARTH LINES.

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.

THE LOW-TEMPERATURE FURNACE

The low-temperature furnace for realisation of metal freezing points is based on the design of Chattle (p.65, "Supplementary Information for the International Temperature Scale of 1990," B.I.P.M., 1990). It is furnished with two independent control systems, one for temperature control using a PRT and the other for over-temperature protection using a type N Thermocouple.

INTRODUCTION

The Isotech Low Temperature Fixed Point Furnace is designed specifically to realize and maintain the freeze plateaux of Isotech Indium, Tin and Zinc Fixed Point Cells, for calibration of thermometers on the International Temperature Scale of 1990.

The Low Temperature Furnace is a single-zone furnace. The recommended procedure for establishing a freeze plateau requires operator attention until the plateau is realized. Following that, the Model 17701 Furnace will maintain the plateau, essentially automatically, for a period of 10 to 12 hours.

The furnace core, into which the freeze-point cell is inserted, is of Aluminium alloy, which provides a very low thermal gradient along the core length. The main furnace heater is of the parallel-tube design as used at NIST. A pre-warming tube is provided.

Two entirely independent over-temperature safety devices are included. A dedicated (on-off) over temperature control circuit provides active safety. A fusible link in the main power circuit provides passive safety.

The Low Temperature Furnace is completely self-contained, castor mounted and requires no external supplies (except power).

NEW FEATURES

The furnace has been upgraded to benefit from the latest technology. Fitted with a crystal clear colour display the furnace is now fully programmable. Programs can be created for the furnace to switch between set temperatures, for example to bring the furnace to the melt or freeze temperature at a desired time, or to lower the furnace temperature after a predetermined time.

The PID control parameters are now dynamically optimised at different temperatures optimizing furnace stability. An Ethernet interface allows the furnace temperature to be monitored across a network whilst a USB Interface allows programs to be copied or for the furnace heat up and cool down history to be exported.

GENERAL LAYOUT

Figure 1: Front View

1. USB Socket
2. Ethernet socket
3. Main Controller
4. Overtemperature controller
5. Main power switch

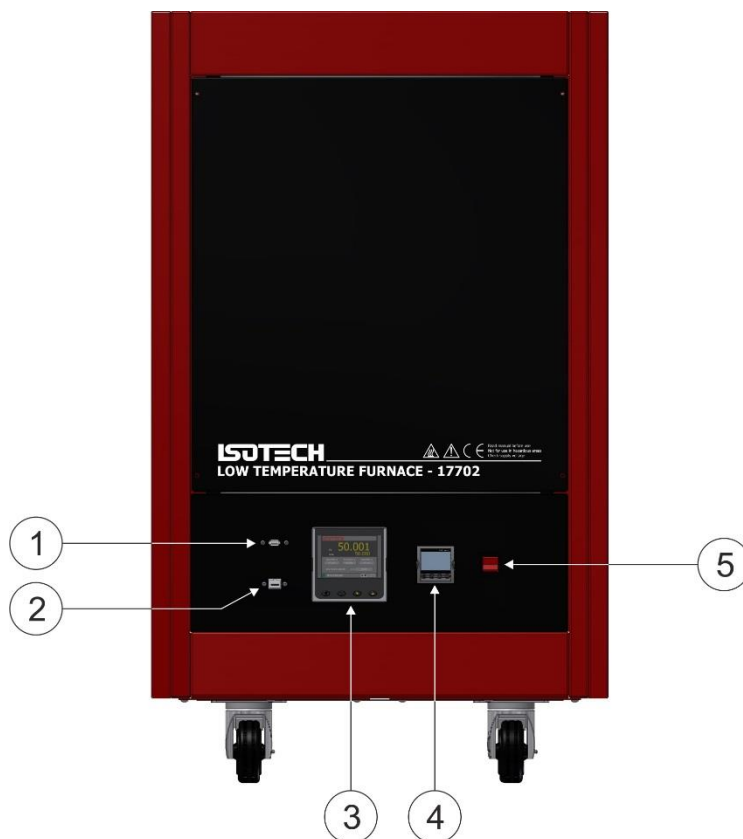


Figure 2: Rear View

1. Mains power cable

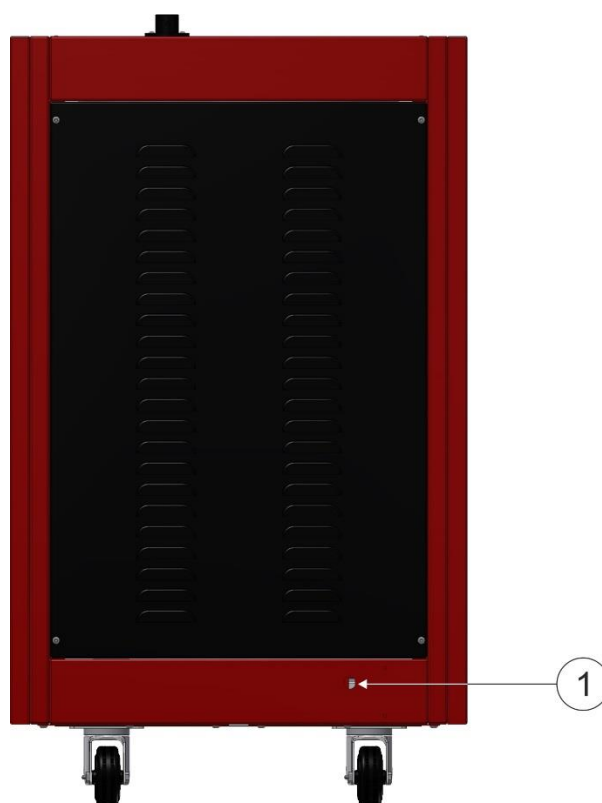
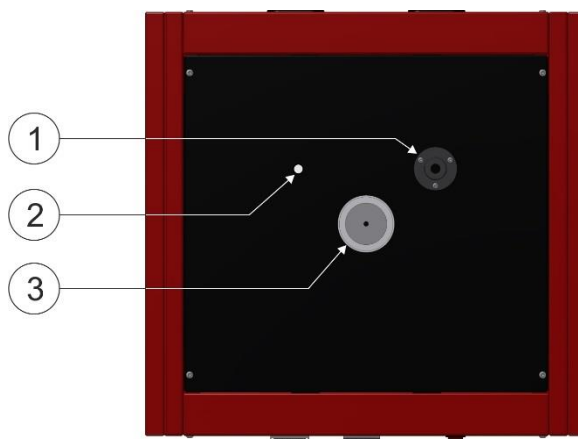


Figure 3: Top View

1. Gantry support
2. Pre warming pocket
3. Furnace core



QUICK START

Locate the furnace in a suitable, level location. Leave a minimum of 150mm of space around and 500mm above the furnace for air circulation. Ensure there is a suitable power supply nearby.

Assemble the furnace as in figure 18, connect the furnace to the electrical supply and power it up.

The Overtemperature controller will power up and display the current temperature of the furnace.

To power up the furnace you will need to enable the main controller. To do this, press the SCROLL button on the Overtemperature controller until Hi Alarm is displayed, using the up/down buttons enter a new temperature for the overtemperature protection.

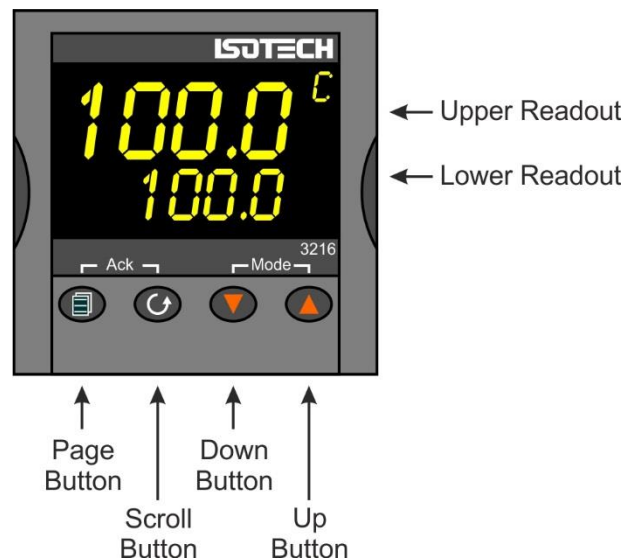
Initially this can be quite a low value, say 200°C, for test purposes. Now press the SCROLL and PAGE buttons together, once to enter the new value and a second time to reset the controller. On the second press the furnace will make a noise as the main control circuit powers up.

The furnace is now active and ready for use. On the main controller, press the SCROLL button to highlight the Setpoint box. Press again to adjust the value and select a suitable value, a value 20°C below the overtemperature is suitable, using the up/down buttons. Enter the value by pressing the Scroll button. The furnace will now rise in temperature to the selected value.

If the furnace has been in transit or not been used for several days then there may be an ingress of moisture into the insulation and other ceramics, the furnace may need to be “dried out” by running at a low value such as 100°C for a period of several hours.

USING THE OVERTEMPERATURE CONTROLLER

Figure 4



The furnace uses an alarm value on the controller to disable the heaters if the furnace goes into the alarm condition. If the furnace goes into the alarm condition, it will power down the Main controller and circuitry.

Adjusting the Overtemperature value on the controller:

1. Press the Scroll button to display **A1 HI**
2. Use the up/down buttons to scroll to the desired alarm setting. This would normally be 10-20°C above the target temperature
3. To enter the value, you will need to press the **PAGE** and **SCROLL** buttons simultaneously
4. To re-set the controller from an alarm condition you will need to press the buttons **again** simultaneously
5. If the controller is in the alarm condition and the complete furnace is powered down, the controller will “remember” the condition and will still need to be manually reset when powered up again

The controller installed on this equipment uses an advanced three loop controller with a multi-channel sensor input.

Other features of the controller are:

- Built in data logging
- Ethernet connection
- USB host for controller updates and data export
- Bright colour display
- Dynamic optimisation of the control PID parameters
- Eight point zone linearization for Main and Annealing furnace
- Built in Programmer, supports 100 programs, each of 25 segments

Before using the controller, please read this manual thoroughly to get the best performance from the equipment.

Certain features used in this controller require different levels of access. The Operator access requires no passcode to use. These functions include:

- Changing the setpoint
- Changing any values using the USER ACCESS PAGE

Other features will require the user to log in as “Supervisor” to carry out these functions. These include:

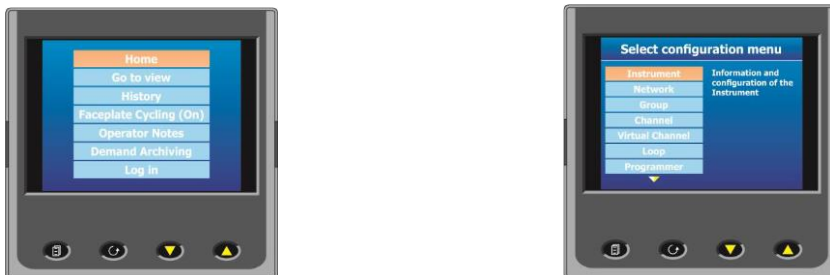
- Adjusting the PID settings
- Using the Programmer function
- Changing the linearity settings
- Extracting data from the recorder function

Configuration changes to the controller require you log in as “Engineer” to perform these functions. These include:

- Change language selection
- Change °C to °F or K
- Change equipment clock and time zone

How to log in as the SUPERVISOR:

Figure 5



1. Press the Page button to display the Home menu
2. Scroll to LOG IN
3. Press the SCROLL button
4. Press up to scroll to SUPERVISOR in dialogue box
5. Press the SCROLL button to prompt for a passcode
6. Select code **15** from the soft keyboard
7. Press PAGE button to prompt to accept the changes
8. Select Yes and enter with SCROLL button to display the SUPERVISOR screen (Figure 5)
9. When logged in as the SUPERVISOR the equipment will control in the normal way

How to log in as the Engineer:

1. Press the Page button to display the Home menu
2. Scroll to LOG IN
3. Press the SCROLL button
4. Press up to scroll to ENGINEER in dialogue box
5. Press the SCROLL button to prompt for a passcode
6. Select code 17 from the soft keyboard
7. Press PAGE button to prompt to accept the changes
8. Select Yes and enter with SCROLL button to display the ENGINEER screen
9. When logged in as the ENGINEER, the equipment will **not** control the temperature of the heat source

USING THE CONTROLLER

The Isotech splash screen and controls:

Figure 6: The **USER INTERFACE** start up screen and controls

1. **ISOTECH** splash screen
2. **PAGE** button
3. **SCROLL** button
4. **DOWN** button
5. **UP** button

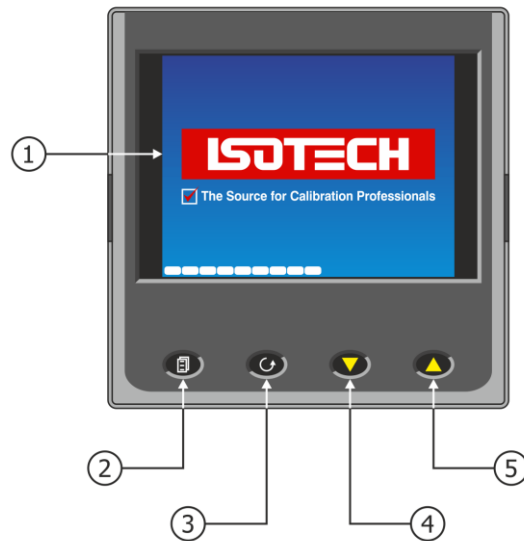


Figure 7: The **HOME** page

1. Furnace temperature
2. Furnace setpoint
3. Furnace setpoint box
4. Data record symbol
5. **PAGE** button
6. **SCROLL** button
7. **Down** button
8. **Up** button

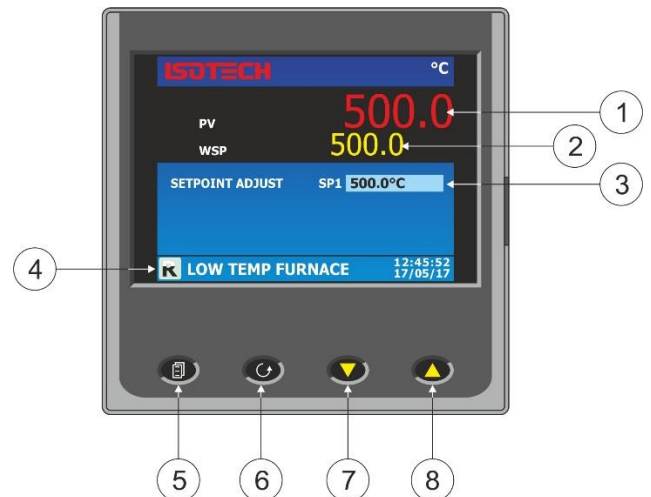


Figure 8: The **USER ACCESS** page

1. Proportional band
2. Integral time
3. Derivative time
4. Autotune enable
5. Display units
6. Fixed offset
7. Network IP type
8. Setpoint Ramp Rate °C/min



Changing the Set Point

The main controller will control temperature of the furnace. The setpoint is accessible from the Home page of the controller. The controller is only active when enabled by the overtemperature controller.

To compensate for any errors in the linearity of the display temperature, the user can adjust the linearity using the 8 point adjustment curve for each furnace. See section below for details on how to do this.

To change the Setpoint of the equipment:

1. From the ISOTECH homepage, press the SCROLL button once, this will highlight the SETPOINT ADJUST box.
2. Press the SCROLL button once more and this will allow adjustment of the setpoint via the UP/DOWN buttons.
3. Scroll to the desired set point as described above.
4. When the temperature is set, press the SCROLL button again to enter and save the setting.
5. The equipment will now raise or lower the furnace temperature as required.

How to globally change from °C to °F or Kelvin:

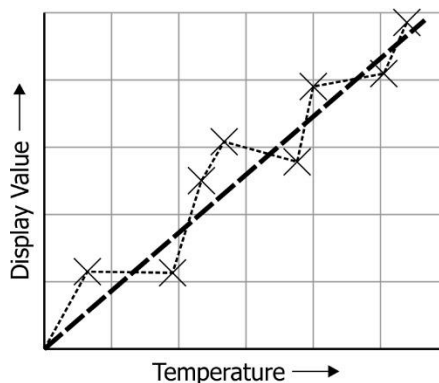
1. Log in as the Engineer
2. Scroll to INSTRUMENT
3. Scroll to DISPLAY
4. Scroll to UNITS
5. Select by pressing the Scroll button
6. Use up/down to select from °C, °F or Kelvin
7. Press the Page button to exit and save the selection

Process to apply a User linearization to the furnace

From time to time the control sensors inside the furnace will need to be adjusted as the sensor drifts with time and use at high temperature. The controller has the capability to compensate for these errors using an eight point correction curve. This will need to be regularly checked for errors and can easily be corrected within the controller using the following procedure.

The image below illustrates a simplified graphical view of the process.

Figure 9



Isotech standard offset values are set at the following temperatures:

This is listed as Channel 1 on the controller

1. 156°C
2. 232°C
3. 420°C
4. 500°C

Points 5, 6, 7 and 8 are unused as standard.

Please follow the procedure below to linearise the controller display value:

1. Set the furnace up as it will be used (Figure 18)
2. Place a reference thermometer inside the cell or equalizing block
3. Set the furnace to the lowest useable temperature (recommendation would be to use the temperatures listed above)
4. Allow the system the thermally stabilise
5. Press the PAGE button to enter the SUPERVISOR menu
6. Scroll to the CHANNELS tab and enter with the SCROLL button
7. Scroll to the ISOTECH tab and enter with the SCROLL button
8. Scroll to CAL STATUS and enter with the SCROLL button
9. Scroll to POINT 1 and enter the value from the reference thermometer

The controller is now ready to accept a value. Ensure the equipment is thermally stable, you will now need to use the temperature value displayed by the reference thermometer.

10. Record the value on the reference thermometer and scroll to DISPLAY VALUE tab and press enter
11. This will bring up a soft keyboard, use this keyboard to enter the exact temperature displayed by the calibrated reference standard
12. Press the PAGE button to enter the value, the controller will ask you to confirm the settings. Select YES and enter the value
13. The controller will now accept the value and move to POINT 2 on the display
14. Exit back to the homepage and check the value on the relevant display window
15. When confirmed, move to the next temperature and repeat the process for point 2 and so on

Important Note: Ensure that Point 1 is always used as the lowest temperature and offsets are entered in ascending order. If the values are place out of order then the controller will disregard them.

Enabling the AUTOTUNE feature:

The equipment has the ability to AUTOTUNE the control parameters to hone the temperature stability at a specific temperature. When enabled it will allow the controller to cycle the temperature and calculate the best values for this temperature. After two cycles it will install the calculated values and use these for the control loop.

You are not required to log in to access this feature.

To enable the AUTOTUNE feature:

1. Scroll to the USER ACCESS page
2. Press the Scroll button and then select the AUTOTUNE feature using the down button
3. Press the Scroll button again to highlight the feature
4. Use the up/down button to select ON
5. Press the Scroll button again to enter the value.
6. The controller will now begin the tuning process

During the tuning process, AT can now be seen to the left of the clock on the ISOTECH homepage.

To disable the tuning feature during a tune, repeat the process above and select OFF. This will now disable the tune and install the previous values.

How to change the Date and Time or the Language of the equipment:

The Date and time of the equipment can be changed to suit local time. This can also incorporate the daylight-saving time in the time zone of your country or region. The display language of some of the display screens can also be change to suit the local language.

The options for this are:

- English
- French
- German
- Italian
- Spanish

Note: Only the Supervisor and Engineer menus will read in the alternative language. The Isotech Home screen will still read in English when this feature is used.

To change the DATE and TIME or LANGUAGE:

- To access these features, you will need to log in as the Engineer.
- When logged in, scroll to the INSTRUMENT tab on the Engineer homepage
- Press the SCROLL button to enter
- Scroll to CLOCK and enter by pressing SCROLL button
- Alternatively scroll to LOCALE and enter by pressing SCROLL button
- Adjust as necessary and exit engineer mode

The NETWORK INTERFACE:

Access to the NETWORK interface is available when logged in as the ENGINEER. This will allow adjustment to various network parameters, should they need to be adjusted.

This menu allows writeable access to:

- IP type (FIXED or DYNAMIC)
- IP address
- Subnet Mask
- Gateway

and Read only access to:

- MAC address
- Client identifier

To access the NETWORK INTERFACE:

1. Log in as the ENGINEER
2. From the ENGINEER page scroll to NETWORK tab and enter with the SCROLL button
3. Scroll to INTERFACE and enter with the scroll button
4. Scroll to and adjust desired parameter using the pop out soft keyboard

DATA LOGGING:

The equipment will always record data for all four channels UNLESS the equipment is in ENGINEERING mode. This is indicated by the green **R** in the bottom left hand side of the ISOTECH homepage (Figure 7)

The data can either be extracted via the USB socket on the front panel or to a PC by means of the FTP protocol (Remote Archiving) using REVIEW software, see page 42 for details.

To back up the data via USB stick on the control panel use the following procedure:

1. Log in as the SUPERVISOR
2. Plug a suitable USB memory stick to the socket on the control panel (Figure 1)
3. Ensure the controller has accepted the memory stick by the icon in the lower left of the ISOTECH homepage
4. Press the page button to scroll to the SUPERVISOR menu
5. Scroll to DEMAND ARCHIVING
6. Scroll to ARCHIVE TO and enter, select USB from the options and enter
7. Scroll to ARCHIVE and select from:
 - a) Bring to date
 - b) All
 - c) Last month
 - d) Last week
 - e) Last day
 - f) Last hour
 - g) None
8. When the selection is entered by pressing the SCROLL button, the transfer will begin (this may take several minutes depending on the selection made)
9. When the transfer is complete, remove the USB stick and exit SUPERVISOR mode

The data will be stored in a folder called HISTORY.

FTP SERVER ARCHIVING:

This allows the archiving of recorder files to a remote computer via the RJ45 type connector at the rear of the recorder, either directly or via a network.

In order to carry out a successful transfer:

1. Details of the remote host must be entered in the **Network Archive** area of configuration
2. The remote computer must be set up as an FTP server. Help from the user's IT department may be necessary in order to achieve this.
3. The remote computer must also be set up to respond to 'pings'. This is because the instrument pings the host whilst establishing connection, if it does not receive a response the archive attempt fails.
4. When accessing files using Microsoft® Internet Explorer, the address (URL) field can be in one of two formats:
 - ftp://<instrument IP address>. This allows a user to log in as the anonymous user (if the recorder has any account with the user name set to 'anonymous' with a blank password).
 - ftp://<user name>:<password>@<instrument IP address> to log in as a specific user.

For IE5 users, Microsoft® Internet Explorer displays, by default, history files only. To quit the history folder, either uncheck the Tools/Internet Options/Advanced/Browsing/'Enable folder view for FTP sites' option, or check the Tools/Internet Options/Advanced/Browsing/'Use Web based FTP' option.

PROGRAMMER FUNCTION:

The equipment has an on-board PROGRAMMER function built in. The PROGRAMMER runs in SEGMENTS and can store up to 100 programs, each containing 25 segments.

This feature is particularly useful for an automated calibration run, the test data can then be extracted and converted to a spreadsheet using the REVIEW software available.

The programs can either be created using the available software, or using the control interface on the equipment.

The software can be downloaded at www.isotech.co.uk/downloads. Once created, a program can be sent directly to the instrument via the network connection or saved to a USB memory stick and imported from there via the USB socket on the controller panel

Figure 10: Programmer display – Program not running

1. Program name
2. Function select box
3. Furnace temperature
4. Current furnace setpoint
5. Program status



Figure 11: Programmer display – Program running

1. Program status
2. Segment type
3. Program progress
4. Segment time remaining
5. Program time remaining
6. Segment progress



Instructions and details of installation are supplied with the download.

Below is the procedure to install a program for Channel1 (furnace)

To create, install and run a PROGRAM via the controller interface please follow the instructions below:

See below for a more detailed description of each of the PROGRAM functions.

PROGRAM DETAILS:

Operation

This allows the user to select one of the following:

Load. Opens the program store and allows the user to select a program to be loaded.

Store. Allows the current program to be saved to the internal program drive. This is useful if you wish to snapshot the current program and store this under a different program name.

Delete. Allows the selected program to be deleted.

Delete All. Deletes all programs.

Copy. Copies the selected program for 'pasting' either from the internal drive to the USB device, or vice-versa. This is useful if you wish to transfer a program to other instruments.

Copy All. As above, for 'Copy', but copies all the programs in the selected directory.

Note: If a 'Store', 'Copy' or 'Copy All' operation would result in there being a total of more than 100 program files in the internal drive, the operation fails and an error message is displayed.

Status

Success. Previous operation was successful.

Failed. Previous operation failed.

Loading. The program is loading.

Copying. The program copy process is underway.

Deleting. The relevant program is being deleted.

Program

The name of the program currently loaded.

Holdback Style

Appears only if 'Holdback' is enabled. See 'Holdback', below.

Program:

Holdback applies to all appropriate segments.

Per Segment: Holdback enabled on a segment by segment basis as described in 'Segment configuration below.

Ch1 Holdback

Appears only if 'Holdback Style' (above) is set to 'Program'.

Off: Holdback is disabled

Low: Holdback is entered when $PV < (PSP - \text{Holdback Value})$

High: Holdback is entered when $PV > (PSP + \text{Holdback Value})$

Band: Holdback is entered when $PV < (PSP - \text{Holdback Value})$ or $PV > (PSP + \text{Holdback Value})$

Ch1 Holdback value

The value to be used in triggering holdback.

Ramp Style

Ramp style applies to all ramp segments in the program. Ramp Style can be edited only when the program is in Reset mode. Setpoints, rates, times etc. are set in the individual segment configurations

Rate. A Ramp Rate segment is specified by a target set-point and the rate at which to ascend/descend to that set-point.

Time

A Ramp Time segment is specified by a target set-point and a time in which to achieve that set-point.

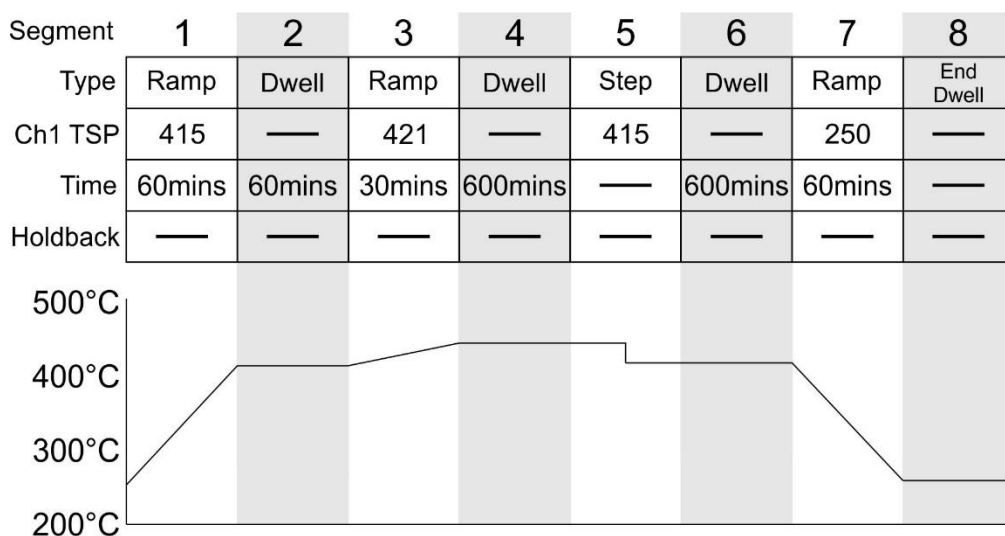
Ch1 Ramp Units

Select 'Per Second', 'Per Minute' or 'Per Hour' for ramp timing units. Ramp Units can be edited only when the program is in Reset mode.

How to create a simple PROGRAM using the controller interface:

Below shows a basic PROGRAM that demonstrates the furnace instrument ability to create a program.

Figure 12



This PROGRAM has 8 segments.

It shows the furnace instrument starting with a setpoint of 250.0°C

- It is then set to RAMP to 415°C
- It is then set to DWELL at this temperature for a period of 60 minutes
- It is then set to RAMP to 421°C over a period of 30 minutes
- It is then set to DWELL at this temperature for a period of 600 minutes
- It is then set to STEP to 415°C
- It is then set to DWELL at this temperature for a period of 600 minutes
- It is then set to RAMP to 250.0°C
- It will then END the program and DWELL until switched off

To enable/RUN this PROGRAM:

1. Log in as the SUPERVISOR and exit menu
2. Scroll to the PROGRAM page (Fig5)
3. Press the SCROLL button to highlight the RESET box
4. Press the UP button to highlight the PAGE icon in the top right hand side of the screen
5. Enter this with the SCROLL button
6. This will access the PROGRAM EDIT menu page
7. Scroll to OPERATION and select LOAD, enter with the SCROLL button
8. Scroll to TEST_1 and enter with the SCROLL button
9. The STATUS will display SUCCESS and the CURRENT PROGRAM will display TEST_1
10. TEST_1 is now the default program
11. To RUN the program TEST_1 exit the PROGRAM EDIT page using the PAGE button until you see the PROGRAM homepage
12. Press SCROLL once to highlight the RESET box
13. Scroll to TEST_1 and press the SCROLL button to initiate the program

The page will now change to show the current program running, as figure 11 above.

To edit an existing PROGRAM:

1. Follow the above procedure to point 10
2. Scroll down to SEGMENT NUMBER and select the SEGMENT you wish to edit using the SCROLL and UP/DOWN buttons
3. Edit the required parameter (RAMP, DWELL, TIME DURATION etc) using the scroll and UP/DOWN buttons and enter the value
4. When the editing is complete, it can either be left as the existing PROGRAM or saved under an alternative file name in the internal memory
5. To leave the alterations under the existing filename simply exit out of the menu and RUN the program as normal
6. To save as an alternative filename in the memory, SCROLL back up to OPERATION and enter using the SCROLL button
7. Scroll to STORE and enter
8. The soft keyboard will open allowing you to save the file as a filename of your choice
9. When the name is selected, press the PAGE button to save the changes
10. Both the original file and the modified file will now exist in the memory

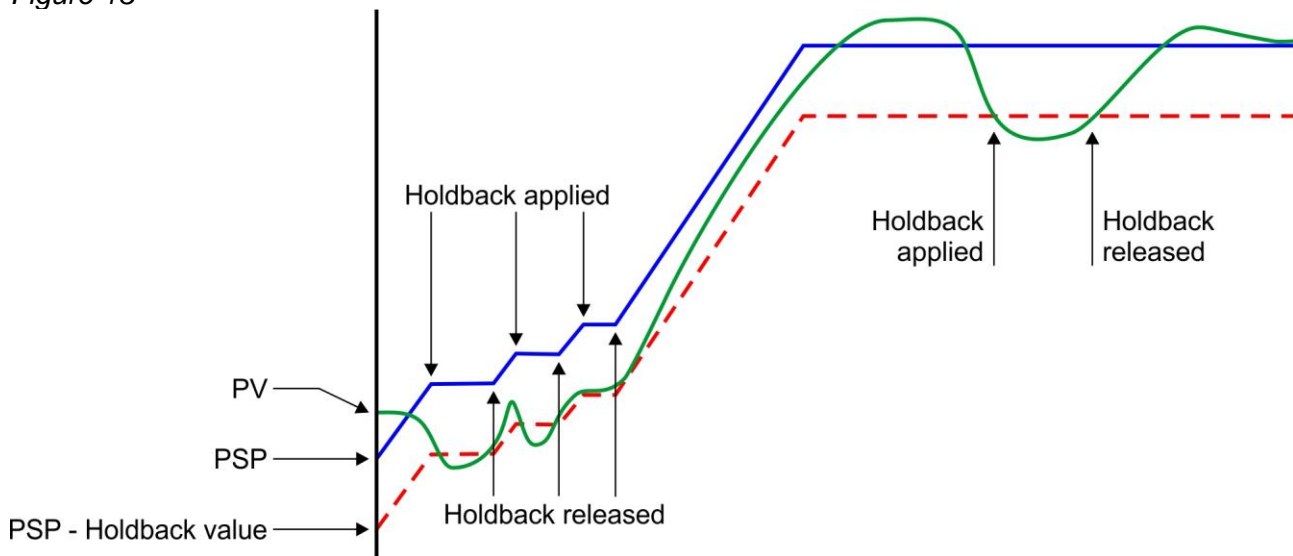
Using the HOLDBACK feature:

The PROGRAMMER has a HOLDBACK feature available.

Holdback pauses the program (freezes the Programmer setpoint (PSP) and the time remaining parameters) if the difference between the Process value (PV) and the PSP exceeds a user-specified amount (Holdback value). The program remains paused until the PV returns to within the specified deviation.

In ramp or step segments, holdback indicates that the PV is lagging the SP by more than the specified amount and that the program is waiting for the process to catch up. In a dwell segment, holdback is used to guarantee that a work piece stays at set-point within a specified tolerance for the specified dwell duration.

Figure 13



This is particularly useful when running a calibration program as it will allow the heat source to eliminate any “lag” between the controller and the heat source itself.

Holdback can be configured to hold either the segment or the complete program.

The USER defined value is used to define the actual value applied to it.

This can either be:

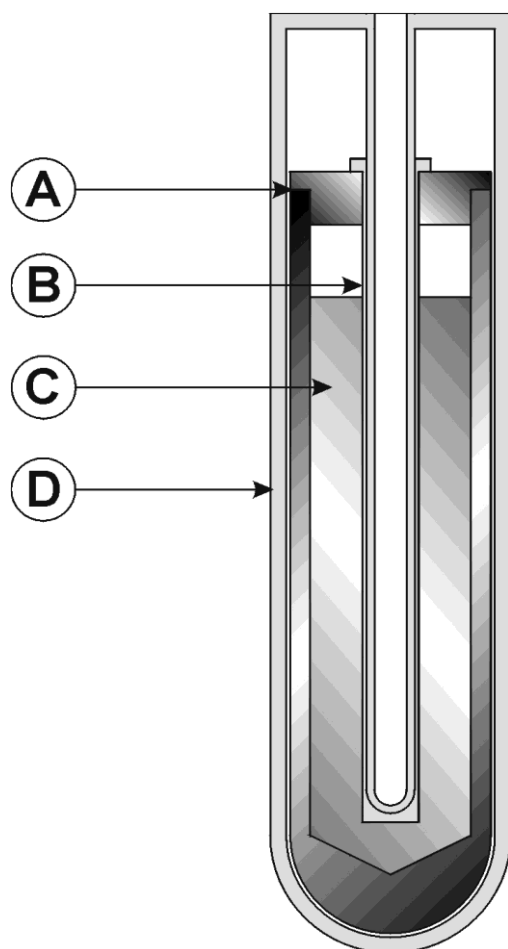
LOW, where the heat source temperature is below the Setpoint. The programmer will hold the value until the heat source has “caught-up” in temperature.

HIGH, where the heat source temperature is above the Setpoint. The programmer will hold the value until the heat source has “caught-up” in temperature.

BAND, where the heat source is outside a band of temperature around the setpoint

To enable this feature:

1. Go to EDIT AN EXISTING PROGRAM and enter the menu
2. Scroll to HOLDBACK STYLE and enter using the SCROLL button
3. Select desired parameter and enter using the SCROLL button
4. Scroll to CH1 HOLDBACK and enter the desired parameter and enter with the SCROLL button
5. Scroll to CH1 HOLDBACK VAL and select the desired temperature, enter using the SCROLL button

FIGURE 14: 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 atmos

FIGURE 15: SEALED METAL FREEZING-POINT CELL BODY DIMENSIONS

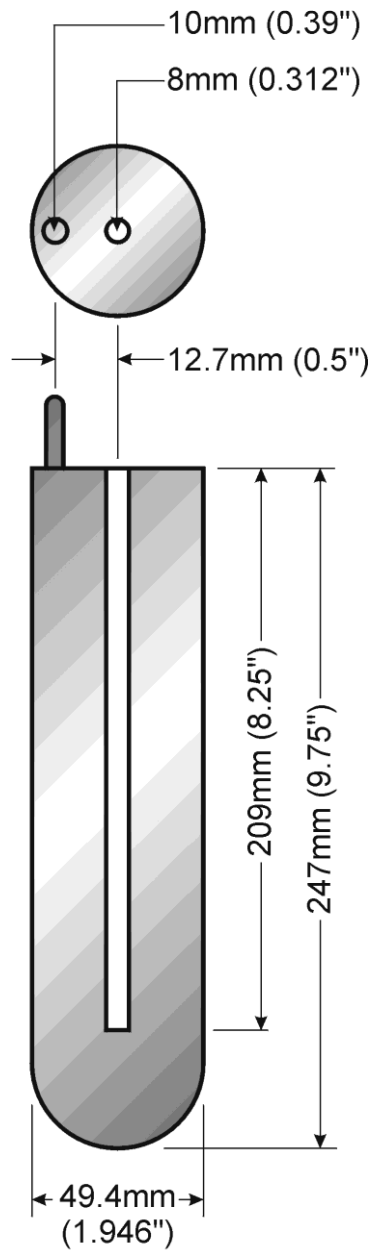
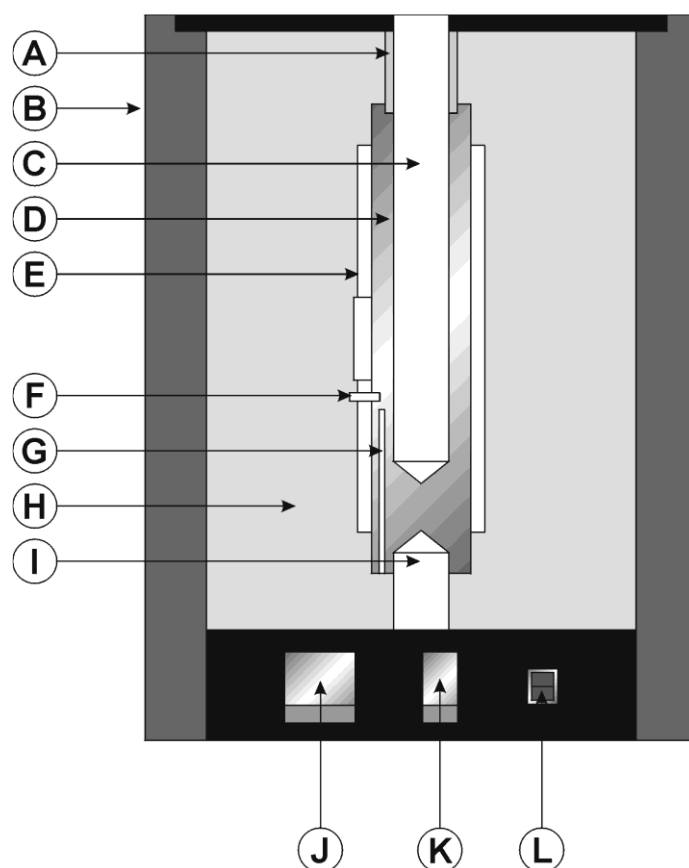


FIGURE 16: SINGLE-ZONE/LOW TEMPERATURE/VERTICAL TUBE FURNACE



- A Ceramic tube
- B Metal cabinet
- C Furnace cavity: internal diameter 54mm (2.12"), cylindrical depth 368mm (14.5")
- D Furnace block
- E Parallel wire heater (including thermal fuse)
- F PRT for temperature control
- G Over-temperature detection thermocouple
- H Fiberfrax insulation (chopped bulk fibre)
- I Ceramic support post
- J Temperature controller
- K Over-temperature controller
- L On/Off power switch

REALISING THE FOLLOWING FIXED POINTS: INDIUM, LEAD, ZINC

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 rod of quartz at ambient temperature. 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).

Depending upon factors such as furnace control and the number of thermometers successively loaded into the cell, plateau durations between an hour and many hours may be achieved. Thermometers may be preheated prior to transfer to the cell. A pocket is provided in the furnace for this purpose. It is wise to ascertain from time to time that the plateau is still in existence, by checking the cell temperature with the monitoring thermometer at intervals within the measurement sequence. Let us suppose that Th_m is the monitoring thermometer and $Th_{1...n}$ are thermometers to be calibrated. If $n=2$ a suitable sequence might be

$Th_m, Th_1, Th_m, Th_2, Th_m$

and if $n=4$

$Th_m, Th_1, Th_2, Th_m, Th_3, Th_4, Th_m$

The Th_m measured last should be equal in indication to that of the Th_m measured first to ensure that the plateau has been present for Th_1 , etc.

At the temperatures of Indium, Lead and Zinc it is generally permissible to withdraw a thermometer, of type Isotech 909, directly into room temperature.

FREEZING THE TIN CELL

Realisation of the Tin plateau is accomplished in a manner similar to those of Indium, Lead 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 quartz rod from ambient temperature. 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.

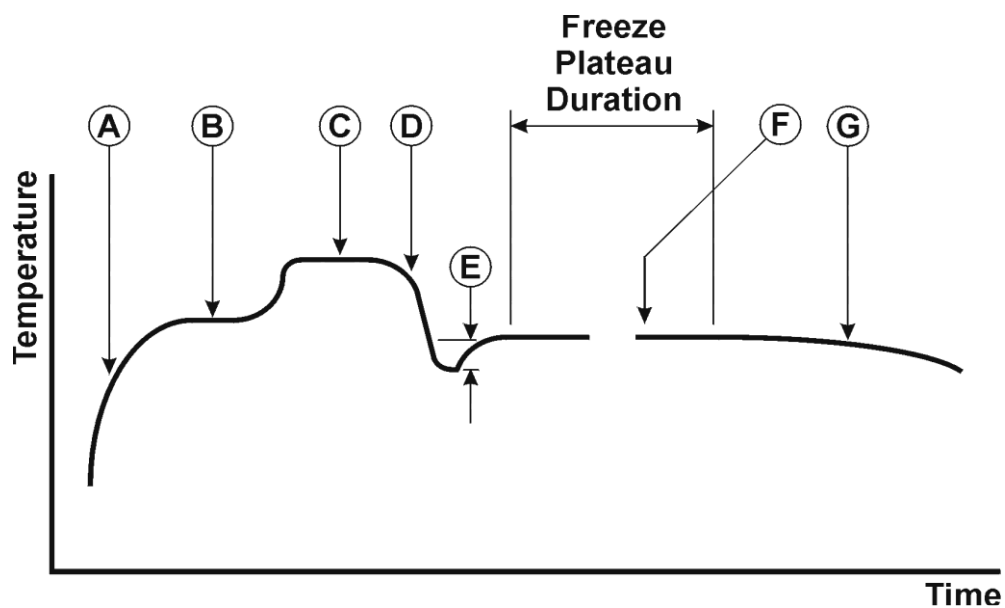
A typical melt/freeze sequence is shown in Figure 17.

A useful hint

When first creating freezes use large under settings - typically 3 to 5°C BELOW the freeze plateau. The result will be a shorter freeze 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 freeze temperature to increase the plateau length.

There follows later a tutorial about Fixed Points.

FIGURE 17: TYPICAL MELT/FREEZE SEQUENCE



- A Initial temperature rise
- B Temperature arrest during melt
- C Furnace controlled temperature
- D Temperature drop caused by furnace controller adjustment
- E Depth of supercool (particularly pronounced for tin)
- F Plateau showing constant temperature during freeze
- G Temperature drop after completion of freeze

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 outgasses 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.

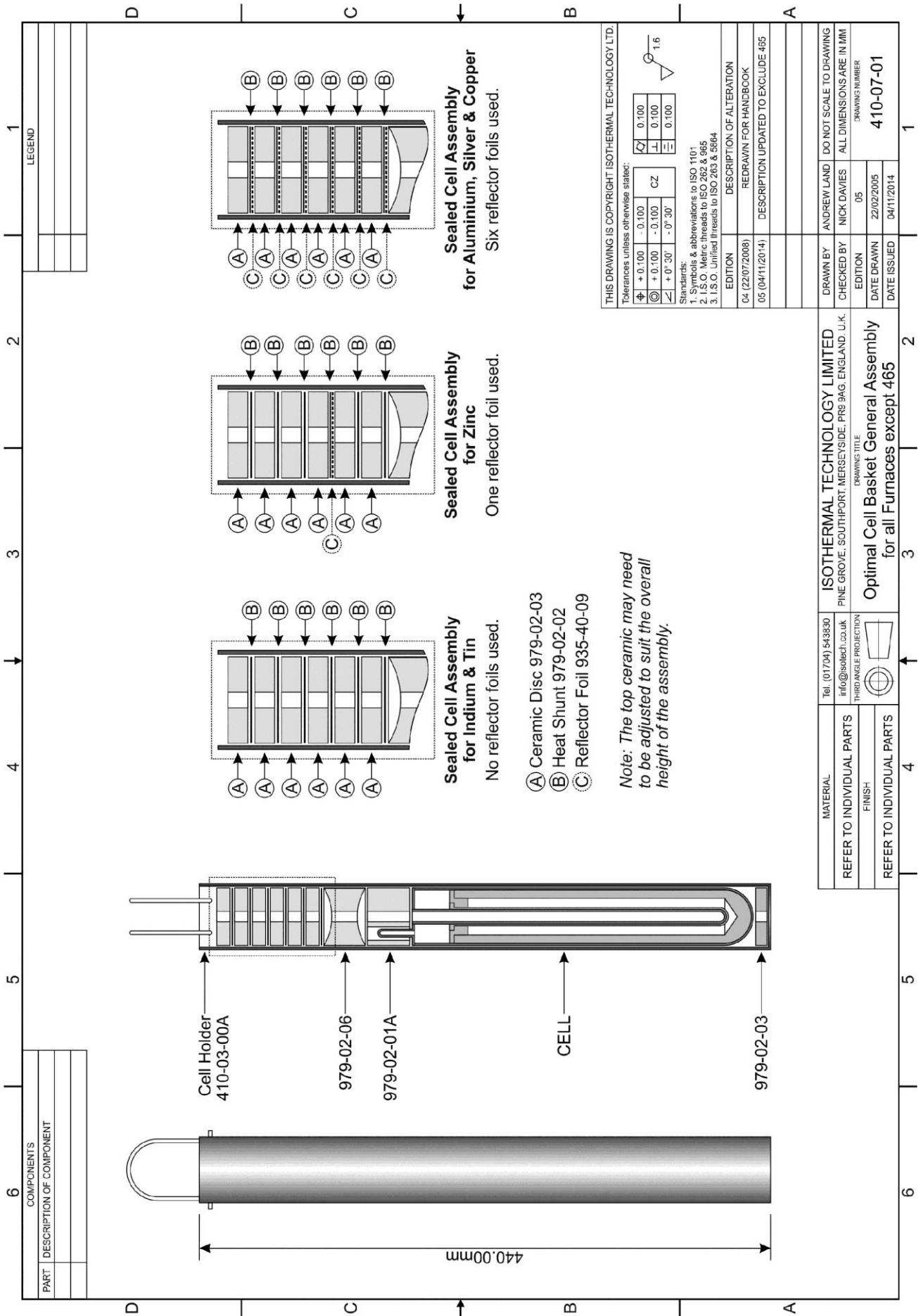
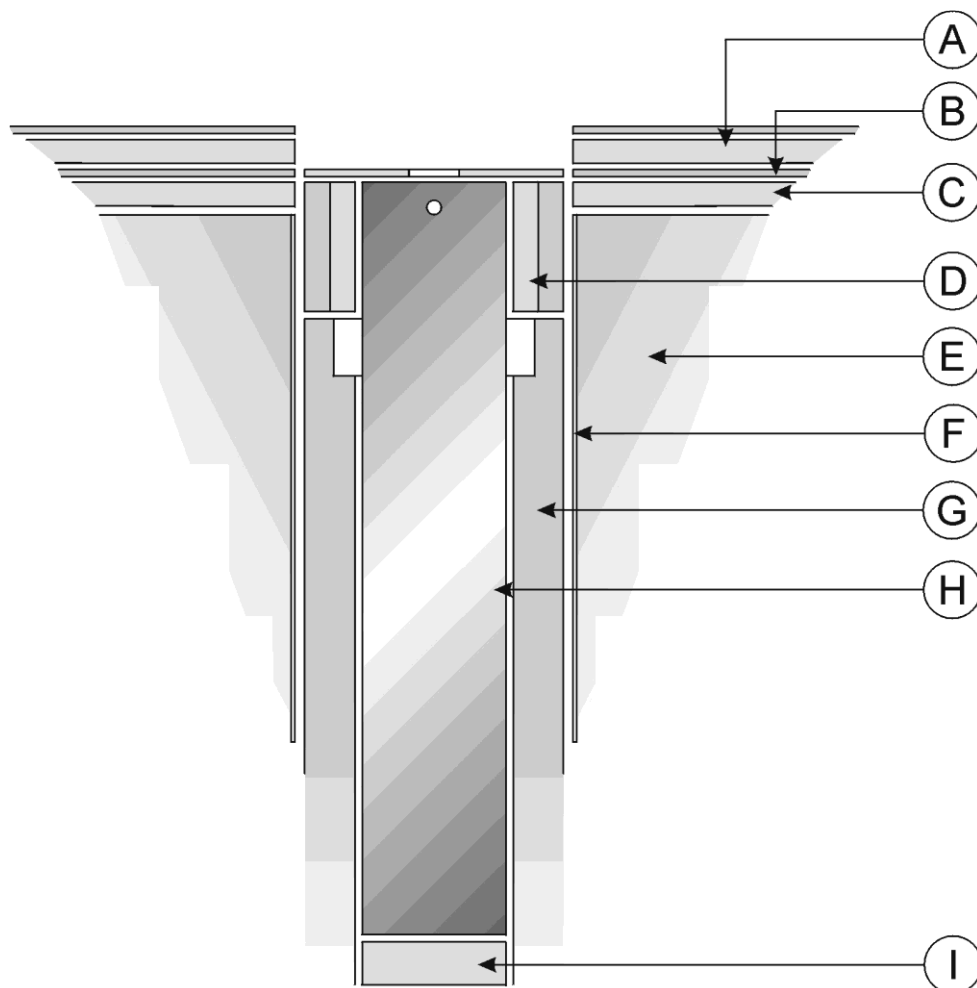


FIGURE 18: LOW TEMPERATURE FURNACE CELL ASSEMBLY



- A Fibreboard
- B Aluminium Foil
- C Fibreboard
- D Fibreblanket
- E Fibrewool
- F Fibreblanket
- G Furnace Block
- H Basket
- I Kaowool Disc

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 (see Defining Fixed Points and Related Data table) 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 1 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 the 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 Defining Fixed Points and Related Data 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	ITL 811	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
TIN FP	231.928	505.078	+3.3	+2.2	ITL-M-17669	ITL-M-17701/3/4
ZINC FP	419.527	692.677	+4.3	+2.7	ITL-M-17671	ITL-M-17701/2*/3
ALUMINIUM FP	660.323	933.473	+7.0	+1.6	ITL-M-17672	ITL-M-17702**
SILVER FP	961.78	1234.93	+6.0	+5.4	ITL-M-17673	ITL-M-17702**

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.

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.

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. Once the cell is in the basket there may be no future need to remove it. 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.

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.

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.

METAL CLAD CELLS

Devitrification does not apply to these cells, but in any case, it is good practice to clean these in the manner described above prior to 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.

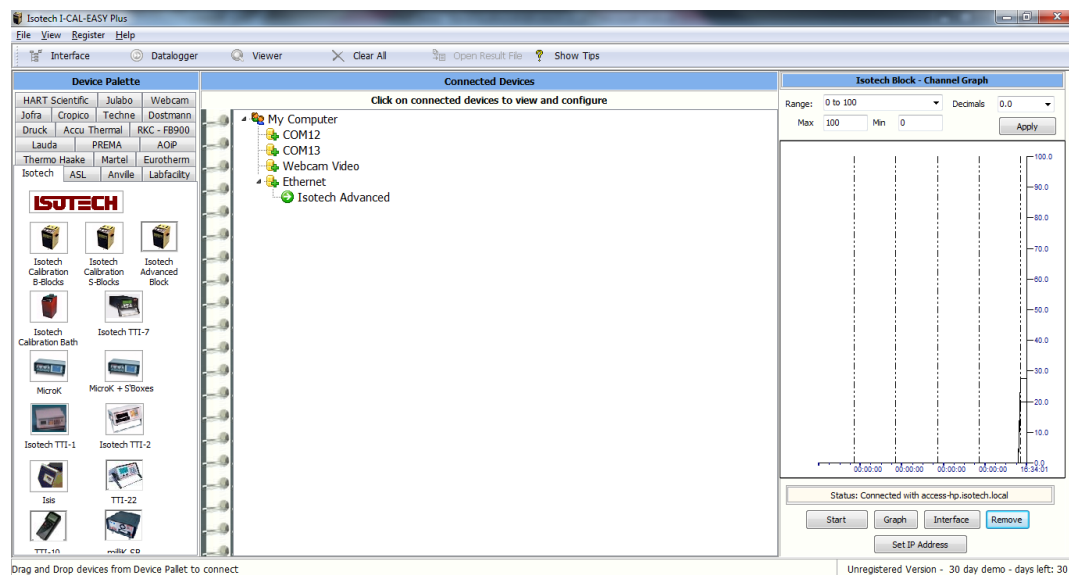
Training is available to customers at an agreed daily rate.

ISOTECH I-CAL EASY

This application allows logging of data from the furnace. Additionally, if a license is purchased it allows for fully automatic calibration including certificate printing and the calculation of coefficients.

Refer to the Isotech I-Cal EASY handbook and help system for more details

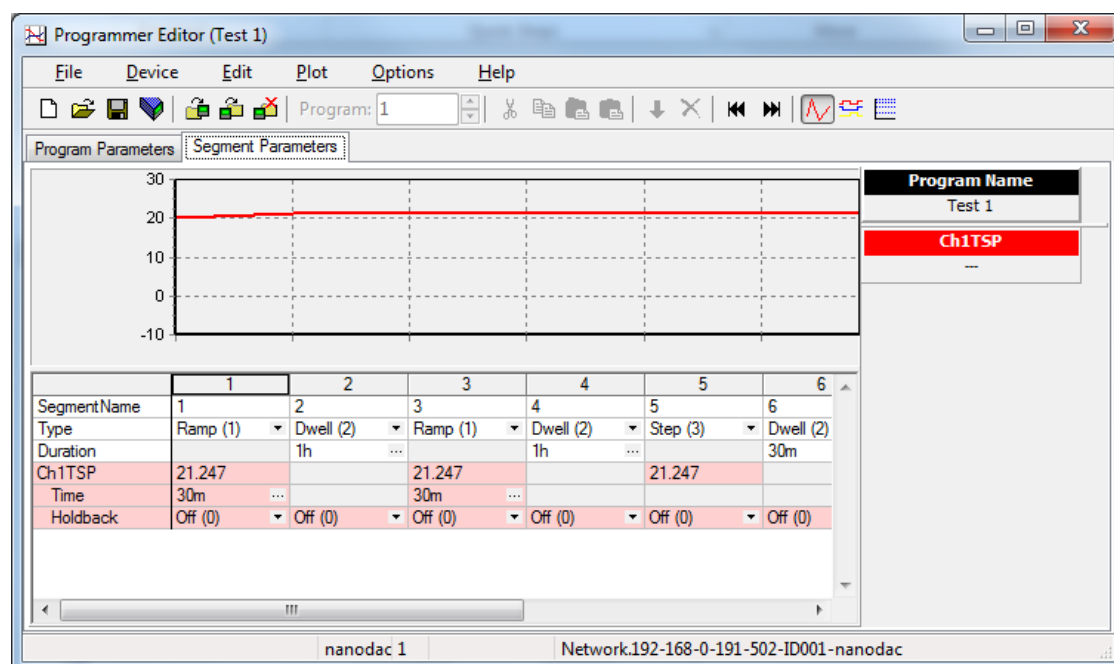
Figure 19



PROGRAM EDITOR

This allows set point programs to be created on a PC and uploaded to the furnace.

Figure 20

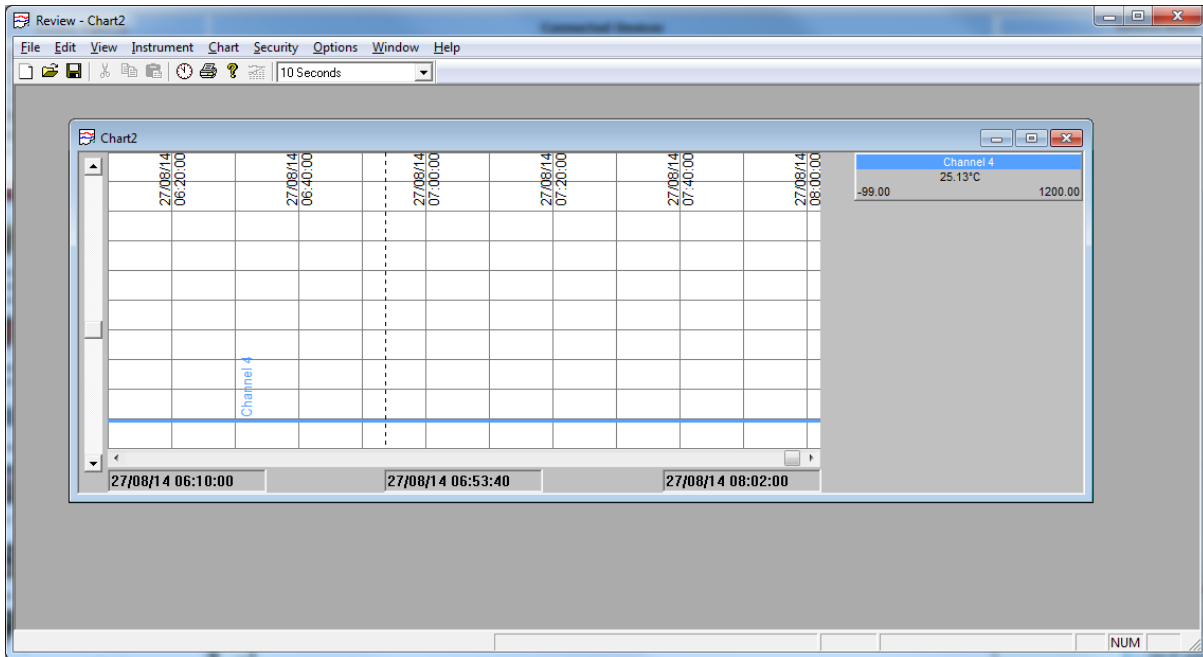


Installation instructions are included with the download

REVIEW

This is a proprietary software package which allows the user to extract 'archive' data from one or more furnaces and to present this data on a host computer, as if on a chart, or as a spreadsheet.

Figure 21



As described in the Review help system, 'Review' allows the user to set up a regular transfer of data (using ftp) from connected instruments into a database on the pc, and then from this database to the chart or spreadsheet. The chart/spreadsheet can be configured to include one or more 'channels' from one or all connected instruments.

It is also possible to archive instrument history files to a memory stick and to use this to transfer the data to the pc.

The user name and password are both 'history and they are not editable.

These applications can be downloaded from the Isotech website <http://www.isotech.co.uk/downloads>

Connection to the unit is via the Ethernet interface, configure the IP address to suit your network and then select that IP address in the software. Consult your network manager for assistance if required.