

**MERCURY APPARATUS MODEL
17725 INCLUDES MERCURY
CELL MODEL 17724**

User Maintenance Manual/Handbook

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The company is always willing to give technical advice and assistance where appropriate. Equally, because of the programme of continual development and improvement we reserve the right to amend or alter characteristics and design without prior notice. This publication is for information only.


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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 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 handbook)
	IEC 417	Caution, Hot Surface

ELECTRICAL SAFETY

This equipment must be correctly earthed.

This equipment is a Class I Appliance. A protective earth is used to ensure the conductive parts cannot become live in the event of a failure of the insulation.

The protective conductor of the flexible mains cable which is coloured green/yellow **MUST** be connected to a suitable earth.

The blue conductor should be connected to Neutral and the Brown conductor to Live (Line).

Warning: Internal mains voltage hazard. Do not remove the panels.

There are no user serviceable parts inside. Contact your nearest Isotech agent for repair.

Voltage transients on the supply must not exceed 2.5kV.

HEALTH AND SAFETY INSTRUCTIONS

1. Read this entire 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, 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 without disconnecting it from the supply and leaving time for it to reach ambient temperature.
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.
13. Each apparatus is protected by an over temperature circuit. Please consult handbook for details.

GUARANTEE

This instrument has been manufactured to exacting standards and is guaranteed for twelve months against electrical break-down or mechanical failure caused through defective material or workmanship, provided the failure is not the result of misuse. In the event of failure covered by this guarantee, the instrument must be returned, carriage paid, to the supplier for examination and will be replaced or repaired at our option.

FRAGILE CERAMIC AND/OR GLASS PARTS ARE NOT COVERED BY THIS GUARANTEE

INTERFERENCE WITH OR FAILURE TO PROPERLY MAINTAIN THIS INSTRUMENT MAY INVALIDATE THIS GUARANTEE

RECOMMENDATION

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



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GENERAL INFORMATION

SYSTEM DESCRIPTION

A temperature standard based upon the triple point of mercury consists of two essential elements, a cell containing high-purity mercury and a cryostat in which to initiate a freeze or melt under triple-point conditions and then to maintain the triple point for a useful period of time. The Isotech Mercury Triple Point Temperature Standard provides both of these facilities. The Isotech Model ITL-M-17724 is a hermetic stainless steel cell as described by G Furukawa, et al. in NBS Special Publication 260-77. The Isotech Model ITL-M-17725 Cryostat is a self-contained, mechanically-refrigerated, system with a main well to house one cell and two auxiliary wells for pre-chilling of thermometers. The cryostat temperature is adjustable from -36°C to -42°C with scale-divisions of 0.1°C (interpolation possible) and 24-hour stability better than $\pm 0.05^{\circ}\text{C}$ measured by an SPRT in the well of a Mercury Cell inside the Cryostat.

The cryostat has several unique features providing outstanding convenience and safety. The refrigeration system has sufficient capacity to bring a cell to operating temperature in about 1 hour. At operating temperature, the cooling rate is about 1 kelvin/minute and the heating rate is about 2 kelvin/minute. This permits rapid changes to be imposed on the temperature of the cell environment to avoid excessive demands on the (low) heat-of-fusion energy of the mercury within the cell. In addition, all temperature control is accomplished through control of refrigerant flow, providing inherently fail-safe operation. Indicators provided for the cryostat are "POWER ON" and "COOLING". The cryostat provides convenient conditions for operating mercury fixed point cells both in heating and in cooling mode.

OPTIMAL CELL SPECIFICATION

Size: Overall height: 488mm (19.25in.)
Outside diameter: 40mm (1.58 in.)
Well inside diameter: 7.75mm (0.305 in.)
Active zone: bottom 200mm (8 in.) of well

CRYOSTAT SPECIFICATION

Cabinet size:

970mm (38 in.) high, 610mm (24 in.) wide. 570mm (22.4 in.) deep

Weight:

90kg (200lb.)

Power Requirements:

220 to 240 VAC, 50Hz or 60Hz (individual units), 3A (15A starting current for less than 1 second)

Well Dimensions:

All wells have a depth of 380mm (15 inches) with cooling applied to the lowest 300mm (12 inches). Internal diameters are:

Main well (for cell): 40mm (1.6 inches)

Pre-chill wells: 14mm (0.55 inch)

Tolerable Ambient Conditions:

Operating: 15 to 30°C (59 to 86°F), <70% RH

Storage: -30 to 55°C (-22 to 131°F), <85% RH

Main Well Temperature

Operating Range: -42 to -36°C

Setting facility: 0.1°C divisions. (interpolation to 0.02°C possible)

Stability $\pm 0.05^\circ\text{C}$ for a 24 hour period as measured by an SPRT in the well of a cell inside the Cryostat set at -41°C.

OPERATING INSTRUCTIONS

INITIAL SETUP

1. Inspect the outside of the cabinet for evidence of shipping damage.
2. Remove the rear panel of the cabinet.
3. Remove the stopper (DO NOT DISCARD) from one of the pre-chill wells and insert a thermometer with a range including -40°C and 25°C .
4. Ensure that the front panel "POWER" switch is off.

 **CAUTION: HAZARDOUS VOLTAGE IS PRESENT INSIDE THE CABINET WHEN POWER IS CONNECTED.**

5. Connect the power cable to an outlet that can supply a continuous current of 3A and 15A (compressor starting current) for about 0.2 second.
6. Locate the sight glass at lower centre-left (viewing from the rear of the cabinet).
7. While observing the sight glass, have someone turn the front panel "POWER" switch on. The sight glass, initially empty, should immediately fill with liquid. This verifies the operation of compressor and liquid-line cut-off valve.
8. Adjust the "SETPOINT" on the controller to -38°C and observe the thermometer reading. The temperature should drop to about -38°C .

NOTE: the pre-chill well is not tightly coupled thermally to the main well and, consequently, will take a longer time to attain a temperature approximately equal to that selected.

9. Observe the "COOLING" indicator light on the front panel. As the temperature approaches the setpoint, the indicator should begin cycling on and off and the temperature should become stable. Cycling should commence about 30-45 minutes after switch-on. This verifies operation of the controller and bypass valve.
10. Disconnect the power supply and replace the rear panel of the cabinet. This completes initial check-out.

GENERAL CRYOSTAT NOTES

The following notes apply to the cryostat, independently of cell operation (which will be covered in detail later):

1. Air-cooled refrigeration systems operate best when kept clean. Cleaning is accomplished by removing the lower front panel and cleaning the front panel screen and the condenser with an "industrial-strength" vacuum cleaner. This should be done whenever any dust build-up is observed.
2. Moisture is a problem in any low-temperature system exposed to the atmosphere. The wells should be kept closed as much as possible during use and should be kept open when at ambient temperature. Moisture may be removed from the wells by swabbing them with alcohol at room temperature, then drying them with dry compressed air, or a paper towel.
3. DO NOT stopper the cryostat wells tightly while they are returning to ambient temperature, lest the stoppers due to expansion of trapped air, unexpectedly become projectiles.
4. The control system on the cryostat will cause an initial overshoot beyond setpoint by as much as 2.5 Kelvins because of a long time-constant. This is normal and will actually be helpful in operating the cell, which has an even longer time-constant.

GENERAL CELL NOTES

1. While stainless steel cells are far more rugged than glass cells, they are still somewhat delicate, especially the thermometer guide tube.
2. Isotech Mercury Triple Point Cells are sealed with an internal pressure of less than 100 micrometers Hg, so that the effect of any argon (used in initial purging during manufacture) remaining in the cell will be negligible and the temperature at a given point in the thermometer well will be the true triple-point temperature plus the mercury-column pressure head correction. That the pressure within the cell is low may be verified by holding the cell in a horizontal position and moving it gently back and forth along its axis. As the mercury sloshes, it should produce sharp clicking sounds caused by the uninhibited collapse of bubbles in the absence of non-condensable gas.
3. The cell is supplied with a collar of closed-cell polyethylene foam around its guide tube. Do not discard this collar as it is the primary defence against moisture entry to the cryostat well when the cell is in use. See drawing 441-02-02 - Figure 3.
4. The thermometer well of the cell should be rinsed with alcohol and dried after each use to prevent build-up of moisture.

OPERATING INSTRUCTION FOR TEMPERATURE STANDARD

The procedures presented in this section have produced results within ± 1 mK of the predicted temperature on melt and freeze plateaux respectively. For measurements of the best possible precision, the procedures used by NBS for the original development of the point can be adapted for use with the Isotech cryostat. These procedures are published in the NBS special publication 260-77, "Application of some Metal SRM's as Thermometric Fixed Points" by G. Furukawa, et al. (SRM = special reference material)

MELTING PROCEDURE

1. Place the cell in the cryostat well. See drawing 441-02-02 for assembly. Ensure that the closed-cell-foam collar and the pre-chill well stoppers are in place.
2. Turn on the cryostat and adjust the "SETPOINT" to -42°C .
3. Allow at least 3.5 hours (overnight may be convenient) for the mercury in the cell to freeze completely.
4. Place a reference thermometer into the well of the cell with enough alcohol to wet the thermometer for at least 150mm (6 inches) but not more than 200mm (8 inches). This depth of heat transfer liquid will establish that thermal coupling is essentially to the cell alone.
5. Place the first thermometers to be calibrated into the pre-chill wells.
6. Observe the temperature indicated by the reference thermometer. As soon as it falls below -38.85°C , select a "SETPOINT" of -38.35°C .
7. Remove the reference thermometer from the Mercury Cell and place in one of the pre-chill wells of the apparatus. Insert an aluminium or copper rod which is at ambient temperature into the mercury cell re-entrant tube and leave in place for approximately 1 minute. After this time remove the rod and replace with the reference thermometer. This procedure melts a small film of mercury around the re-entrant tube of the cell and results in a flatter plateau.
8. When the temperature indicated by the reference thermometer becomes stable, the cell is ready for use and should produce a plateau-duration between 6 and 12 hours, depending upon loading, and set point.

FREEZING PROCEDURE

1. Place the cell in the cryostat well. Ensure that the closed-cell-foam collar and pre-chill well stoppers are in place.
2. Turn on the cryostat with "SETPOINT" selected to -41°C . (For daytime work, the cryostat may be turned on the evening before intended use with the "SETPOINT" set to -38.85°C , and then set to -41°C in the morning.)
3. Place a reference thermometer into the well of the cell with enough alcohol to wet the thermometer for at least 150mm (6 inches) but not more than 200mm (8 inches).
4. Place the first thermometers to be calibrated into the pre-chill wells.
5. Observe the temperature indicated by the reference thermometer. It should drop below the triple point temperature as the mercury in the cell under cools (0.5 to 1.5 Kelvin), and then rise rapidly to it as the mercury begins to freeze.
6. As soon as the temperature in the cell begins to rise, select a "SETPOINT" of -39.35°C .
7. When the temperature indicated by the reference thermometer becomes stable, the cell is ready for use and should produce a plateau-duration between 6 and 12 hours, depending upon loading and set point.

THEORY OF OPERATION

(Please refer to Figures 1 and 2)

REFRIGERATION SYSTEM

The refrigeration system is a sealed system with capillary-tube expansion and hot-gas-bypass control. Although the principle is simple, the implementation is complicated by the use of several specialised techniques to achieve a control stability of $\pm 0.05^{\circ}\text{C}$ as measured by an SPRT in the well of a Mercury Cell inside the Cryostat at -41°C .

Hot-gas-bypass is a standard technique for controlling refrigeration. It works by providing a controlled flow of heated-super vapour from the outlet of the compressor to the evaporator and/or the compressor suction line. This method provides rapid response and an excellent control range because the full capacity of the refrigeration system is maintained, and, at the other end of the scale, can produce strong active heating of the evaporator up to the condenser temperature.

In this system the hot gas is introduced through a special adaptor in the form of a "T" fitting which directs the end of the bypass capillary tube towards the compressor intake, about 2 inches downstream from the lower end of the (down-flow) evaporator coil. Bypass operation is controlled by a (normally-closed) solenoid valve and 1.2m (4ft) of 1.6mm (0.064 in.) I.D. capillary tubing to restrict gas flow when the valve is open. The valve cycle-time is controlled by the temperature controller electronics, and varies from about 5 to 30 seconds, depending on the load on the refrigeration system.

Expansion control is provided by 3m (10ft) of 0.7mm (0.028 in.) I.D. capillary tube. The original design incorporated control of mass flow of refrigerant through the capillary tube by operation of a small heater connected to its inlet end. The heater function was to vaporize a small portion of the refrigerant, thus reducing total mass flow and providing a lower temperature in the evaporator at the expense of cooling capacity. Heater-operation was initiated by the controller electronics whenever the by-pass valve was closed and the evaporator temperature was below -30°C .

An improvement in compressor performance under standard conditions has made the heater largely redundant. It has been retained in the system, however, but is normally isolated by a switch, located behind the rear panel, and can be operated when ambient temperature is high. Operating the heater under normal conditions can upset stability.

A heat exchanger is provided between the downstream end of the capillary tube and the exit of the evaporator to improve efficiency and reduce temperature gradients in the evaporator.

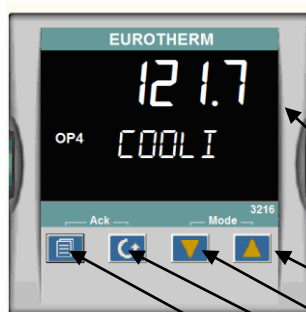
The evaporator coil is wound to be a close fit around the main well, with refrigerant entering the top of the evaporator and any excess liquid draining into the suction line. The evaporator tubing has an oval cross-section, which improves thermal coupling to the main well and provides free flow of refrigerant vapour over the downward-flowing liquid. The evaporator tube cross section is approximately 4mm (0.16 in.) x 10mm (0.4 in.), which provides sufficient space for the liquid and vapour to remain in equilibrium throughout the whole length of the evaporator, with minimum pressure difference. This makes the evaporator act as an externally-driven heat pipe, producing excellent temperature uniformity and providing for rapid return to room temperature by transferring heat from the compressor suction service valve.

The evaporator assembly, including all three well-tubes, most of the capillary tube, the hot-gas-bypass "T", the heat exchanger and the temperature sensors used by the control electronics, is a potted semi-hermetic copper shell surrounded by foam-in-place closed-cell urethane insulation all encased in an aluminium housing. This aim to exclude moisture results in the entire assembly being a single non-repairable component. Consequently, two pairs of temperature sensors are included at the time of manufacture. The controller is calibrated with respect to the pair labelled "MAIN" and will require re-calibration of the "SETPPOINT" dial if the "AUX" pair is used. (The sensors are Isotech type 46004 thermistors and may be used for monitoring well-temperature with an adaptor cable and Isotech Model 42-SC or 42-SL Telethermometer).

Colour-codes of sensor leads are as follows:

RT1 (coil)	MAIN: White
RT2 (coil)	AUX: Yellow
RT1 (well)	MAIN: Black
RT2 (well)	AUX: Orange

CONTROLLER



- Well temperature
- Up button
- Down button
- Scroll button
- Page button

The controller used in this bath is a three term PID, the set point limit is set from -36°C to -42°C . The controller operates by opening and closing the by pass valve to regulate the refrigeration system. The controller set point is adjustable in 0.1°C steps and is controlled by pressing the up/down buttons. Other features of the controller promoted to allow user adjustment are:

- Change of units from $^{\circ}\text{C}$, K and $^{\circ}\text{F}$
- Set point ramp feature, this allows the control of the change in the set point in $^{\circ}\text{C}/\text{minute}$. By default this is set to off, meaning the bath will heat/cool 100% after a change in setpoint.
- Controller PC address
- Proportional band, Integral time and Derivative time (PID). These values are factory set to give best results but should the user wish to adjust these it can be done using the up/down buttons. Note: Only operators with experience of temperature PID control should adjust these values.

To access these additional features, press the SCROLL button and use the UP/DOWN button to adjust.

CONTROLLER CALIBRATION

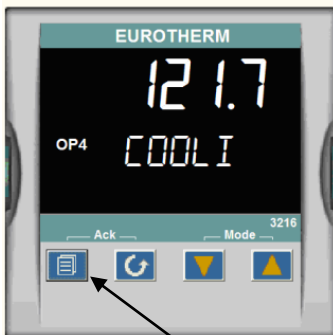
Controller calibration can be carried out using the following procedure:

Procedure to calibrate a controller by entering the measured temperature into the instrument. The instrument then computes the Point and Offset values from the entered temperature.

Caution: This procedure will change the controller calibration and is non reversible.

Recommendation: Make a note of the original calibration data before starting. This information is essential if it is required to revert to the original calibration.

Set the set point to the required calibration temperature and measure the temperature of the cell with a suitably calibrated thermometer.



Press and Hold Page Key until the controller shows



Use the UP key to select Level 2



The Display will then Change to Level 2, and then prompt for the Pass Code

Enter the Pass Code

The controller Pass Code 3

The instrument will now return to normal operating mode but there are now extra parameters available, reached by pressing the Scroll Key

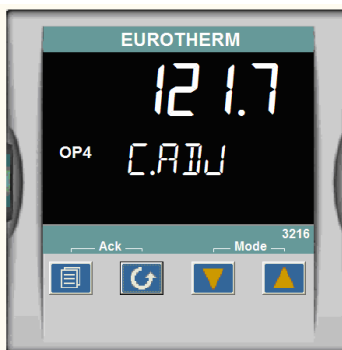


Press the *Scroll* Key until the Calibration Parameter is reached,

To Adjust the First Point press the UP key to select Point 1



Now Press the Scroll Key to gain access to the Calibration Adjust parameter



Now use the UP and DOWN keys to adjust the displayed temperature to the measured temperature of the cell.

The cell can now be set to the next calibration point and the process repeated.

There are a maximum of five points that the block can be adjusted at. Set the cell to the required calibration temperature and repeat the procedure setting the Point parameter to a value from 1 to 5 as required.

Important Notes

1: The Calibration Points must be set in ascending temperature order

E.g. -42°C , -39°C , -36°C is good

-36°C , -39°C , -42°C , is bad

2: At the end of the calibration set the Point parameter to a value of "On"



3: Setting the Point parameter to a value of Reset will clear out all the calibration data!



Take care to select this unless you really want to clear the calibration.

Recommended calibration points would be -42°C , -39°C and -36°C . This allows a calibration curve to be used at the extremes of the range and very near to the Mercury fixed point.

TROUBLESHOOTING AND REPAIR

SYSTEM TROUBLESHOOTING

Symptom	Probable Cause
No front panel lights; no sound from compressor	Main fuse (located inside rear panel at lower left) blown
No front panel lights; compressor runs	Controller fault (see "Controller Troubleshooting")
Front panel lights on; compressor does not start	Compressor start relay or capacitor fault
Front panel lights on; compressor runs; no (or limited) cooling	Refrigeration system fault (see "Refrigeration Troubleshooting")

CONTROLLER TROUBLESHOOTING

Remove controller from cabinet and troubleshoot it on the bench

 **CAUTION: HAZARDOUS VOLTAGES ARE PRESENT IN THE CONTROLLER, PARTICULARLY NEAR THE CHASSIS END PANEL**

Symptom	Probable Cause
No lights with power on	Fuse (located on chassis end panel) blown
Blows fuses	Transformer, diodes (6), filter caps (3) malfunction

REFRIGERATION TROUBLESHOOTING

Remove the rear panel and the lower front panel from the cabinet. The left side panel may also be removed to improve access.

 **CAUTION: HAZARDOUS VOLTAGES ARE PRESENT INSIDE THE CABINET, PARTICULARLY NEAR THE CONTROLLER.**

Leave the system power off for at least 15 minutes, then perform the following tests, turning the power on for only a few seconds, as necessary, for each test:

1. Observe the sight glass (centre-left from rear) while someone switches on the power. The sight glass, initially empty, should fill immediately.
2. Measure the voltage across the cut-off valve solenoid (terminals 1 and 2 of the terminal strip at lower left from back) as the power is switched on. The indications should be 12 VDC for about ¼ second, and then drop to about 3V. Note that terminals 2, 4 and 6 of the terminal strip are connected to the + 12V supply.
3. Measure the voltage across the bypass valve solenoid (terminals 3 and 4). The reading should be less than 0.5V.

Symptom	Probable Cause
Sight glass fills; insufficient cooling	Clogged capillary; air in system
No voltage on cutoff solenoid	Controller fault (see "Controller Troubleshooting")
Sight-glass dry; cutoff solenoid voltage stays at 12V	Valve solenoid open circuit
Bypass valve voltage = 12V	Controller fault (see "Controller Troubleshooting")

TESTING WITH GAUGES

The following actions should be carried out by someone familiar with refrigeration servicing.

Connect pressure gauges to the service valves on the condensing unit and observe the pressures with the power off and then with the power on, if required.

Note that the cut-off valve prevents the system from equalizing in pressure (this valve is necessary to prevent compressor flooding by reason of the down-flow evaporator.)

Symptom	Probable Cause
High side low with power off	Leak in high side
High side high with power off	Leak in low side, allowing air-ingress
Low side equal to high side	Cutoff bypass valve leaking
Low side goes lower than 15" Hg vacuum with power on	Plugged capillary
Low side does not reach 5" Hg vacuum with power on	Bypass valve leaking

Figure 1: Schematic Diagram of Cryostat

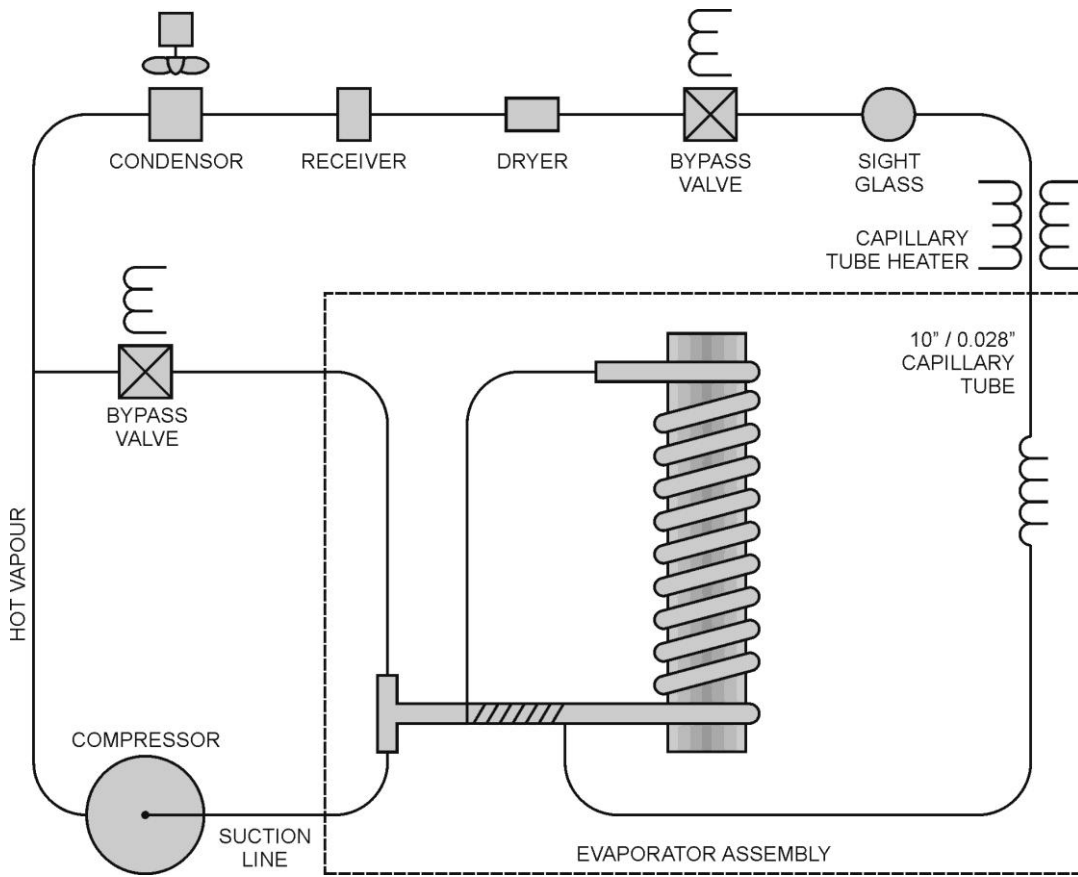
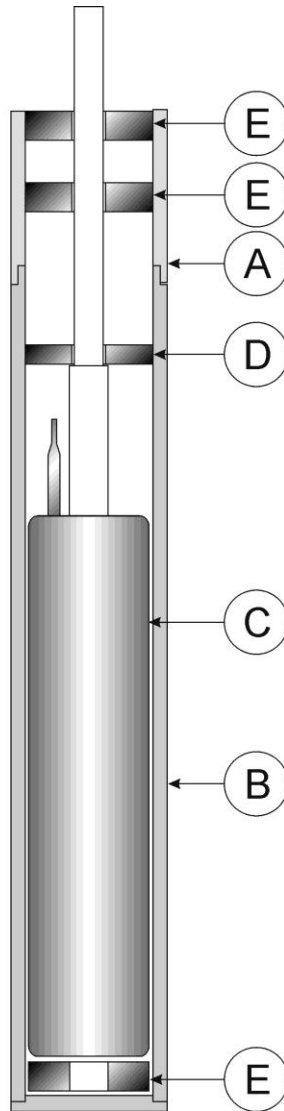


Figure 2: Standard Sized Mercury Well General Arrangement



A	Well Assembly Socket	440-02-06
B	Evaporator Well Assy.	440-02-04
C	Mercury Cell	441-00-00
D	Copper Washer	440-02-34
E	Ethafoam Insulation	440-02-35