WATER TRIPLE POINT MAINTENANCE BATH MODEL 803

Isotech North America
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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. Failure caused by misuse is not covered. 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 PROPERLY TO MAINTAIN THIS INSTRUMENT MAY INVALIDATE THIS GUARANTEE

RECOMMENDATION

The life of your ISOTECH instrument will be prolonged if regular maintenance and cleaning to remove dust and debris is carried out.

SERIAL NO:.......................................

DATE:...

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CE

EMC INFORMATION

This product meets the requirements of the European Directive on Electromagnetic Compatibility (EMC) 89/336/EEC as amended by EC Directive 92/31/EEC. To ensure emission compliance please ensure that any serial communications connecting leads (RS232 or RS422 or 485) are fully screened.

The product meets the susceptibility requirements of EN 50082-1, criterion B.

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.

HEALTH AND SAFETY INSTRUCTIONS

1. Read all of this handbook before use.

2. Wear appropriate protective clothing.

3. Operators of this equipment should be adequately trained in the handling of hot and cold items and liquids.

4. Do not use the apparatus for jobs other than those for which it was designed, ie. 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.

GENERAL INFORMATION

SYSTEM DESCRIPTION

The Water Triple Point Bath is designed to provide an environment in which up to three Water Triple Point Cells can be maintained for use as primary standards of temperature. The life of the ice mantle in a triple point cell is dependent upon the initial geometry of the mantle, the use of the cell and the temperature of its environment. The bath has sufficient stability to maintain a mantle for six weeks or more, provided that the cell is not thermally overloaded.

The bath contains water which is stirred by a magnetic follower. The temperature of the water is maintained at 0.010°C by four thermoelectric cooling modules. The current through the cooling modules is controlled by a proportional controller which uses a precision, high stability, thermistor probe to sense bath temperature.

The cooling system has sufficient cooling capacity to bring the bath to operating temperature from 25°C in about two days. The limited cooling capacity provides some protection for the cells in case of control failure, since the water in the bath would freeze only very slowly. Additional protection is provided by a fault detection system which is triggered if either of the cooling surfaces drops below a preset limit or by the formation of ice on the cooling surfaces of the bath. Triggering of either fault detector will, after a built-in delay, activate the fault alarm and remove power from the coolers until the system is reset by turning the power switch off and back on.

SPECIFICATIONS

Size:	800mm high, 400mm wide, 620mm deep
Weight:	Approximately 66kg dry
Acceptable Ambient Conditions:	
Operating:	Temperature 15°C to 26°C; relative humidity 10% to 80%
Storage:	Temperature -30°C to 55°C; relative humidity less than 85%
Power Requirements:	115 or 230v AC, 50/60 Hz, 150 VA max
Bath Fluid:	De-ionised water, approximately 34 litres (7½ gal)
Bath Temperature:	0.010°C nominal (adjustable -0.5°C to +0.5°C, approximately) ±2mK/month
Weight:	45Kg

OPERATING INSTRUCTIONS

INSTALLATION

LOCATION

The typical industrial standards laboratory provides an environment suitable for the bath, which is designed to operate at an ambient temperature between 15 and 26°C, in air that is relatively free of particulate material (particularly soluble salts) and soluble or corrosive gases such as ammonia or sulphur dioxide. The clearance necessary for cooling air to circulate through the back of the cabinet is provided by the extended frame around the air filter. This frame may be placed directly against a wall as long as the lower opening is not obstructed. The cabinet is equipped with castors so that it may be moved easily; however, care must be taken to avoid splashing water out of the bath. Power requirements need no special consideration; maximum power consumption is 150VA, and interruptions of a few minutes will not affect the condition of the cells because of the large thermal inertia of the bath.

START UP

1. Visually inspect the system to ensure that it has not been damaged in transit.

2. Connect the mains cable to a suitable supply socket outlet and turn the power switch on. The fault alarm light should light steadily for several seconds, then fade off, and the cooler voltage meter should indicate about 20V.

   NOTE - All controls are factory-set for proper operation.

3. Fill the tank to the top of the cell holder tubes with clean, de-ionised water.

4. Monitor the water temperature. Without ice, the temperature should drop about 0.6°C per hour (from 20°C).

5. When the cooler voltage drops below its "full voltage" level of about 20V, the bath is ready for use.

OPERATION

Three openings are provided in the lid of the bath to permit access to the cells. The openings should be kept closed with the split-plugs provided (these have thermometer channels so that closure may be maintained during use). The bath does not have sufficient cooling capacity to operate with the top open to ambient air, and must be covered with at least one layer of plastic film if it must be operated with the lid open.

The water level in the bath should just cover the top of the main body of the cells. Whenever the number of cells kept in the bath is changed, the water level should be adjusted to compensate. The water level and air filter should be checked weekly and maintained correctly. ***See Maintenance.***

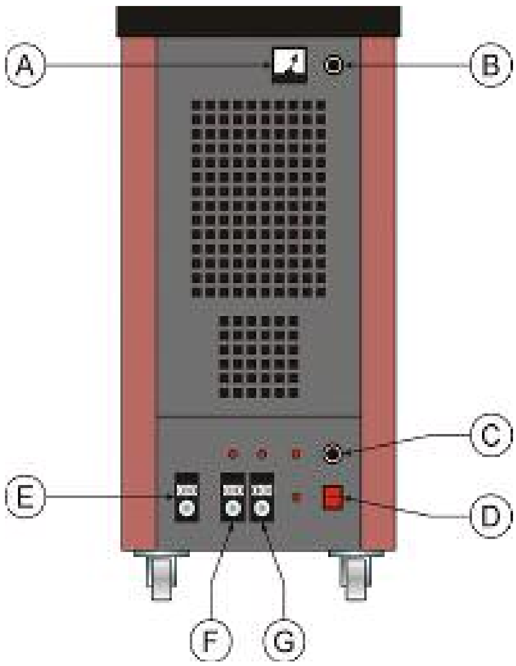
During normal operation, the "cooler voltage" meter provides a general indication of system function. The normal range is 10 to 17V, with higher voltage required at higher ambient temperature and humidity. Any change in cooler voltage which does not correspond to ambient conditions indicates a potential malfunction. See Maintenance.

Some micro-organisms are capable of growing in water near 0°C. Cloudy water and/or slimy surfaces in the bath may indicate such growth. See Maintenance.

Due to the long cool down time we recommend that the bath is left running continuously; this maximises the life of the bath.

Internal trips and temperature alarms protect the system and running costs are very low. We do not recommend powering the bath up then down for short periods of operation.

A, Cooler Voltage
B, Air Flow
C, Audible Alert
D, On/Off switch
E, Main Set point
F, Alarm 1
G, Alarm 2



CONTROLS AND INDICATORS

On the cabinet front is a control panel containing the power switch, "POWER" and "ALARM" indicator lights and an adjusting potentiometer dials for the controller set point and the two fault detector trigger points, indicator lights for "POWER", for "ALARM" and for both fault detector circuits, and an audible alarm device.

The air flow control adjusts the amount of air pumped to the stirring tubes. It should be adjusted to the minimum value that will reliably maintain a flow through both tubes.

The power switch on the control panel serves the normal function of a system power switch and also serves as a system reset following a fault condition. Resetting the system is accomplished by turning the power off for several seconds, then turning it back on. The "POWER" indicator light indicates only that power is applied to the controller circuit, not that it is functioning. The "ALARM" indicator light indicates a fault condition only when it is flashing; it will light steadily for a few seconds after power is turned on while the system performs its automatic power-up reset.

The fault detector circuits trigger only on an under-temperature condition, since that is the only condition which is potentially destructive to the cells kept in the bath. About 20 minutes after either fault detector is triggered, power to the coolers will be cut off until the system is reset.

The controller "SET POINT" adjustment has a span of 1 degree Celsius (1 kelvin) for dial readings of 000 to 999. Thus the nominal dial setting for 0°C would be 500; however, only the relative calibration of 1mK per dial digit can be considered relevant, since a significant portion of the span is used by component tolerances. Both fault detector "TRIGGER POINT" adjustments are calibrated in the same way as the controller set point. Each trigger point adjustment has an associated indicator light which lights as soon as that circuit detects an under-temperature condition. The output of each fault detector has a delay of about 20 minutes, so that the system fault alarm and cooler shutdown are not triggered by transient conditions. The power "ON" and fault alarm lights are duplicates of those on the control panel. The audible alarm device is activated whenever the system fault alarm is triggered; it emits a 2kHz beep for about 0.15 second each second.

MAINTENANCE

ROUTINE MAINTENANCE

Any change in cooler voltage which does not correspond to ambient conditions indicates a potential malfunction. An increase in cooler voltage could be caused by a dirty air filter or other obstruction to air flow, or by excessive air leakage around the lid gaskets, or by reduced flow of stirring air. A decrease in voltage could indicate low water level or a controller malfunction. The maximum voltage available to the coolers is approximately 20V. Any of several conditions may cause the controller to apply full power to the coolers.

Full power is required for up to 48 hours to cool the bath from ambient to operating temperature. The controller will normally apply full power for several minutes if water is added to the bath, or if any warm object is placed into the bath. Ice formation on the cooling surfaces will cause the controller to apply full power as the water releases its heat of fusion, eventually resulting in an under-temperature fault as the thermal resistance of the ice layer increases. Failure of the cooling fans or total obstruction of the cooling air will result in a full power condition, which would eventually open one or both of the thermal fuses on the cooling module heat sinks as the bath temperature increases towards ambient.

NOTE: The cooler voltage meter will indicate the residual thermoelectric potential of the cooling modules, (about 2V with the bath 20°C below ambient) even with power disconnected. The meter will read true zero only with the 12-contact "Cinch" connectors on the rear panel of the controller disconnected.

Check the water level weekly.

Cloudy water and/or slimy surfaces may indicate growth of micro-organisms. The bath may be disinfected by adding about 0.5 litre of household bleach (5% sodium hypochlorite), and leaving it in the bath for a few hours, then changing the water in the bath. Since the bleach is corrosive, all metal surfaces, including the lining of the access holes, should be rinsed with de-ionised water after disinfecting.

CALIBRATION

Calibration of cells is not required since the water triple point temperature is a constant of nature. However, proper adjustment of the bath will optimise the life of the ice mantles in the cells. (See Appendix B for a procedure to verify the integrity of the cell).

ADJUSTMENT

Adjust the bath temperature to 0.010°C using the following procedure. This procedure requires an operating water triple point cell and a thermometer with a resolution and short-term stability of 0.002K (2mK) or better.

1. Adjust the stirring air flow control on the front panel for the lowest flow rate that will reliably maintain flow through all of the holes in both stirring tubes.

2. Calibrate the thermometer at 0.010°C in the triple point cell.

3. Place the thermometer in the bath adjacent to one of the cell holders, or in one of the pre-chill tubes.

4. Allow the thermometer to stabilise for about ½-hour, and determine the temperature difference between the bath temperature and the water triple point (0.01°C).

5. Observe and record the setting of the "controller set-point".

6. Adjust the "controller set-point" dial to eliminate the difference. (For example, if the bath temperature is 3mK below the triple point, and the dial setting is 524.4, set the dial to 527.4. Note that each dial digit represents 1mK and each digit is divided into 5 linear increments).

7. Allow the bath to stabilise (as indicated by the cooler voltage) and repeat steps 4 to 6 if necessary to make the bath temperature equal to the triple point within the readability of the thermometer or 1mK. (Note that the bath has a thermal time constant of about 20 minutes due to its large mass and limited cooling capacity).

8. Observe and record the settings of both fault detectors "trigger point" dials.

9. Unlock one trigger point dial and increase its setting until the indicator light lights.

   NOTE: Do not keep the light on for more than a few minutes at a time. (The system fault alarm circuit has a delay of approximately 20 minutes and a reset time of less than 10 seconds).

   Observe and record the dial setting for the off-to-on transition of the light.

10. Set the dial 100mK below the transition setting. (The trigger point dials are calibrated for 1mK per least significant digit, the same as the controller set point).

11. Repeat steps 8 and 9 for the other trigger point dial, and record the new settings of all three dials for future reference.

12. Test the fault alarm and shutdown logic by setting one trigger point dial a few mK above its transition setting. Within 30 minutes the system "FAULT" indicator should begin flashing and the alarm signal should begin beeping. The cooler voltage should drop to approximately 2 volts as soon as the system fault alarm is triggered. (Under these conditions, the "COOLER VOLTAGE" meter is reading the residual thermo-electric voltage generated by the coolers). Reset the trigger point dial to the setting recorded in step 10, and lock it.

13. Reset the system by turning the power switch off for a few seconds and then turning it back on. The "FAULT" light should light steadily for a few seconds then fade off each time the system is reset.

14. Repeat steps 11 to 13 with the other trigger point dial.

THEORY OF OPERATION

REFRIGERATION

Refrigeration is provided by four thermoelectric modules. These modules are sandwich structures of ceramic, copper and semiconductor material. They use the Peltier effect of the semiconductor material (bismuth telluride) to produce a current-dependent heat pumping action. Each thermoelectric module is mounted between a heat sink and an aluminum block. Two of these assemblies are mounted to an aluminum plate attached to the lower part of each side of the stainless steel tank. The cooled area of each side is about 230 by 280mm (9" x 11"). The tank is embedded in moulded foam insulation which has been formed to provide air channels over the heat sinks and across the bottom to the fans located near the centre of the horizontal panel that supports the tank assembly. The cooler circuit has two thermal fuses in series with the four modules to protect the system in case of fan failure. These fuses are mounted on one heat sink on each side of the tank assembly. Current through the thermo-electric modules is regulated by the controller circuitry.

Three precision thermistor probes monitor the temperature of the tank and the water. Two of these probes are inserted into wells from the rear of the tank assembly. These probes are connected to the fault detector circuits. The other probe is the main control probe and is mounted to the top panel of the tank assembly such that it extends down into the water in the bath.

CONTROLLER

POWER SUPPLY

The +27V supply provides power to the thermoelectric cooling modules and the audible alarm, the +5V regulated supply provides logic power, and the analogue circuits operate from the -6V and +24V regulated supplies. Note that the main control circuit has separate -6V and +24V regulators to provide additional decoupling from the other circuits, and to improve controller reliability.

MAIN CONTROL CIRCUIT

The main control circuit (schematic diagram, reference number 450-20-01) is a precision DC linear proportional controller. The controller input is basically a wheatstone bridge circuit consisting of R101 thru R106 and the main control probe. The control probe has a nominal resistance of 10k ohms at 25°C and 29.49k ohms at 0°C. Bridge current of 36 microamps is provided through R108, producing a bridge voltage of approximately 0.95V at normal operating conditions. The voltage at TP3 (the input of the first amplifier, U101) is nominally 0.52V with a change of -23 millivolts/kelvin [mV/K]. Negative feedback around U101 is provided through R107 and C101, giving a DC gain of the approximately 100 and a low-pass cut off frequency of about 3Hz. The output of U101(TP4) is nominally 0V at null with a change of -2.3V/K. The output amplifier (U102) has no resistive feedback, and thus has a DC gain of the order of 10,000. However, negative feedback is provided through C103 so that the AC gain is limited to about 250. Another way of looking at the output amplifier is to consider it a proportional-plus-integral controller with a proportional band equivalent to about 35mK and a reset time of about 20 minutes. C103 is a special low-leakage tantalum capacitor. Note that D101, R111 and R112 assure that the bias on C103 is always the correct polarity. The output of U102, through R113, drives Q101, a Darlington power transistor connected as an emitter-follower to control voltage to the thermoelectric coolers from the 27V supply.

The tank assembly has four thermoelectric coolers and two thermal fuses (72°C) all in series. The DC resistance of this circuit is about 8 ohms at room temperature. Note that the resistance would be difficult to measure when the bath is cold due to a thermoelectric voltage of as much as 2V being generated by the coolers.

FAULT DETECTOR CIRCUITS

The fault detector circuits are two identical under-temperature sensor circuits that provide an immediate indication of an under-temperature condition, then trip the system alarm and shut down the coolers after a delay of about 20 minutes. One of these circuits is described below. The input is a wheatstone bridge consisting of a probe and R201 through R205. This circuit is the same as the main controller circuit, except that no negative feedback is provided. Amplifier U201 operates as an open-loop voltage comparator; its output goes positive as the probe temperature goes below the limit set by front panel control R205. A positive output from U201 through R207 and D201 turns on Q201 to light fault detector indicator D203. This also applies a positive voltage of about 1.2V to the delay circuit R208 and C202. As C202 charges, the voltage at the gate of Q202 rises from -5V towards 0V, which, after about 20 minutes, turns Q202 on, thus tripping the system alarm logic and cutting power to the coolers. The gate of Q202 is protected from turn-off transients by R210. Note that D202 quickly discharges C202 if the output of U201 goes negative, thus erasing the effect of transient fault conditions. The fault detector probes are located at the upper edge of the cooling plates on the sides of the bath; thus a brief under-temperature indication may occur during transient load recovery, particularly when ambient temperature is high.

SYSTEM ALARM LOGIC

The alarm logic section controls cooler shut-down and audible and visual alarm signals. The alarm logic is reset by the power-on reset signal from Q401.

The gate of Q401 is held negative by C401 for several seconds after power-on to allow time for the operator to verify operation of the fault alarm lights, D404 and D405. The reset signal from Q401 is inverted by U402-B to reset the R/S flip-flop consisting of U401-A and B. U402-A and E are the buffer and driver for Darlington power transistor Q402, which cuts power to the coolers if the system alarm is tripped. Timer U403 is set up for an output of 1 pulse/second at 15% duty. This is used through gates U401-C and D to drive the audible alarm at 15% duty, and the fault alarm lights at 85% duty. Zener diode D403 protects the audible alarm from over-voltage since the 27V supply is relatively unloaded whenever the alarm is sounding.

SCHEMATIC NOTES

1. Fuse. F1 is a slow blow type (Buss MDL or equivalent). It is 1½ A for 115 volts, or 750mA for 230 volts.

2. Resistances are in ohms. K = 10^3, M = 10^6.

3. Capacitors C103, 202 and 302 are special low-leakage tantalum (Sprague Type 109 D). The effect of leakage of C103 is about 0.16 kelvin/microamp.

4. Capacitances are in microfarads, except "p" indicates picofarads.

5. Numbered circles (Drawing No. 450-20-01) indicate the boundaries of the pc board.

6. Numbers in square brackets (Drawing No. 450-2-01) indicate wire colours.

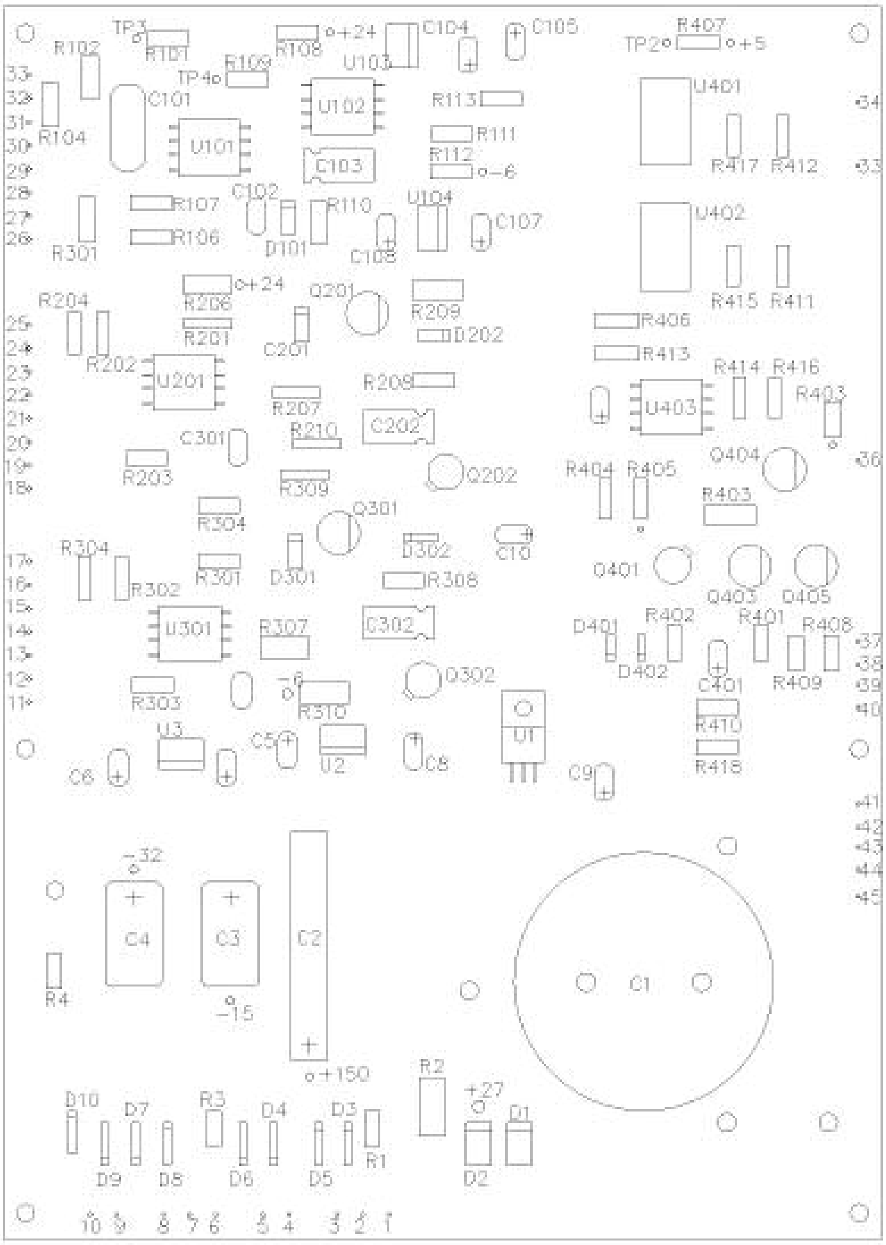
7. U401 is a 4011 CMOS quad NAND gate, U402 is a 75492 hex open collector inverter/buffer, and U403 is the CMOS version of a 555 timer. (A standard 555 could be substituted for U403 if necessary).

8. Connect 3 pin neutrik plug on the back of the controller chassis. The three probe connectors (J1-J3) are identical. Make sure that the probe cables are properly tagged before disconnecting them. (The probes are, in fact, interchangeable except for calibration).

9. Test points are designated component leads; see the component layout for physical locations.

   TP1 is the system reset line and is low during normal operation. It is high for several seconds after power-up.
   TP2 is the alarm trip and is normally high. It will go low after about 20 minutes of continuous fault indication by either of the fault detector circuits.
   NOTE: Connecting a meter to this test point will probably trip the alarm. This can be avoided by using a 100K ohm resistor between the meter probe and the test point, then calculating the voltage from the meter reading and its input resistance.
   TP3 is the main control probe. The voltage at this point will be about 540mV during normal operation, or about 400mV with the probe at room temperature. The voltage change is about -24mV/°C at the normal operating point.
   NOTE: Connecting a meter to this test point will probably disturb the operation of the controller.
   TP4 is the output of the main controller first amplifier, and is nominally 0V with a change of about 2.5V/°C at the operating point.

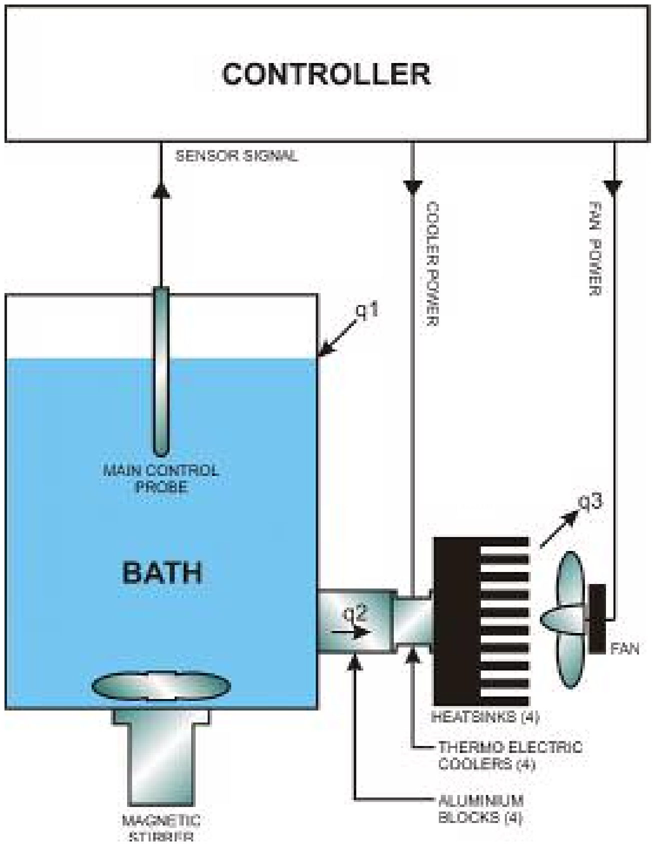
10) The air-flow controller (R5) is located on the rear panel of the controller chassis, adjacent to the air hose connection.



1 RED
2 RED
3 YELLOW
4 ORANGE
5 ORANGE
6 GREEN
7 GREEN
8 YELLOW
9 BROWN
10 BROWN
11 BLUE
12 RED
13 SHIELD
14 SHIELD
15 BLUE
16 YELLOW
17 RED
18 WHT/ORG
19 BLUE
20 RED
21 SHIELD
22 SHIELD
23 BLUE
24 YELLOW
25 RED
26 WHT/RED
27 SHIELD
28 BLUE
29 RED
30 SHIELD
31 BLUE
32 YELLOW
33 RED
34 BROWN
35 BLUE
36 WHT/YEL
37 WHT/BLUE
38 BLUE/WHT
39 WHT/GRN
40 GRN/WHT
41 RED
42 RED
43 BLACK
44 YELLOW
45 YELLOW

NOTE: TEST POINTS ARE DESIGNATED COMPONENT LEADS AS INDICATED. VOLTAGES ARE REFERRED TO CHASSIS COMMON. SEE 'SCHEMATIC NOTES' FOR DESCRIPTION OF TEST POINT SIGNALS.

REFRIGERATION DIAGRAM:



q1 = Rate of heat flow from ambient to the bath.
q2 = Heat flow from the bath to the four thermo electric coolers.

(This is the same rate of heat flow as q1, but at about 0°C).

q3 = Heat flow from the heat sinks to ambient. (This is equal to q2 + P, where P is the electrical power used to drive the thermo electric heat pumps).

APPENDIX A

AN INTRODUCTION TO THE WATER TRIPLE POINT

The International Temperature Scale of 1990 (ITS 90), the scale most used in science and industry, is defined by a series of fixed points. Defining fixed points are two-phase or three-phase equilibria of ideally pure materials to which temperature values are assigned. These values are derived from fundamental methods of thermometry and are considered to be exact by definition. Two-phase equilibria may be solid-liquid, liquid-vapour, or solid-vapour. All are pressure-dependent, at least to some extent, with those involving the vapour phase having much larger pressure coefficients than solid-liquid equilibria. Equilibria in which all three phases are present (triple points) define fixed temperatures, the pressure is, in each case, uniquely defined by the simultaneous existence of all three phases of one material in the absence of other influences (the vapour pressure of the material in the containing vessel will be negligible as will be its dissolution in the thermometric material).

Of all defining fixed points, the triple point of water (the equilibrium point of liquid and solid water under its vapour pressure) is the most important for both theoretical and practical reasons.

1. The triple point of water is the single defining point of the Kelvin Thermodynamic Temperature Scale (KTTS) on which ITS-90 is based and is the accepted reference temperature for SPRT characteristics.

2. The triple point of water is, at present, the most accurately realisable of the defining fixed points. Properly used, it is not difficult to realise the temperature of the triple point, 0.01°C, with an accuracy of ±0.0001°C. (To put this in context, it is difficult to prepare and use an ice bath with accuracy of better than 0.002°C).

3. Computer-interpolated calibration printouts which accompany an SPRT provide tables of temperature versus the ratio of resistance of the thermometer at that temperature to its resistance at the triple point of water:

   $$W(t°C) = R(t°C)/R(0.01°C)$$

   For measurements of the highest precision, proper technique for use of an SPRT is as follows:-

   a) Measure the thermometer resistance at temperature required.
   b) Measure the thermometer resistance at the water triple point.
   c) Calculate the ratio by dividing (a) by (b).
   d) Find the temperature in the table corresponding to this ratio.

4. The water triple point is relatively inexpensive to acquire, and the equilibrium can be maintained for long periods with a minimum of effort

5. The water triple point is an excellent check and quality assurance control on SPRT's and other thermometers. If a thermometer resistance remains constant at the water triple point, it is probably in calibration at all temperatures within its range.

We at Isothermal Technology Limited, Southport, manufacture a range of Water Triple Point Cells which are designated as E11 (11mm bore) with McCleod gauge and C12 (with 12mm bore).

The illustration overleaf shows the configuration and dimensions of cells available from Jarrett-Isotech.

The Isotech Water Triple Point Bath, as furnished, will accommodate A-11 (11mm bore) and A-13 (13mm bore) cells; the cell holding tubes will accommodate B-16 and B-19 cells with modification of the top bushing and bottom plug. Accommodation of B-22 cells requires a different cell holder. Please call Isotech for information on special cell holder.

TYPE A CELLS (Fig 1) were designed by Dr. H. F. Stimson at NBS. A tubular glass extension at the top of the cell serves as a convenient handle for lifting and carrying the cell, as a hook for supporting it in an ice bath, and as an indicator of partial pressure of air in the cell.

TYPE B CELLS (Fig 2) were designed at NRC of Canada. The thermometer well extends 100mm above the top of the cell.

FIGURE ONE

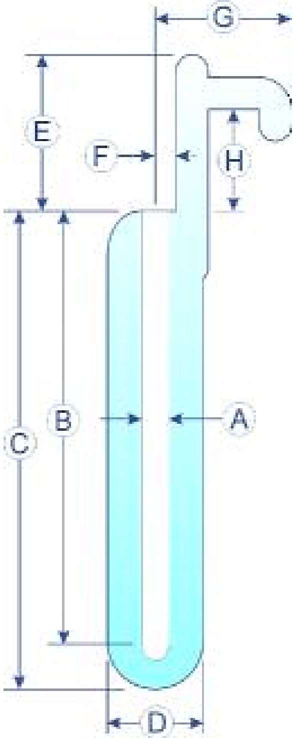
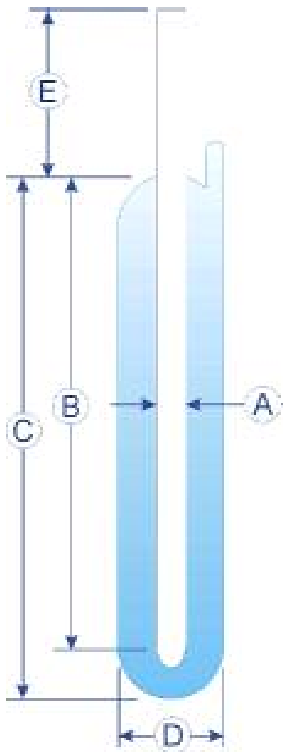


FIGURE TWO



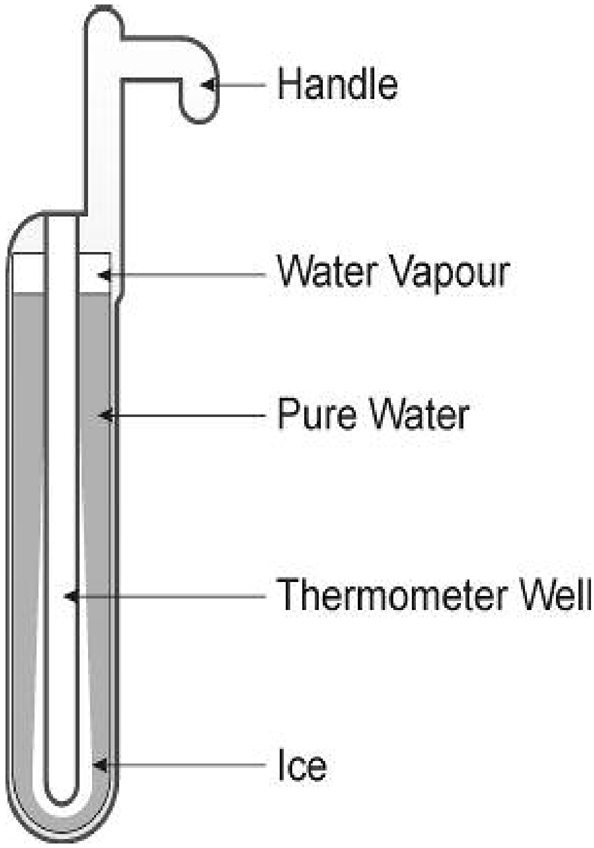
Standard or Special (ST/SP)	Model Fig.	Number	A*	Nominal Dimensions in Millimetres B	C	D	E	F	G	H
ST	1	A-11	11	317	348	51	104	14	93	70
SP	1	A-13	13							
ST	2	B-11	11	317	348	64	100			
SP	2	B-13	13							
SP	2	B-16	16							
SP	2	B-19	19							
SP	2	B-22	22							

*Precision bore tubing

APPENDIX B

TRIPLE POINT CELL OPERATION

The illustration below shows a water triple point cell .



The ice mantle may be frozen in one of several ways. The procedure for the most common of these ways is as follows:

1. Pre-chill the cell in a bath of ice and water or in the Isotech Water Triple Point Bath.

   NOTE: The large thermal mass of a cell, coupled with the limited cooling capacity of the bath, will produce a significant disturbance in bath temperature when a cell is cooled from ambient temperature in the bath. Therefore, if the bath contains an operating cell, the cell to be frozen should be cooled to about 4°C in an ordinary refrigerator before putting it into the bath. If a room-temperature cell is put directly into the bath, the thickness of the mantle at the bottom of an operating cell may be reduced by about 1mm.

2. Carefully invert the cell to drain the thermometer well

   C A U T I O N - Tilt the cell very carefully through the horizontal; the flowing of water from one end of the cell to the other will cause vapour bubbles to collapse and reform. The collapse of vapour bubbles produces a sharp clicking sound, and the glass can be broken if the water is moving rapidly.

3. Fill the thermometer well with alcohol and drain it again to remove the remaining water.

4. Put several drops of alcohol into the well (so that it contains about 1ml). (The alcohol serves as a heat transfer fluid during the freezing of the mantle).

5. Fill the thermometer well with crushed dry ice (solid carbon dioxide) obtained from block dry ice or by expansion from a siphon-tube tank of liquid carbon dioxide.

6. Observe the surface of the outside of the thermometer well. This will suddenly be coated with spicules of ice as ice forms from the super-cooled water surrounding the well.

7. Continue to add dry ice until the ice mantle is of a sufficient thickness (usually 4 to 10mm). The ice mantle may be made as thick as desired, with the caution that, because of the circular cross-section of the cell and the refractive index of water, the mantle appears larger than its actual size, and incremental growth of a thick mantle is difficult to observe. If the mantle grows so that it impacts the inside diameter of the outer tube, the glass may be broken. The actual size of the mantle may be observed by gently inverting the cell.

8. During the process of freezing the mantle, care should be taken to see that an ice bridge does not form between the thermometer well and the outer tube at the top of the water column. Such a bridge can break the glass. If a bridge begins to form, warm the water locally by holding it with the hand to melt the ice bridge.

   Isotech assumes no responsibility for the breakage of cells due to excess ice.

9. When the well is free of dry ice, remove as much of the alcohol as possible, and place the cell in the Isotech Water Triple Point Bath. At this time, an inner melt may be formed as described below; however, the temperature of the cell may be as much as 0.0005 Kelvin below 0.01°C, due to stress in the newly formed ice mantle. A rest in the bath of 24 hours will relieve this stress and realise the correct temperature.

   A much easier procedure for production of the mantle in a cell can be followed by making use of Isotech's heatpipe apparatus Model 452 which uses either solid carbon dioxide or liquid nitrogen. Ask for details.

During use, whilst the frozen cell is maintained in the bath, it is prudent practice to examine the cell at intervals to assure that (1) no ice bridge is formed and (2) the mantle is not even growing close to the inside of the outer wall of the cell.

The water triple point equilibrium can be maintained in the Isotech bath for long periods of time, depending upon the frequency of use and the heat added to the cell by inserted thermometers.

The ice mantle may be conveniently viewed by holding the cell at an angle in the open central portion of the bath so that the water in the bath compensates for refraction at the cell's cylindrical surface.

To measure a thermometer at the water triple point, the following is recommended:

1. Remove a cell from the bath.

2. Initially free the ice mantle by inserting a room temperature glass, quartz or aluminum rod, or a thermometer, for a few seconds. The purpose is to melt a film of water, the "inner melt", between the outside of the thermometer well and the ice mantle. When a gentle axial twist of the cell causes the mantle to spin freely, the cell is ready for use.

   CAUTION - On no account attempt to axially twist the cell unless the mantle is first freed or this can rupture the reentrant tube of the cell.

   Return the cell to the bath.

3. To prolong the equilibrium as long as possible, it is suggested that any thermometer be pre-chilled before being inserted into the cell. Pre-chill tubes are provided in the Isotech bath for this purpose.

4. During all measurements, and during the entire time the cell is maintained in the bath, the water level should be such that the thermometer well is full of bath water (Type A cell).

5. For those thermometers whose stems can act as pipes for radiant energy, particularly SPRT's with quartz tubes, there is some evidence that minor errors in temperature can be caused by the conductance of radiant energy into the cell. A source of such energy can be laboratory ceiling lights. It is good practice to cover the head of the thermometer and the cover of the bath with an opaque black cloth during measurements.

6. The temperature 0.01°C is defined at the liquid-vapour interface. This is not the location of the thermometer sensing element. Whilst the cell temperature is independent of ambient pressure, it requires correction for the hydrostatic head of the internal water. This correction is -7×10^{-6} kelvin per cm of water column height, a total, for the Jarrett A-11 or A-13 cells, of about -0.0002 kelvin.

The integrity of the cell may be established absolutely as follows. (Note that these instructions refer to a chilled cell whether or not an ice mantle is frozen in it. The phenomena will be less noticeable in a room-temperature cell).

1. Gently invert the cell. As the water column strikes the opposite end, un-cushioned by air, a distinct clicking noise will be heard. This ensures that the cell is free of air. The pressure of water vapour at the water triple point is about 4.6mm Hg.

2. Gently invert the cell so as to capture the bubble of water vapour in the handle. Continue to invert. The bubble will be compressed by the water and will shrink to the size of a small pea or less.

If these tests can be met, the cell is qualified. If not, there is probably a minute crack in the glass which has admitted air.

Transportation of water triple point cells is a matter for extreme care. It is preferable to hand carry them in the upright position. The impact of rapid motion of the water column along the cylindrical axis has been known to break cells. In any transportation, it is obvious that the cell must be protected from freezing.