

**FLUKE**®

**Hart Scientific**®

# ***9115 Furnace***

*User's Guide*

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To obtain warranty service, contact your nearest Hart authorized service center or send the product, with a description of the difficulty, postage, and insurance prepaid (FOB Destination), to the nearest Hart authorized service center. Hart assumes no risk for damage in transit. Following warranty repair, the product will be returned to Buyer, transportation prepaid (FOB Destination). If Hart determines that the failure was caused by misuse, alteration, accident or abnormal condition or operation or handling, Hart will provide an estimate or repair costs and obtain authorization before commencing the work. Following repair, the product will be returned to the Buyer transportation prepaid and the Buyer will be billed for the repair and return transportation charges (FOB Shipping Point).

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# Table of Contents

<b>1</b>	<b>Before You Start . . . . .</b>	<b>1</b>
1.1	Introduction . . . . .	1
1.2	Symbols Used . . . . .	1
1.3	Safety Information . . . . .	2
1.3.1	WARNINGS . . . . .	2
1.3.2	CAUTIONS . . . . .	4
1.4	Authorized Service Centers. . . . .	5
<b>2</b>	<b>Specifications and Environmental Conditions . . . . .</b>	<b>7</b>
2.1	Specifications . . . . .	7
2.2	Environmental Conditions . . . . .	7
<b>3</b>	<b>Installation . . . . .</b>	<b>9</b>
3.1	Unpacking . . . . .	9
3.2	Location . . . . .	9
3.3	Power . . . . .	9
3.4	Plumbing . . . . .	11
3.5	Heat Pipe Installation . . . . .	11
<b>4</b>	<b>Parts and Controls . . . . .</b>	<b>15</b>
4.1	Control Panel . . . . .	15
4.1.1	Controller . . . . .	15
4.1.2	System Fuses . . . . .	16
4.2	Furnace Core . . . . .	16
4.3	Access Well . . . . .	18
4.4	Back Panel. . . . .	18
<b>5</b>	<b>General . . . . .</b>	<b>19</b>
5.1	Sodium Heat Pipe Information . . . . .	19
5.1.1	Minimum Temperature and Heat-Up Rate . . . . .	19
5.1.2	Safety Precautions . . . . .	19
5.1.3	Life Expectancy . . . . .	19
<b>6</b>	<b>Controller Operation. . . . .</b>	<b>21</b>
6.1	Well Temperature . . . . .	21
6.2	Temperature Set-point. . . . .	21
6.2.1	Programmable Set-points . . . . .	21

6.2.2	Set-point Value . . . . .	23
6.2.3	Temperature Scale Units . . . . .	23
6.3	Scan . . . . .	24
6.3.1	Scan Control . . . . .	24
6.3.2	Scan Rate . . . . .	24
6.4	Ramp and Soak Program . . . . .	24
6.4.1	Program Points . . . . .	25
6.4.2	Number of Program Points . . . . .	25
6.4.3	Editing Program Set-Points . . . . .	25
6.4.4	Program Function Mode . . . . .	27
6.4.5	Program Control . . . . .	27
6.5	Secondary Menu. . . . .	28
6.6	Heater Power . . . . .	28
6.7	Proportional Band . . . . .	28
6.8	Controller Configuration . . . . .	30
6.9	Operating Parameters . . . . .	30
6.9.1	High Limit. . . . .	30
6.9.2	Soft Cut-out . . . . .	30
6.10	Serial Interface Parameters . . . . .	31
6.10.1	Baud Rate . . . . .	31
6.10.2	Sample Period. . . . .	31
6.10.3	Duplex Mode . . . . .	32
6.10.4	Linefeed . . . . .	32
6.11	Calibration Parameters . . . . .	33
6.11.1	Hard Cutout . . . . .	33
6.11.2	CT1, CT2, CT3 . . . . .	33
6.11.3	CE1, CE2, CE3 . . . . .	33
<b>7</b>	<b>Digital Communication Interface . . . . .</b>	<b>35</b>
7.1	Serial Communications . . . . .	35
7.1.1	Wiring . . . . .	35
7.1.2	Setup . . . . .	35
7.1.2.1	Baud Rate . . . . .	36
7.1.2.2	Sample Period. . . . .	36
7.1.2.3	Duplex Mode . . . . .	36
7.1.2.4	Linefeed. . . . .	36
7.1.3	Serial Operation. . . . .	36
7.2	Interface Commands . . . . .	37
<b>8</b>	<b>Freeze Point Realization . . . . .</b>	<b>41</b>
8.1	General . . . . .	41
8.2	Installing the Metal Freeze Point Cell. . . . .	41
8.3	How to realize the freezing point of silver . . . . .	43
8.4	Safety Precautions. . . . .	44

<b>9</b>	<b>Calibration Procedure . . . . .</b>	<b>45</b>
9.1	Calibration Points . . . . .	45
9.2	Calibration Procedure . . . . .	45
<b>10</b>	<b>Maintenance . . . . .</b>	<b>47</b>
<b>11</b>	<b>Troubleshooting. . . . .</b>	<b>51</b>
11.1	Troubleshooting . . . . .	51

# Figures and Tables

Table 1	International Electrical Symbols . . . . .	1
Figure 1.	Furnace Core Diagram . . . . .	10
Figure 2	Thermocouple wire routing and heat pipe ground wire. . . . .	12
Figure 3.	Front Control Panel (cover door remove) . . . . .	15
Figure 4.	Back Panel . . . . .	17
Figure 5.	Controller Operation Flowchart . . . . .	22
Figure 6.	Serial Cable Wiring . . . . .	35
Table 2.	Digital Communications Commands . . . . .	38
Table 2.	Digital Communications Commands continued . . . . .	39
Figure 7.	Metal Freeze Point Cell installed in canister. . . . .	42
Figure 8.	Testing Uniformity . . . . .	48

# 1 Before You Start

## 1.1 Introduction

The Hart Scientific 9115 Furnace has a temperature range of 550° C to 1000° C and is designed for use in achieving aluminum or silver freezing point measurements. The 9115 may also be used to make comparison measurements using a specially designed quartz/graphite equilibration block.

The furnace utilizes a sodium heat pipe to maintain a uniform temperature over the length of the metal freeze point cell. The temperature controller is programmable, a feature that may be conveniently used to simplify the melting, freeze initiation, and plateau control. The temperature control and uniformity of the furnace allows the user to achieve plateaus ranging many hours in length.



















**NOTE:** Many of the illustrations and examples used in this manual assume the use of the silver point cell. Either of the cells indicated may be used with the appropriate set-points.

## 1.2 Symbols Used

Table 1 lists the International Electrical Symbols. Some or all of these symbols may be used on the instrument or in this manual.

**Table 1** International Electrical Symbols

Symbol	Description
	AC (Alternating Current)
	AC-DC
	Battery
	CE Complies with European Union Directives
	DC
	Double Insulated
	Electric Shock

Symbol	Description
	Fuse
	PE Ground
	Hot Surface (Burn Hazard)
	Read the User's Manual (Important Information)
	Off
	On
	Canadian Standards Association
<b>CAT II</b>	OVERVOLTAGE (Installation) CATEGORY II, Pollution Degree 2 per IEC1010-1 refers to the level of Impulse Withstand Voltage protection provided. Equipment of OVERVOLTAGE CATEGORY II is energy-consuming equipment to be supplied from the fixed installation. Examples include household, office, and laboratory appliances.
	C-TIC Australian EMC Mark
	The European Waste Electrical and Electronic Equipment (WEEE) Directive (2002/96/EC) mark.

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## 1.3 Safety Information

Use this instrument only as specified in this manual. Otherwise, the protection provided by the instrument may be impaired.

The following definitions apply to the terms “Warning” and “Caution”.

- “WARNING” identifies conditions and actions that may pose hazards to the user.
- “CAUTION” identifies conditions and actions that may damage the instrument being used.

### 1.3.1 WARNINGS

To avoid personal injury, follow these guidelines.

- **DO NOT** operate this unit without a properly grounded, properly polarized power cord.



- **DO NOT** connect this unit to a non-grounded, non-polarized outlet.
- **DO USE** a ground fault interrupt device.
- **HIGH VOLTAGE** is used in the operation of this equipment. **SEVERE INJURY OR DEATH** may result if personnel fail to observe safety precautions. Before working inside the equipment, turn power off and disconnect power cord.
- If this equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.
- Before initial use, or after transport, or after storage in humid or semi-humid environments, or anytime the instrument has not been energized for more than 10 days, the instrument needs to be energized for a "dry-out" period of 2 hours before it can be assumed to meet all of the safety requirements of the IEC 1010-1. If the product is wet or has been in a wet environment, take necessary measures to remove moisture prior to applying power such as storage in a low humidity temperature chamber operating at 50°C for 4 hours or more.
- This unit contains ceramic fiber or other refractories, which can result in the following:
  - May be irritating to skin, eyes, and respiratory tract.
  - May be harmful if inhaled.
  - May contain or form cristobalite (crystalline silica) with use at high temperatures (above 1600°F) which can cause severe respiratory disease. Possible cancer hazard based on tests with laboratory animals. Animal studies to date are inconclusive. No human exposure studies with this product have been reported.
  - Service personnel coming into contact with these materials should take proper precautions when handling them. Before maintaining this equipment, read the applicable MSDS (Material Safety Data Sheets).
- **HIGH TEMPERATURES PRESENT** in this equipment **FIRES AND SEVERE BURNS** may result if personnel fail to observe safety precautions.
- **DO NOT** use this unit for any application other than calibration work.
- **DO NOT** use this unit in environments other than those listed in the user's manual.
- Continuous use of this equipment at high temperatures for extended periods of time requires caution.
- Completely **unattended high temperature operation is not recommended** for safety reasons.
- In the unlikely event that the heat pipe should leak: **DO NOT** attempt to put out the fire with water or chemical fire extinguishers. **SMOTHER THE FIRE WITH DRY SODA ASH.** See the Material Safety Data Sheet (MSDS).
- Follow all safety guidelines listed in the user's manual.

- The furnace generates extreme temperatures. Precautions must be taken to prevent personal injury or damage to objects. Probes may be extremely hot when removed from the furnace. Cautiously handle probes to prevent personal injury. Carefully place probes on a heat resistant surface or rack until they are at room temperature. SPRT's should be placed in an annealing furnace if removed at temperatures greater than 500°C.
- Use only grounded AC mains supply of the appropriate voltage to power the instrument. The furnace requires 12 amps at 230V ( $\pm 10\%$ ), 50/60 Hz.
- The 9115 Furnace utilizes high voltages and currents to create high temperatures. Caution should always be maintained during installation and use of this instrument to prevent electrical shock and burns. Fire can be a hazard for any device that produces high temperatures. Proper care and installation must be maintained. Responsible use of this instrument will result in safe operation.
- Calibration Equipment should only be used by Trained Personnel.

### 1.3.2 CAUTIONS

To avoid possible damage to the instrument, follow these guidelines.

- Components and heater lifetimes can be shortened by continuous high temperature operation.
- Operate the instrument in room temperatures between 5-50°C (41-122°F). Allow sufficient air circulation by leaving at least 6 inches of space between the furnace and nearby objects. Nothing should be placed over the top of the furnace. The furnace should not be placed under cabinets or tables. Extreme temperatures can be generated out the top of the well. If the furnace is equipped with cooling coils, use cold water circulation when the furnace is used above 600°C. (For specifics see Section 4.5 Plumbing.)
- The furnace is a precise instrument. Although it has been designed for optimum durability and trouble free operation, it must be handled with care. The instrument should not be operated in wet, oily, dusty or dirty environments. Keep the well of the instrument free of any foreign matter. Do not operate near flammable materials.
- Do not use fluids to clean out the well.
- The instrument is equipped with operator accessible system fuses. If a fuse blows, it may be due to a power surge or failure of a component. Replace the fuse once. If the fuse blows a second time, it is likely caused by failure of a component. If this occurs, contact an Authorized Service Center. Always replace the fuse with one of the same rating, voltage, and type. Never replace the fuse with one of a higher current rating.
- If a main supply power fluctuation occurs, immediately turn off the furnace. Power bumps from brown-outs and black-outs can damage the instrument. Wait until the power has stabilized before re-energizing the furnace.

- The unit is not equipped with wheels. It is considered to be permanently set once it has been installed. If the unit must be moved for some reason, be sure that the fixed point cell has been removed before moving the furnace. Any movement of the furnace with the cell inside can damage the cell. The unit is not designed to be lifted or carried. If it must be picked up, it is advisable that two people pick the unit up by placing their hands under the unit and carefully lifting at the same time. Never move the furnace if it is hot.
- Air circulated through the gap surrounding the furnace core keeps the chassis cool. **DO NOT SHUT OFF THE FURNACE WHILE AT HIGH TEMPERATURES.** The fan will turn off allowing the chassis to become hot. Alternatively, if used, the cooling water should remain on until the furnace is cool.
- Once the unit has been taken to high temperatures (over 800°C), it takes days for the unit to cool completely.

## 1.4 Authorized Service Centers

Please contact one of the following authorized Service Centers to coordinate service on your Hart product:

### **Fluke Corporation, Hart Scientific Division**

799 E. Utah Valley Drive  
American Fork, UT 84003-9775  
USA

Phone: +1.801.763.1600  
Telefax: +1.801.763.1010  
E-mail: support@hartscientific.com

### **Fluke Nederland B.V.**

Customer Support Services  
Science Park Eindhoven 5108  
5692 EC Son  
NETHERLANDS

Phone: +31-402-675300  
Telefax: +31-402-675321  
E-mail: ServiceDesk@fluke.nl

### **Fluke Int'l Corporation**

Service Center - Instrimpex

Room 2301 Sciteck Tower  
22 Jianguomenwai Dajie  
Chao Yang District  
Beijing 100004, PRC  
CHINA

Phone: +86-10-6-512-3436  
Telefax: +86-10-6-512-3437  
E-mail: xingye.han@fluke.com.cn

**Fluke South East Asia Pte Ltd.**

Fluke ASEAN Regional Office  
Service Center  
60 Alexandra Terrace #03-16  
The Comtech (Lobby D)  
118502  
SINGAPORE

Phone: +65 6799-5588  
Telefax: +65 6799-5588  
E-mail: antng@singa.fluke.com

When contacting these Service Centers for support, please have the following information available:

- Model Number
- Serial Number
- Voltage
- Complete description of the problem

## 2 Specifications and Environmental Conditions

### 2.1 Specifications

Temperature Range	550°C to 1000°C (1022°F to 1832°F)
Accuracy	±3.0°C
Stability	±0.3°C
Uniformity	+0.1°C (+ only is intentional)
Control Probe	Type R thermocouple
Resolution	0.1°C/°F below 1000°C/°F 1°C/°F above 1000°C/°F
Readout	Switchable °C or °F
Controller	Digital controller with data retention
Fault Protection	High temperature cutout (Type R cutout thermocouple) Sensor burnout and short protection
Cutout Accuracy	±10°C
Power	230 VAC (±10%), 50/60 Hz, 12 A, 2500 W
Heater	2500 W
System Fuses	15 A 250 V fast acting
Exterior Dimension	838 mm H x 610 mm W x 406 mm D (33 x 24 x 16 in)
Weight	82 kg (180 lb.)
Safety	OVERVOLTAGE (Installation) CATEGORY II, Pollution Degree 2 per IEC-61010-1

### 2.2 Environmental Conditions

Although the instrument has been designed for optimum durability and trouble-free operation, it must be handled with care. The instrument should not be operated in an excessively dusty or dirty environment. Maintenance and cleaning recommendations can be found in the Maintenance Section of this manual.

The instrument operates safely under the following conditions:

- temperature range: 5 - 50°C (41 - 122°F)
- ambient relative humidity: maximum 80% for temperature <31°C, decreasing linearly to 50% at 40°C
- pressure: 75kPa - 106kPa
- mains voltage within ± 10% of nominal

- vibrations in the calibration environment should be minimized
- altitude does not effect the performance or safety of the unit

If the unit is operating at temperatures above 600°C, cooling coils are accessible on the rear of the chassis to prevent the furnace heat from loading down the room air conditioning system. (See [Section 3.4 Plumbing](#))

## **3 Installation**

### **3.1 Unpacking**

Verify that the following components are present:

- Furnace
- Cutout and Control Thermocouples
- Radiation Guard (Quartz tube packed separately)
- Fixed Point Basket
- Fixed Point Basket Lid
- Fixed Point Basket removal tool
- Sodium Heat Pipe
- Extra Insulation:
- Paper for the fixed point cell
- Small circles for fixed point basket
- Miscellaneous for packing around the fixed point cell

Unpacking should be done carefully. Several parts are packed disassembled for safe shipment. Small parts may be packed in a separate box inside the crate. Check carefully for all parts. If there is any damage due to shipment, notify your carrier immediately.

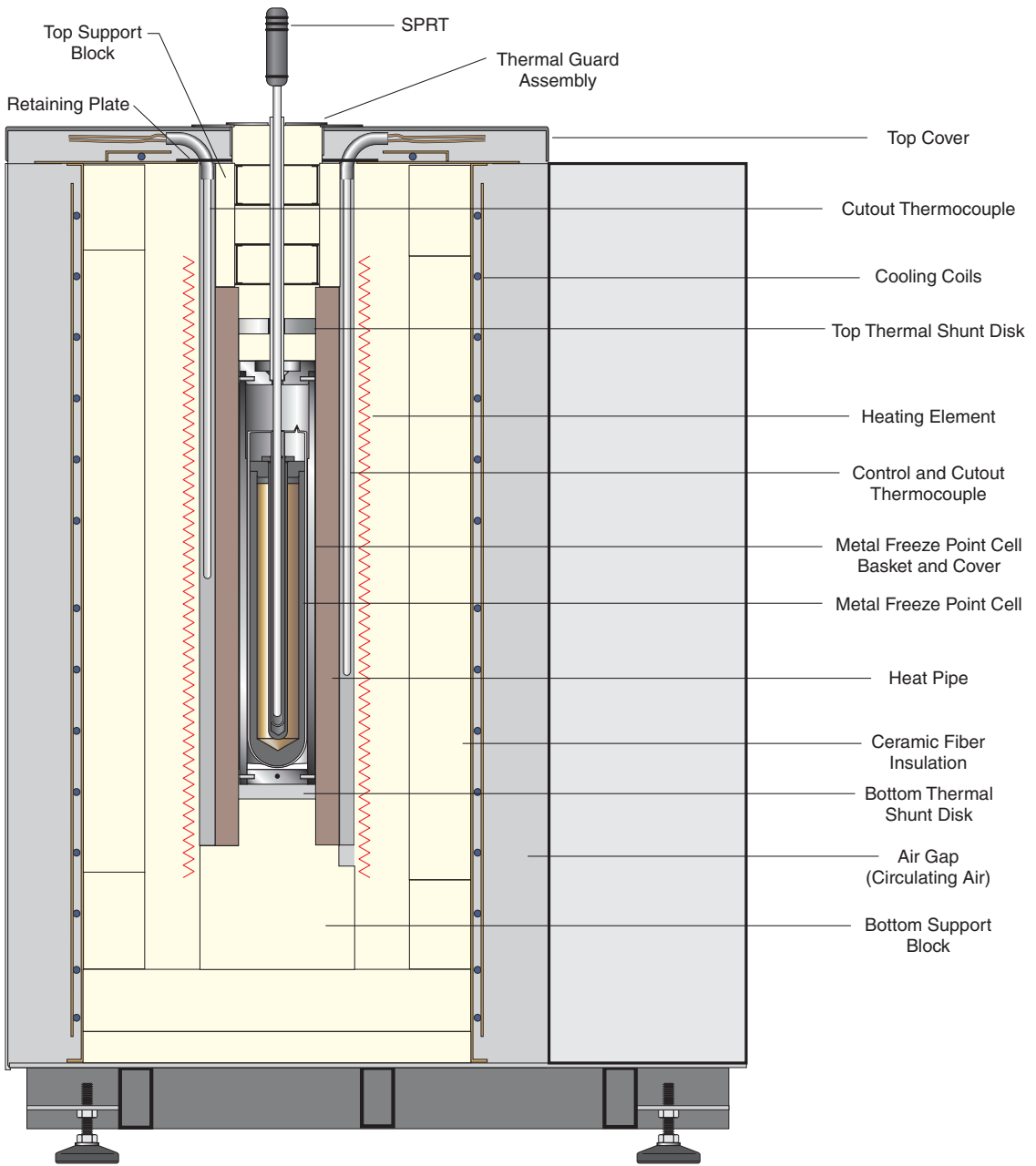
### **3.2 Location**

A furnace of this type is typically installed in a calibration laboratory where temperature conditions are generally well controlled. Best results will be obtained from this type of environment. Avoid the presence of flammable materials near the furnace. Allow 6 or more inches of air space around the furnace. Adjust the levelers on the bottom of the furnace to level the furnace and to keep it from rocking.

### **3.3 Power**

The 9115 furnace power requirements are listed in Section 2.1 on page 7. The furnace is supplied with a 14-gauge, 2-conductor plus ground cable and connector. Since building electrical installations may vary, the connector and cable may be removed at the furnace back panel and another used so long as it is rated for the specified current and voltage. (See Figure 4, Back Panel on page 17.)

Be sure that the furnace chassis is always solidly grounded. A shock hazard may exist if it is not. All switches are double pole for safety in such hot-hot-neutral installations and both lines are fused.



**Figure 1.** Furnace Core Diagram



## 3.4 Plumbing

The cooling coils are accessible from the back panel of the 9115 chassis (See Figure 4, Back Panel on page 17). The cooling tubes are 6.35 mm (0.25 in) copper. Water cooling prevents much of the furnace heat from loading down air conditioning systems. Provide cold tap water with a valve convenient for operation near the rear of the furnace. A flow rate of about 0.4 GPM of tap water is required. Pressure should not exceed 60 PSIG. Drain the warm exit water into an appropriate sump.

## 3.5 Heat Pipe Installation

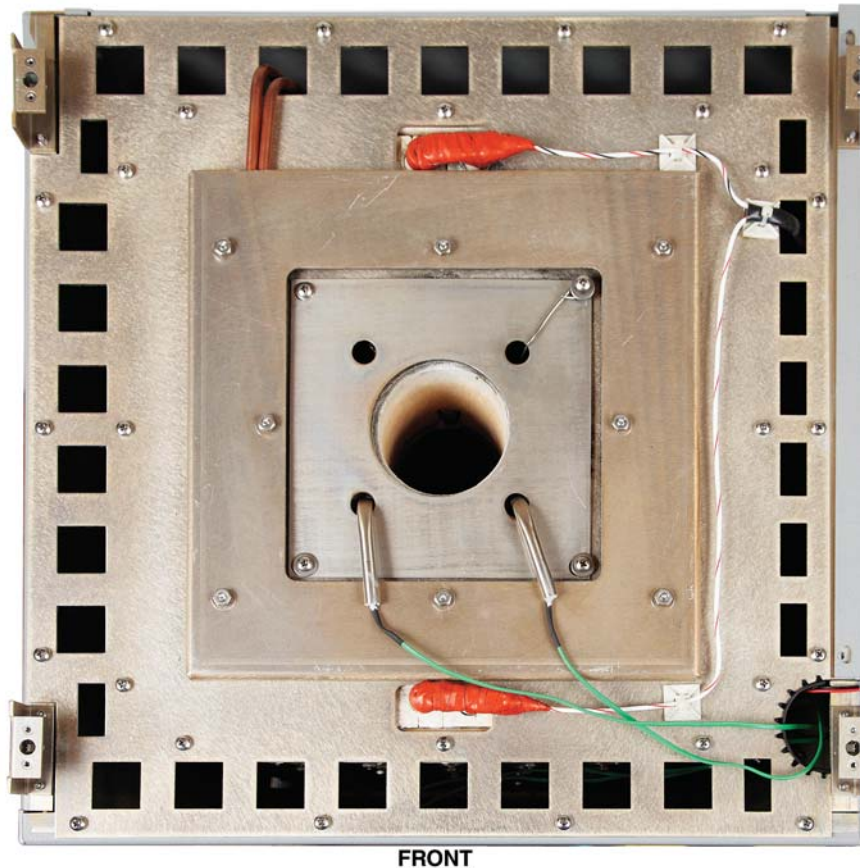


**NOTE:** *The first heat of the furnace should be done without the heat pipe to verify operation and to become familiar with the furnace. Remove any packing material before operating.*

The heat pipe is shipped separately in order to prevent damage to the heater and delicate fiber ceramic insulation. Re-installation of the temperature Control and Cutout thermocouples is also required. Follow the instructions carefully. Many of the materials are fragile. Refer to Figure 1 on page 10. Should the furnace be relocated, the heat pipe must be removed and reinstalled at the new location. See Figure 1 which shows the internal components of the furnace core.

1. Remove the top cover of the furnace. Remove the metal retaining plate by detaching the four screws holding it in. Remove the top support block and any packing material (used for shipping) from the central furnace core. Leave the bottom support block in place at the bottom of the core.
2. Locate the heat pipe and the heat pipe installation tool. Use cotton gloves to handle the heat pipe. Finger prints will cause corrosion of the Inconel at high temperatures. The installation tool has a handle with heavy gauge wire hooks which fit into the tabs on the top of the furnace. Remove any packing materials from the heat pipe. *(Any finger oils must be removed with alcohol before installation.)* The heat pipe has a protrusion on the top between the installation tabs which served as a filling port. This port must be rotated appropriately to fit the matching notch on the top support block. Note the orientation of the notch on the top support block and be sure to rotate the heat pipe accordingly. The larger notches in the top support block are toward the rear of the furnace. There is a grounding wire attached to the top flange of the heat pipe. Be careful that it does not damage the ceramic heaters as the heat pipe is lowered into the furnace.
3. Attach the installation tool to the heat pipe and lower it very carefully into the furnace. Maintain the heat pipe in the center of the well so as to allow the bottom support block to slide into the cavity at the bottom of the heat pipe. The fiber-ceramic material is very fragile. The heater and the support can easily be damaged if care is not taken during heat pipe installation.

4. Once the heat pipe is in place, position the top support block over the heat pipe. Be careful to position the notch onto the heat pipe, the flanges of the heat pipe into the slits in the support block and the grounding wire through the right rear access notch (when facing the furnace) in the support block. If necessary, use a knife to cut pieces of the ceramic blanket material provided to center and firmly locate the top support block in place.
5. Re-attach the metal plate using the four screws. The ground wire from the heat pipe must be routed through the right rear access hole in the plate and attached to the closest screw using two star washers and a screw. Make sure that the ground wire is securely attached to the furnace chassis. Insure that the wire routing for the Control and Cutout probe is as shown in Figure 2.



**Figure 2** Thermocouple wire routing and heat pipe ground wire.

6. Reinstall the thermocouples. They must be inserted far enough to clear the top cover. They should also be centered in the air space between the element and the heat pipe. Small pieces of the fiber ceramic material can be used to fix their positions. The wires from the thermocouples must be properly routed and connected to the controller. Remove the cover over the electronics panel. Route the thermocouple wires through the hole in the top of the furnace (at the front right of the furnace when facing the furnace) and down to the access hole into the electronics compartment (at the center left when facing the electronics panel). Connect the thermocouple wires to the bottom of the controller. The labels on the thermocouples and the controller must be used to correctly complete the wiring. The wires should be firmly attached using the screw-down terminals on the controller printed circuit boards. Make sure that the leads from the thermocouples are directed away from the central well of the furnace, and will not be pinched or interfere with other parts.
7. Reinstall the top cover. Locate the cover over the ball catches and press down firmly.
8. Become familiar with the operation of the furnace before installing the metal freeze point cell. Use the program installed in the furnace if desired.

## 4 Parts and Controls

The Model 9115 consists of a control panel, furnace core, and a back panel. Each part and control is described below.

### 4.1 Control Panel

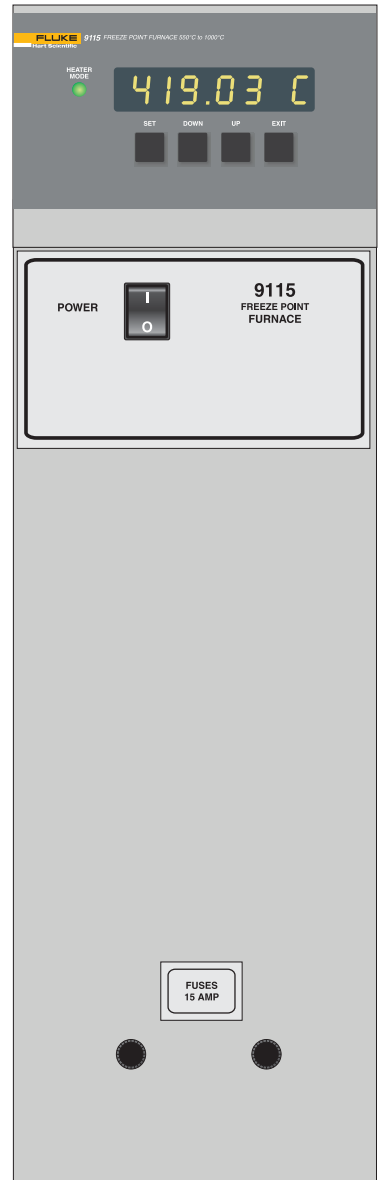
The controls to the furnace are located on panels to the right of the instrument. The upper portion of the panel is sloped and contains the controller which is regularly used during operation of the furnace. A green LED on the main display indicates the unit is energized. Beneath the controller is the power switch. The system fuses are behind a hinged door under the power switch.

#### 4.1.1 Controller

The controller has overall control of the furnace. This sloped panel is located on the upper right portion of the furnace (see Figure 3). The controller itself is a hybrid analog/digital device utilizing the high stability of analog circuitry with the flexibility of a micro-processor interface and digital controls.

The following controls and indicators are present on the primary controller panel: (1) the digital LED display, (2) the control buttons, and (3) the power indicator light.

- (1) The digital display shows the set and actual temperatures as well as various other functions, settings and constants. The temperature can be set in scale units of either °C or °F.
- (2) The control buttons (SET, DOWN, UP, and EXIT) are used to set the furnace temperature set-point, access and set other operating and calibration parameters.



**Figure 3.** Front Control Panel (cover door remove)

A brief description of the functions of the buttons follows:

**SET** - Used to display the next parameter in a menu and to store parameters to the displayed value.

**DOWN** - Used to decrement the displayed value of parameters.

**UP** - Used to increment the displayed value.

**EXIT** - Used to exit from a menu. When “EXIT” is pressed any changes made to the displayed value are ignored.

(3) The LED indicator lets the user visually see that the unit is energized.

### 4.1.2 System Fuses

For easy access by the user, the system fuses of appropriate amperage are located behind the door of the lower panel.

## 4.2 Furnace Core

The furnace core consists of the heater, insulating materials, heat pipe, heat pipe support blocks, and the housing with water cooling. Refer to Figure 1 on page 10.

The heater is embedded in a fiber ceramic insulating block. A hollow section through the center contains the heat pipe.

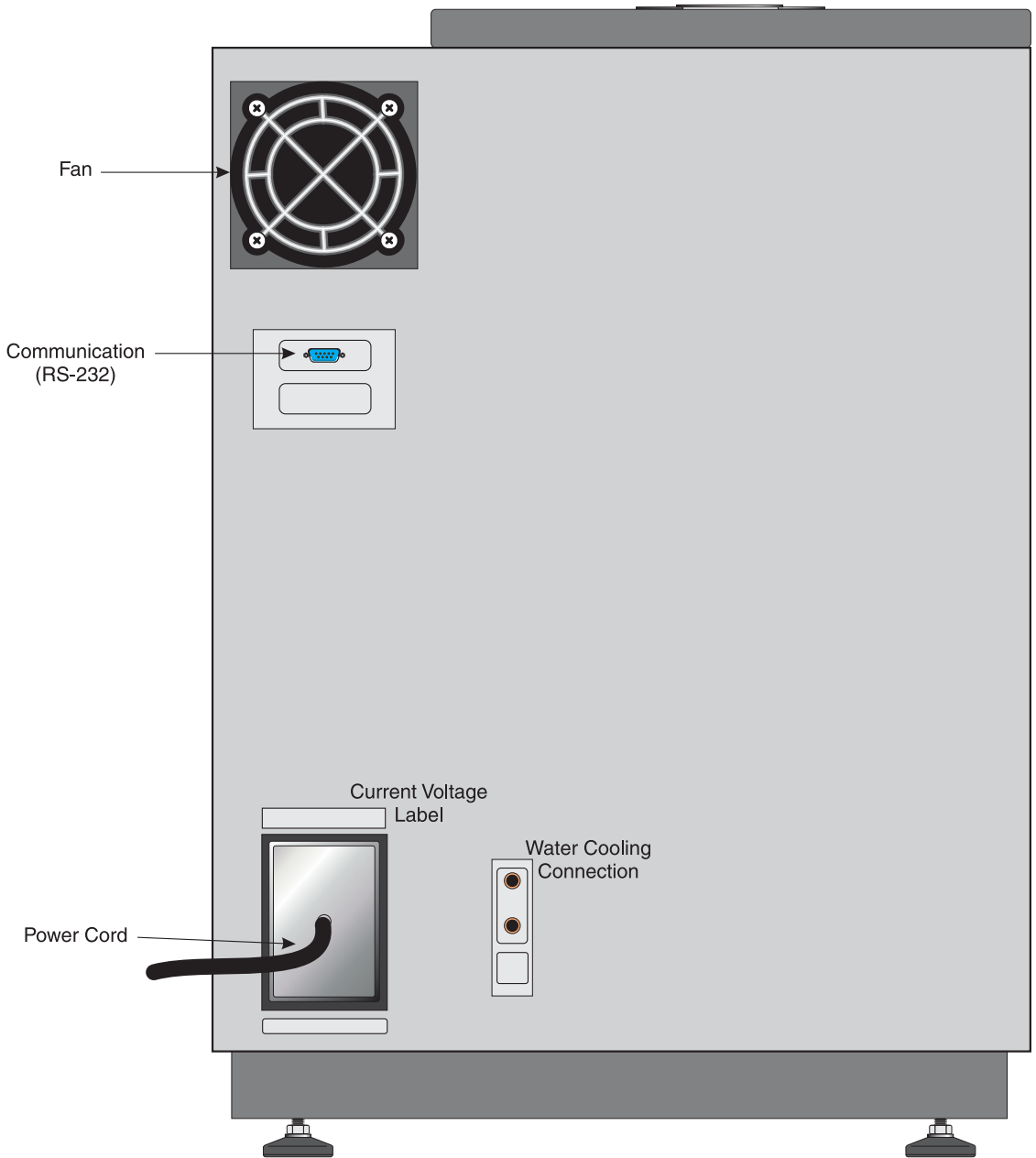
The heat pipe is a double wall Inconel cylinder containing sodium. The minimum working temperature of the Sodium heat pipe is about 500°C. The heat pipe must be heated slowly (about 1-2 hours) to this temperature. The temperature may then be raised more quickly to the desired set-point. When the working temperature is achieved, the sodium circulates throughout the tube providing a uniform temperature. The heat pipe has a lifetime of many years. Do not use the heat pipe unnecessarily at high temperatures which reduces the lifetime. Refer to the Section 5 for more information.



**CAUTION:** *In the unlikely event that the heat pipe should leak, do not attempt to put out the fire with water or chemical fire extinguishers. Smother the fire with dry soda ash in accordance with the MSDS sheets at the end of this manual.*

The heat pipe is centered in the heating element and supported by means of fiber ceramic blocks. One block fits into the bottom of the heat pipe and a second fits over the top centering the heat pipe in the heater assembly. The top block also supports the thermocouples. The heat pipe is shipped separately since its weight would damage the heater and supports if it were in place during shipment.

The entire heater and heat pipe assembly are contained and supported by a sheet metal housing. Copper cooling coils are attached to the outside of this housing. These cooling coils allow some of the heat lost to be removed from



**Figure 4.** Back Panel

the lab area reducing the lab heat load. They are accessible on the rear of the furnace chassis (see Figure 4).

Air is circulated through the gap surrounding the furnace core. This keeps the chassis cool. Do not shut off the furnace while at high temperatures or the fan will turn off allowing the chassis to become hot. Alternatively, if used, the water cooling could remain on until the furnace is cool.

### **4.3 Access Well**

The furnace access well is visible on top of the furnace.

The furnace access well is where the freeze point cell is inserted and removed from the furnace. After a freeze point cell is inserted into the furnace, a thermal shunt disk and thermal guard assembly are installed over it. This provides a block to the heat loss from the well and provides more temperature uniformity for the cell.

### **4.4 Back Panel**

The back panel consists of an exhaust fan, a serial communications connector, a power cord, and cooling water ports. See Figure 4 on page 17.

1. The exhaust fan allows air circulation around the electrical components. Be sure to keep this fan free of foreign objects that could hinder air flow.
2. The serial communication connector is a DB-9 connector for interfacing the furnace to a computer or terminal with serial RS-232 communications. (See Section 7 starting on page 35 for details.)
3. The power cord is a non-removable cord.
4. The cooling water ports are provided for connecting to cooling water to reduce the heat load. See Section 4.5 Plumbing for details.

## 5 General

### 5.1 Sodium Heat Pipe Information

#### 5.1.1 Minimum Temperature and Heat-Up Rate

The heat pipe should not be inserted into a hot furnace. The minimum operating temperature is 500°C. The heat pipe should be installed in a cold furnace and heated to the minimum operating temperature over a minimum 1 hour period. Above the minimum operating temperature the heat-up rate is not important.

#### 5.1.2 Safety Precautions

The heat pipe was designed for long-term maintenance free operation. It was performance tested at the factory and in the furnace.

In the event of an accident which results in a rupture of the heat pipe, the small quantity of sodium may burn. **DO NOT** use water or standard fire extinguishers on sodium fires. (Refer to MSDS) Standard commercially available materials for extinguishing sodium fires are dry soda ash ( $\text{Na}_2\text{CO}_3$ ) or powdered graphite. *A container of one of these products should be kept in the laboratory near the furnace in case of an accident.*

Mechanical damage to the fill tube may cause in-leakage of air at low temperatures. This will evidence itself by cold regions in the heat pipe when at operating temperature. Operation should be discontinued and Hart Scientific Customer Service should be consulted.

#### 5.1.3 Life Expectancy

The heat pipe is designed for a minimum of 2 years of operation at 1040°C. The heat pipe will function for a short-time (1000 hours) at 1100°C. *A record should be kept of time of operation at or near the maximum temperature.*



## 6 Controller Operation

This section discusses in detail how to operate the furnace temperature controller using the front control panel. By using the front panel key-switches and LED display the user may monitor the well temperature, adjust the set-point temperature in degrees C or F, monitor the heater output power, adjust the controller proportional band, and program the probe calibration parameters, operating parameters, serial interface configuration, and controller calibration parameters. Operation of the functions and parameters are shown in the flowchart in [Figure 5](#) on page 22. This chart may be copied for reference.

In the following discussion a button with the word SET, UP, DOWN, or EXIT inside indicates the panel button while the dotted box indicates the display reading. Explanation of the button or display reading are to the right of each button or display value.

### 6.1 Well Temperature

The digital LED display on the front panel allows direct viewing of the actual well temperature. This temperature value is what is normally shown on the display. The units, C or F, of the temperature value are displayed at the right. For example,

962.3 C

*Well temperature in degrees Celsius*

The temperature displayed function may be accessed from any other function by pressing the “EXIT” button.

### 6.2 Temperature Set-point

The temperature set-point can be set to any value within the range and resolution as given in the specifications. Be careful not to exceed the safe upper temperature limit of any device inserted into the well.

Setting the temperature involves two steps: (1) select the set-point memory and (2) adjust the set-point value.

#### 6.2.1 Programmable Set-points

The controller stores 8 set-point temperatures in memory. The set-points can be quickly recalled to conveniently set the calibrator to a previously programmed temperature set-point.

To set the temperature one must first select the set-point memory. This function is accessed from the temperature display function by pressing “SET”. The number of the set-point memory currently being used is shown at the left on the display followed by the current set-point value.

100.0 C

*Well temperature in degrees Celsius*

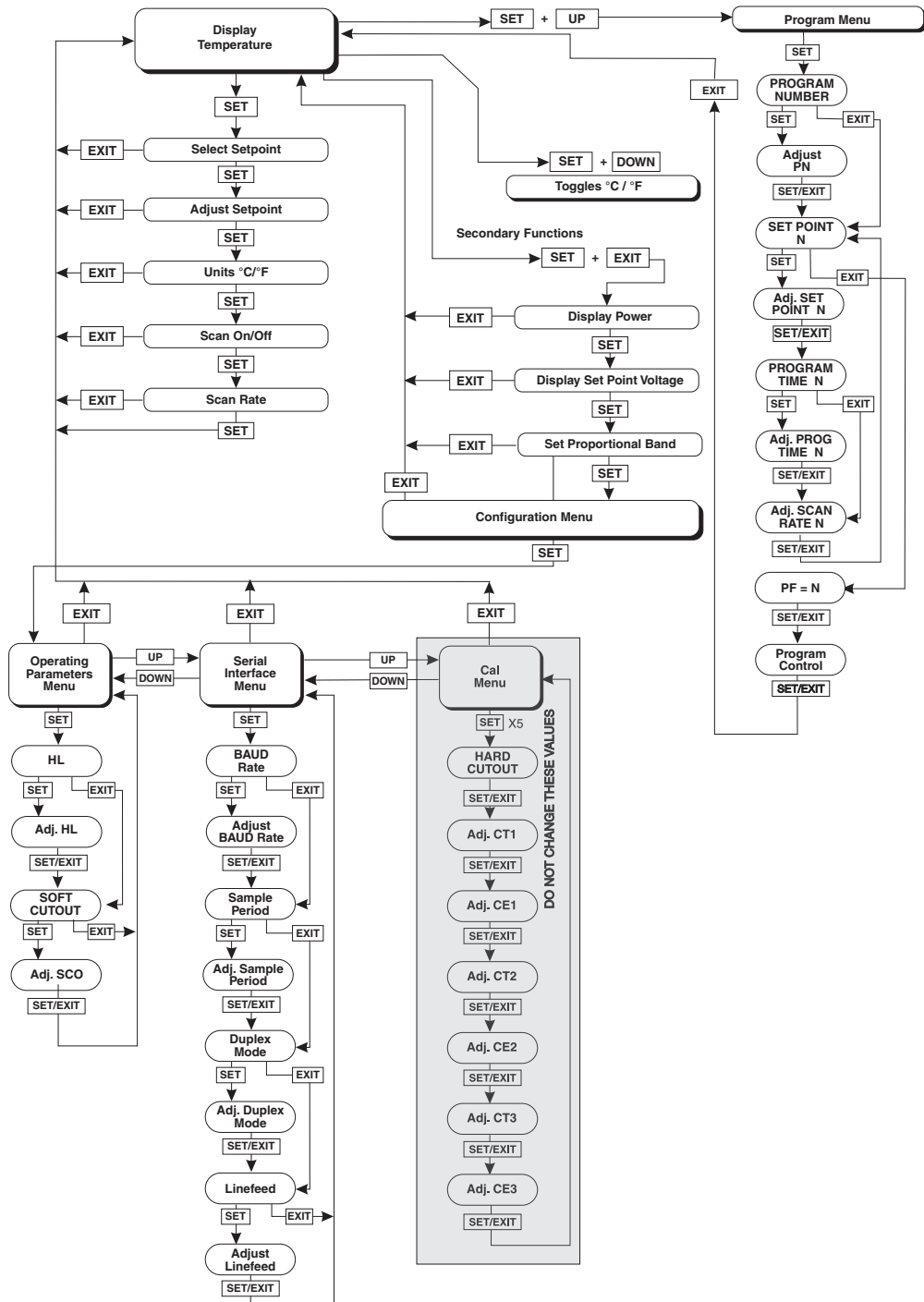


Figure 5. Controller Operation Flowchart



*Access set-point memory*

1. 25

*Set-point memory 1, 25.0°C currently used*

To change the set-point memory press “UP” or “DOWN”.

5. 962

*New set-point memory 5, 962.0°C*

Press “SET” to accept the new selection and access the set-point value.



*Accept selected set-point memory*

## 6.2.2 Set-point Value

The set-point value may be adjusted after selecting the set-point memory and pressing “SET”.

0962.0

*Set-point value in °C*

If the set-point value is correct then press “EXIT” to resume displaying the well temperature. Press “UP” or “DOWN” to adjust the sign of the temperature positive and negative. The sign will be flashing on and off. If the sign is correct press “SET”. The first digit of the temperature should now be flashing. Adjust this digit by pressing “UP” or “DOWN”.

0962.7

*New set-point value*

Press “SET” to accept the first digit and repeat until the last digit has been adjusted. Press “SET” to accept the new set-point. If “EXIT” is pressed all changes made to the set-point are discarded.



*Accept new set-point value*

## 6.2.3 Temperature Scale Units

Temperature Scale Units of the controller are set by the user to degrees Celsius (°C) or Fahrenheit (°F). The units are used in displaying the well temperature, set-point, and proportional band.

Press “SET” after adjusting the set-point value to change display units.

U n = C

*Scale units currently selected*

Press “UP” or “DOWN” to change the units.

U n = F

*New units selected*

## 6.3 Scan

The scan rate can be set and enabled so that when the set-point is changed the furnace heats or cools at a specified rate (degrees per minute) until it reaches the new set-point. With the scan disabled the furnace heats or cools at the maximum possible rate.

### 6.3.1 Scan Control

The scan is controlled with the scan on/off function that appears in the main menu after the set-point function.

 *Scan function off*

Press “UP” or “DOWN” to toggle the scan on or off.

 *Scan function on*

Press “SET” to accept the present setting and continue.

 *Accept scan setting*

### 6.3.2 Scan Rate

The next function in the main menu is the scan rate. The scan rate can be set from .1 to 99.9°C/minute. The maximum scan rate however is actually limited by the natural heating or cooling rate of the instrument. This rate is often less than 100°C/minute, especially when cooling.

The scan rate function appears in the main menu after the scan control function. The scan rate units are in degrees C per minute.

 *Scan rate in °C/min.*

Press “UP” or “DOWN” to change the scan rate.

 *New scan rate*

Press “SET” to accept the new scan rate and continue.

 *Accept scan rate*

## 6.4 Ramp and Soak Program

The ramp and soak program feature for the 9115 allows the user to program a number of set-points, cycle the furnace automatically between the temperatures at a scan rate set by the user, and hold the furnace at each temperature for a period of time set by the user. The user can select one of four different cycle func-

tions. The Ramp and Soak Menu is accessed by pressing “SET” and “UP” simultaneously.

### 6.4.1 Program Points

The 9115 contains eight “program points”. Each program point contains a set-point, scan rate, and soak time. When the unit is in program mode the unit heats or cools to the current program set-point at the current program scan rate. Once the program set-point is reached the unit waits for the program soak time before heating or cooling to the next program set-point. To access the Ramp and Soak Program Menu press “SET” and “UP” simultaneously.

962.4 *Well temperature*

 +  *Access Ramp and Soak Program Menu*

### 6.4.2 Number of Program Points


The first parameter in the program menu is the number of program points to cycle through. Up to 8 set-points can be used in a ramp and soak program.

P n = 8 *Number of program points to cycle through*

Use the “UP” and “DOWN” buttons to change the program points. The valid range is from 2 to 8.

P n = 4 *Number of program points*

Press “SET” to continue. Pressing “EXIT” causes any changes made to the parameter to be discarded.

 *Accept the new number of program points.*

### 6.4.3 Editing Program Set-Points

The controller allows the user to adjust up to eight program points. These are accessed by pressing “SET” after setting the number of program points as described in Section 6.4.2. Each program point has three associated parameters: the program set-point, the program scan rate, and the program hold (or soak) time. After adjusting the number of program points press “SET”.

SP 1 *Program point 1*

Use the “UP” or “DOWN” buttons to select any of the program points. The controller only allows the user to edit program points that are less than or equal to the number of programs points selected as explained in Section 6.4.2. For example, if the user has selected 4 program points program points 5, 6, 7, and 8 cannot be edited.

SP 4 *Program point 4*

Press “SET” to edit a program point.

 *Edit program point*


The first value to edit is the program set-point.

+ 0962.7 *Program set-point value in °C*

Use “UP”, “DOWN”, and “SET” to adjust the set-point as each digit flashes.

+ 0970.0 *New program set-point value for program point 4*


Press “SET” to save the new set-point value or “EXIT” to discard changes.

 *Accept the program point set-point*

The next value to edit is the program soak time.

Pt 4 *Program point 4 soak time*


Press “SET” to edit the program soak time.

 *Edit program point soak time*

Use “UP”, “DOWN”, and “SET” to adjust the program soak time. This value can be any integer from 0 to 14400. This time is the minutes the program set-point maintains after the temperature of the furnace has settled and before proceeding to the next set-point. Each digit flashes individually to indicate that it can be adjusted.

00200 *Program point 4 soak time set for 200 minutes*


Press “SET” to save the new soak-time value or “EXIT” to discard changes

 *Accept the program point soak time*

The next value to edit is the program scan rate. This value is ignored if scan is not enabled for the unit (See Section 6.3.1).

Sc 4 *Program point 4 scan rate*

Press “SET” to edit the program scan rate.

 *Edit the program point scan rate*

10.0 *Current program point 4 scan rate*

Use “UP” and “DOWN” to adjust the program scan rate.

11.3 *New program point 4 scan rate*

Press “SET” to save the new scan rate value.

SET *Accept the program point scan rate*

After “SET” is pressed the controller advances to the next program point or, if there are no more program points to edit, exits to the Program Function Menu. Repeat the above steps to edit any program point.

## 6.4.4 Program Function Mode

The next parameter is the program function or cycle mode. There are four possible modes which determine whether the program scans up (from set-point 1 to n) only or both up and down (from set-point n to 1), and also whether the program stops after one cycle or repeats the cycle indefinitely. The table below shows the action of each of the four program mode settings.

Function	Action
1	up-stop
2	up-down-stop
3	up-repeat
4	up-down-repeat

PF = 1 *Program mode*

Use the “UP” or “DOWN” buttons to change the mode.

PF = 4 *New mode*

Press “SET” to continue.

SET *Save new setting*

## 6.4.5 Program Control

The final parameter in the program menu is the control parameter. You may choose between three options to either start the program from the beginning, continue the program from where it was when it was stopped, or stop the program.

P = OFF *Program presently off*

Use the "UP" or "DOWN" buttons to change the status.

P = 00

*Start cycle from beginning*

or

P = cont

*Continue the program from where it was when it was stopped*

Press “SET” to activate the new program control command and return to the temperature display.

SET

*Activate new command*

## 6.5 Secondary Menu

Functions which are used less often are accessed within the secondary menu. The secondary menu is accessed by pressing “SET” and “EXIT” simultaneously and then releasing. The first function in the secondary menu is the heater power display. (See Figure 5 on page 22.)

## 6.6 Heater Power

The temperature controller controls the temperature of the furnace by pulsing the heater on and off. The total power being applied to the heater is determined by the duty cycle or the ratio of heater on time to the pulse cycle time. By knowing the amount of heating the user can tell if the calibrator is heating up to the set-point, cooling down, or controlling at a constant temperature. Monitoring the percent heater power lets the user know how stable the well temperature is. With good control stability the percent heating power should not fluctuate more than  $\pm 1\%$  within one minute.

The heater power display is accessed in the secondary menu. Press “SET” and “EXIT” simultaneously and release. The heater power is displayed as a percentage of full power.

962.4

*Well temperature*

SET + EXIT

*Access heater power in secondary menu*

41.5

*Heater power in percent*

To exit out of the secondary menu press “EXIT”. To continue on to the proportional band setting function press “SET”.

## 6.7 Proportional Band

In a proportional controller such as this the heater output power is proportional to the well temperature over a limited range of temperatures around the



set-point. This range of temperature is called proportional band. At the bottom of the proportional band the heater output is 100%. At the top of the proportional band the heater output is 0. Thus as the temperature rises the heater power is reduced, which consequently tends to lower the temperature back down. In this way the temperature is maintained at a fairly constant temperature.


The temperature stability of the well and response time depend on the width of the proportional band. If the band is too wide the well temperature deviates excessively from the set-point due to varying external conditions. This deviation is because the power output changes very little with temperature and the controller cannot respond very well to changing conditions or noise in the system. If the proportional band is too narrow the temperature may swing back and forth because the controller overreacts to temperature variations. For best control stability the proportional band must be set for the optimum width.

The proportional band width is set at the factory to about 30.0°C. The proportional band width may be altered by the user if he desires to optimize the control characteristics for a particular application.


The proportional band width is easily adjusted from the front panel. The width may be set to discrete values in degrees C or F depending on the selected units. The proportional band adjustment can be accessed within the secondary menu. Press “SET” and “EXIT” to enter the secondary menu and show the heater power. Then press “SET” twice to access the proportional band.

 +  *Access heater power in secondary menu*

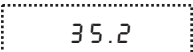
 *Heater power in percent*


 *Access set-point voltage*

 *Set-point voltage in millivolts*

 *Access proportional band*

To change the proportional band press “UP” and “DOWN”. Pressing “EXIT” exits the secondary menu ignoring any changes just made to the proportional band value.

 *Proportional band*

 *Accept the new proportional band setting*

## 6.8 Controller Configuration

The controller has a number of configuration and operating options and calibration parameters which are programmable via the front panel. These are accessed from the secondary menu after the proportional band function by pressing “SET”. Pressing “SET” again enters the first of three groups of configuration parameters—operating parameters, serial interface parameters and calibration parameters. The groups are selected using the “UP” and “DOWN” keys and then pressing “SET”.

## 6.9 Operating Parameters

The operating parameters menu is indicated by...

 *Operating parameters menu*

Press “SET” to enter the menu. The operating parameters menu contains the HL (High Limit) parameter and the Soft cut-out Parameter.

### 6.9.1 High Limit

The HL parameter adjusts the upper set-point temperature. The factory default and maximum are set to 1000. For safety, a user can adjust the HL down so the maximum temperature set-point is restricted.

 *High Limit parameter*

Press “SET” to enable adjustment of HL.

 *Current HL setting*

Adjust the HL parameter digit by digit using “UP”, “DOWN”, and “SET” as each digit flashes.

 *New HL setting*

Press “SET” to accept the new temperature limit.

### 6.9.2 Soft Cut-out

The “Soft Cut-out” temperature parameter is used by the controller to shut the unit down during over-temperature conditions.

 *Soft Cut-out parameter*

Press “SET” to enable adjustments of Soft Cut-out

 *Current Soft Cut-out setting*

Adjust this parameter by using “UP”, “DOWN”, and “SET” as each digit flashes.

`975.0` *New Soft Cut-out setting*

Press “SET” to accept the new temperature limit.

If the temperature of the unit is ever greater than the “Soft Cut-out” temperature the controller shuts itself down and displays, alternately, “5℃0.0” and “Err B”.

## 6.10 Serial Interface Parameters

The serial RS-232 interface parameters menu is indicated by,

`SERIAL` *Serial RS-232 interface parameters menu*

The serial interface parameters menu contains parameters which determine the operation of the serial interface. These controls only apply to instruments fitted with the serial interface. The parameters in the menu are: BAUD rate, sample period, duplex mode, and linefeed.

### 6.10.1 Baud Rate

The baud rate is the first parameter in the menu. The baud rate setting determines the serial communications transmission rate.

The baud rate parameter is indicated by,

`BAUD` *Serial baud rate parameter*

Press “SET” to choose to set the baud rate. The current baud rate value is then be displayed.

`2400 b` *Current baud rate*

The baud rate of the serial communications may be programmed to 300 600, 1200, 2400, 4800, or 9600 baud. 2400 baud is the default setting. Use “UP” or “DOWN” to change the baud rate value.

`4800 b` *New baud rate*

Press “SET” to set the baud rate to the new value or “EXIT” to abort the operation and skip to the next parameter in the menu.

### 6.10.2 Sample Period

The sample period is the next parameter in the serial interface parameter menu. The sample period is the time period in seconds between temperature measurements transmitted from the serial interface. If the sample rate is set to 5, the in-

strument transmits the current measurement over the serial interface approximately every five seconds. The automatic sampling is disabled with a sample period of 0. The sample period is indicated by,

`S P E r` *Serial sample period parameter*

Press “SET” to choose to set the sample period. The current sample period value is displayed.

`S P = 1` *Current sample period (seconds)*

Adjust the value with “UP” or “DOWN” and then use “SET” to set the sample rate to the displayed value.

`S P = 6 0` *New sample period*

### 6.10.3 Duplex Mode

The next parameter is the duplex mode. The duplex mode may be set to full duplex or half duplex. With full duplex any commands received by the calibrator via the serial interface are immediately echoed or transmitted back to the device of origin. With half duplex the commands are executed but not echoed. The duplex mode parameter is indicated by,

`d U P L` *Serial duplex mode parameter*

Press “SET” to access the mode setting

`d = F U L L` *Current duplex mode setting*

The mode may be changed using “UP” or “DOWN” and pressing “SET”.

`d = H A L F` *New duplex mode setting*

### 6.10.4 Linefeed

The final parameter in the serial interface menu is the linefeed mode. This parameter enables (on) or disables (off) transmission of a linefeed character (LF, ASCII 10) after transmission of any carriage-return. The linefeed parameter is indicated by,

`L F` *Serial linefeed parameter*

Press “SET” to access the linefeed parameter.

`L F = 0 n` *Current linefeed setting*

The mode may be changed using “UP” or “DOWN” and pressing “SET”.

LF=OFF

*New linefeed setting*

## 6.11 Calibration Parameters

The operator of the 9115 has access to the furnace calibration constants. These values are set at the factory and must not be altered. The correct values are important to the accuracy and proper and safe operation of the furnace. Access to these parameters is available to the user only so that in the event the controller memory fails, the user may restore these values to the factory settings. The user should have a list of these constants and their settings with the manual.



**CAUTION: DO NOT** change the values of the furnace calibration constants from the factory set values. The correct settings of these parameters is important to the safety and proper operation of the furnace.

The calibration parameters menu is indicated by,

CR L

*Calibration parameters menu*

Press "SET" five times to enter the menu. The calibration parameters menu contains the parameters Hard Cutout, CT1, CE1, CT2, CE2, CT3, and CE3.

### 6.11.1 Hard Cutout

This parameter is the temperature above which the unit shuts down automatically. The parameter is set at the factory to approximately 1025°C and cannot be changed by the user.

Cut-out

*Hard Cutout display*

Press "SET" to display the current Hard Cutout value. This parameter can only be changed internally. Contact Hart Scientific Customer Service if the parameter needs to be changed.

Press "SET" or "EXIT" to skip to the next parameter.

### 6.11.2 CT1, CT2, CT3

The calibration parameters CT1, CT2, and CT3 are the calibration temperatures.

### 6.11.3 CE1, CE2, CE3

The calibration parameters CE1, CE2, and CE3 are the calibration errors corresponding to the calibration temperatures.

# 7 Digital Communication Interface

The furnace is capable of communicating with and being controlled by other equipment through the RS-232 digital interface.

With a digital interface the instrument may be connected to a computer or other equipment. This allows the user to set the set-point temperature, monitor the temperature, and access any of the other controller functions, all using remote communications equipment.

## 7.1 Serial Communications

The calibrator is installed with an RS-232 serial interface that allows serial digital communications over fairly long distances. With the serial interface the user may access any of the functions, parameters and settings discussed in Section 6 with the exception of the BAUD rate setting.

### 7.1.1 Wiring

The serial communications cable attaches to the calibrator through the DB-9 connector at the back of the instrument. Figure 6 shows the pin-out of this connector and suggested cable wiring. To eliminate noise, the serial cable should be shielded with low resistance between the connector (DB9) and the shield.

### 7.1.2 Setup

Before operation the serial interface must first be set up by programming the BAUD rate and other configuration parameters. These parameters are programmed within the serial interface menu.

To enter the serial parameter programming mode first press “EXIT” while pressing “SET” and release to enter the secondary menu. Press “SET” repeatedly until the display reads “CONFIG”. This is the menu selection. Press “UP” repeatedly until the serial interface menu is indicated with “SERIAL”. Finally press “SET” to enter the serial parameter menu. In the serial interface parameters menu are the BAUD rate, the sample rate, the duplex mode, and the linefeed parameter.

#### RS-232 Cable Wiring for IBM PC and Compatibles

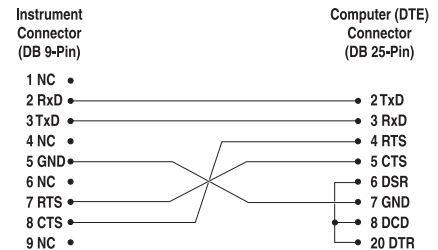
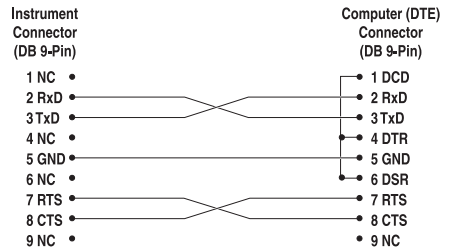


Figure 6. Serial Cable Wiring

### 7.1.2.1 Baud Rate

The baud rate is the first parameter in the menu. The display will prompt with the baud rate parameter by showing “*BAUD*”. Press “SET” to choose to set the baud rate. The current baud rate value will then be displayed. The baud rate of the 9115 serial communications may be programmed to 300, 600, 1200, 2400, 4800, or 9600 baud. The baud rate is pre-programmed to 2400 baud. Use “UP” or “DOWN” to change the baud rate value. Press “SET” to set the baud rate to the new value or “EXIT” to abort the operation and skip to the next parameter in the menu.

### 7.1.2.2 Sample Period

The sample period is the next parameter in the menu and prompted with “*SAMPLE*”. The sample period is the time period in seconds between temperature measurements transmitted from the serial interface. If the sample rate is set to 5 for instance then the instrument will transmit the current measurement over the serial interface approximately every five seconds. The automatic sampling is disabled with a sample period of 0. Press “SET” to choose to set the sample period. Adjust the period with “UP” or “DOWN” and then use “SET” to set the sample rate to the displayed value.

### 7.1.2.3 Duplex Mode

The next parameter is the duplex mode indicated with “*dUPL*”. The duplex mode may be set to half duplex (“*HALF*”) or full duplex (“*FULL*”). With full duplex any commands received by the thermometer via the serial interface will be immediately echoed or transmitted back to the device of origin. With half duplex the commands will be executed but not echoed. The default setting is full duplex. The mode may be changed using “UP” or “DOWN” and pressing “SET”.

### 7.1.2.4 Linefeed

The final parameter in the serial interface menu is the linefeed mode. This parameter enables (“*On*”) or disables (“*OFF*”) transmission of a linefeed character (LF, ASCII 10) after transmission of any carriage-return. The default setting is with linefeed on. The mode may be changed using “UP” or “DOWN” and pressing “SET”.

## 7.1.3 Serial Operation

Once the cable has been attached and the interface set up properly the controller will immediately begin transmitting temperature readings at the programmed rate. The serial communications uses 8 data bits, one stop bit, and no parity. The set-point and other commands may be sent via the serial interface to set the temperature set-point and view or program the various parameters. The interface commands are discussed in [Section 7.2](#). All commands are ASCII character strings terminated with a carriage-return character (CR, ASCII 13).

## 7.2 Interface Commands

The various commands for accessing the calibrator functions via the digital interfaces are listed in this section (see Table 2 starting on page 38). These commands are used with the RS-232 serial interface. The commands are terminated with a carriage-return character. The interface makes no distinction between upper and lower case letters, hence either may be used. Commands may be abbreviated to the minimum number of letters which determines a unique command. A command may be used to either set a parameter or display a parameter depending on whether or not a value is sent with the command following a “=” character. For example “s”<CR> will return the current set-point and “s=50.00”<CR> will set the set-point to 50.00 degrees.

In the following list of commands, characters or data within brackets, “[” and “]”, are optional for the command. A slash, “/”, denotes alternate characters or data. Numeric data, denoted by “n”, may be entered in decimal or exponential notation. Spaces may be added within command strings and will simply be ignored. Backspace (BS, ASCII 8) may be used to erase the previous character. A terminating CR is implied with all commands.



**Table 2.** Digital Communications Commands

Command Description	Command Format	Command Example	Returned	Returned Example	Acceptable Values
<b>Display Temperature</b>					
Read current set-point	s[etpoint]	s	set: 9999.9 {C or F}	set: 150.00 C	
Set current set-point to <i>n</i>	s[etpoint]= <i>n</i>	s=450			Instrument Range
<b>Set temperature units:</b>	<b>u[nits]=c/f</b>				C or F
Set temperature units to Celsius	u[nits]=c	u=c			
Set temperature units to Fahrenheit	u[nits]=f	u=f			
Read scan function	sc[an]	sc	scan: {ON or OFF}	scan: ON	
<b>Set scan function:</b>	<b>sc[an]=on/off[f]</b>				ON or OFF
Turn scan function on	sc[an]=on	sc=on			
Turn scan function off	sc[an]=off[f]	sc=of			
Read scan rate	sr[ate]	sr	srat: 999.99 {C or F}/min	srat: 10.0 C/min	
Set scan rate to <i>n</i> degrees per minute	sr[ate]= <i>n</i>	sr=5			.1 to 99.9
<b>Secondary Menu</b>					
Read proportional band setting	pr[op-band]	pr	pb: 999.9	pr: 15.9	
Set proportional band to <i>n</i>	pr[op-band]= <i>n</i>	pr=8.83			0.1 to 100
Read heater power (duty cycle)	po[wer]	po	p%: 999.9	po: 1	
<b>Ramp and Soak Menu</b>					
Read number of programmable set-points	pn	pn	pn: 9	pn: 2	
Set number of programmable set-points to <i>n</i>	pn= <i>n</i>	pn=4			1 to 8
Read programmable set-point number <i>n</i>	ps <i>n</i>	ps3	ps <i>n</i> : 9999.99 {C or F}	ps1: 50.00 C	
Set programmable set-point number <i>n</i> to <i>n</i>	ps <i>n</i> = <i>n</i>	ps3=50			1 to 8, Instrument Range
Read program set-point soak time	pt <i>n</i>	pt3	ti: 999	ti: 5	
Set program set-point soak time to <i>n</i> minutes	pt <i>n</i> = <i>n</i>	pt3=5			0 to 14400
Read program scan rate	px <i>n</i>	px3	sr <i>n</i> : 99.9	sr3: 11.3	
Set program scan rate	px <i>n</i> = <i>n</i>	px3=10			.1 to 99.9
Read program control mode	pc	pc	prog: {OFF or ON}	prog: OFF	
<b>Set program control mode:</b>	<b>pc=g[o]/s[top]/c[ont]</b>				GO or STOP or CONT
Start program	pc=g[o]	pc=g			

Command Description	Command Format	Command Example	Returned	Returned Example	Acceptable Values
Stop program	pc=s[top]	pc=s			
Continue program	pc=c[ont]	pc=c			
Read program function	pf	pf	pf: 9	pf: 3	
Set program function to <i>n</i>	pf= <i>n</i>	pf=2			1 to 4
<b>Configuration Menu</b>					
<b>Operating Parameters Menu</b>					
Read soft cutout	scut	scut	scut: 9999.9	scut: 1150.0	
<b>Set soft cutout setting:</b>	<b>cu[tout]=<i>n</i></b>				
Set soft cutout to <i>n</i> degrees	cu[tout]= <i>n</i>	cu=500			0.0 to 1150.0
<b>Serial Interface Menu</b>					
Read serial sample setting	sa[mple]	sa	sa: 9	sa: 1	
Set serial sampling setting to <i>n</i> seconds	sa[mple]= <i>n</i>	sa=0			0 to 4000
<b>Set serial duplex mode:</b>	<b>du[plex]=f[ull]/h[alf]</b>				FULL or HALF
Set serial duplex mode to full	du[plex]=f[ull]	du=f			
Set serial duplex mode to half	du[plex]=h[alf]	du=h			
<b>Set serial linefeed mode:</b>	<b>lf[eed]=on/off</b>				ON or OFF
Set serial linefeed mode to on	lf[eed]=on	lf=on			
Set serial linefeed mode to off	lf[eed]=off	lf=of			
<b>Cal Menu</b>					
Read CT <i>n</i> calibration parameter	ct <i>n</i>	ct1	ct <i>n</i> : 9999.9C	ct1: 550.0C	
Set CT <i>n</i> calibration parameter to <i>n</i>	ct <i>n</i> = <i>n</i>	ct1=550.0			0 to 1000
Read CE <i>n</i> calibration parameter	ce <i>n</i>	ce1	ce <i>n</i> : 99.9C	ce1: -10.1C	
Set CE <i>n</i> calibration parameter to <i>n</i>	ce <i>n</i> = <i>n</i>	ce1=-10			-99.9 to 99.9
<b>These commands are only used for factory testing.</b>					
<b>Miscellaneous (not on menus)</b>					
Read firmware version number	*ver[sion]	*ver	ver.9999,9.99	ver.9122,3.54	
Read structure of all commands	h[elp]	h	list of commands		
Legend:	[] Optional Command data {} Returns either information n Numeric data supplied by user 9 Numeric data returned to user x Character data returned to user				
Note:	When DUPLEX is set to FULL and a command is sent to READ, the command is returned followed by a carriage return and linefeed. Then the value is returned as indicated in the RETURNED column.				

## 8 Freeze Point Realization

### 8.1 General

This discussion assumes SPRT calibrations at the silver point. Other freeze points are similar.

Successful silver point realization requires a cell of the following specifications:

- The purity of silver: 99.9999%
- The reproducibility: 2 mK
- The expanded uncertainty: 10 mK<sup>†</sup>
- The outer diameter of the cell: 48 mm

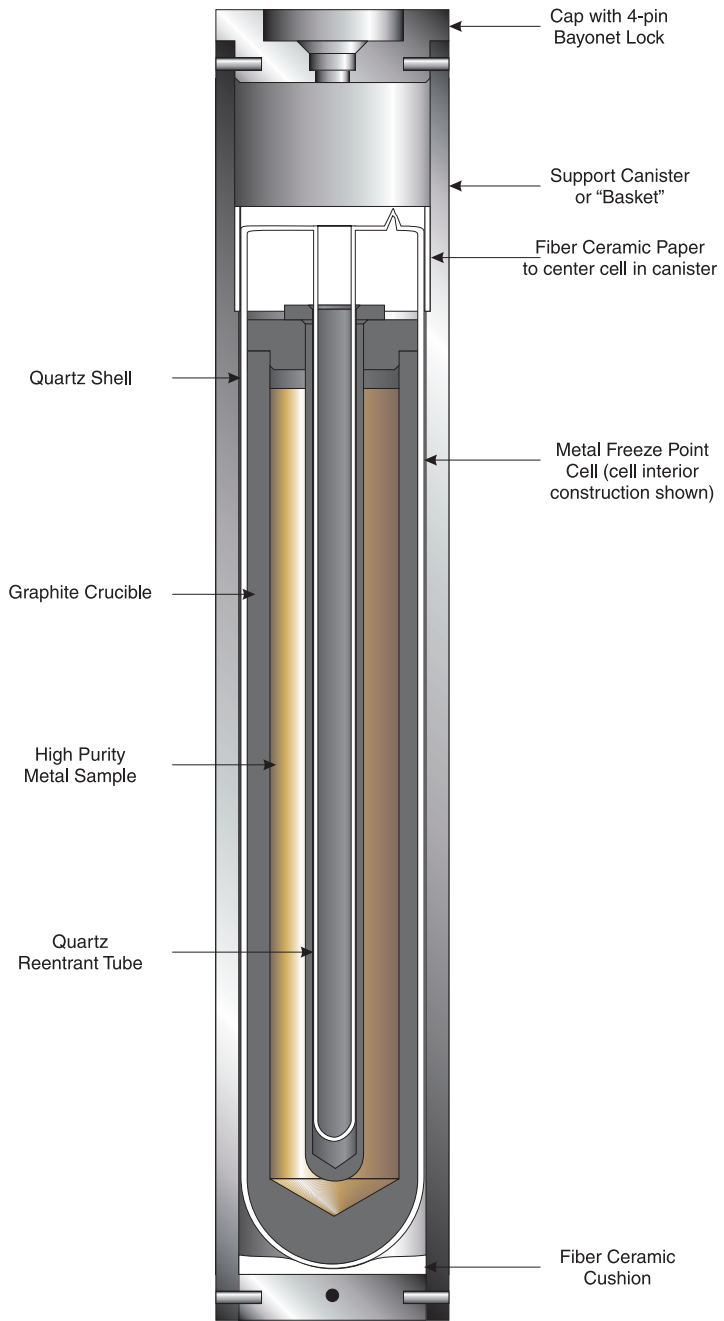
<sup>†</sup>The expanded uncertainty was evaluated at the level of two standard deviations (95% confidence).

### 8.2 Installing the Metal Freeze Point Cell

A metal freeze point cell must always be handled with extreme care due to its high value and fragility. It must also be kept free of any foreign material such as finger oils. Alkaline from these oils cause devitrification or physical breakdown of the quartz shell. **Handle the cell with cotton gloves. Discard the gloves before they become appreciably soiled.** Any foreign material should be carefully removed with high purity alcohol. Refer to [Figure 7](#) on page 42. Note: The support canister must also be free of oils and other contaminating materials.

The freeze point cell is first installed into the support canister. The cell must be laid on its side for installation. Use especially gentle handling since there may be considerable stress on the reentrant tube from the weight of the metal sample and graphite crucible. With both the cell and the support canister on their sides, carefully slide the cell into the canister opening and push it against the fiber ceramic cushion on the bottom of the canister. To reduce friction and to prevent scratching the quartz, a strip of paper may be inserted part way into the canister and under the cell during the sliding process. Use very clean paper such as Kim Wipes™ cut approximately 2 inches wide. Carefully turn the canister and cell upright and remove all of the paper strip. Two people are required to complete the process. To center the cell, shim the cell with fiber ceramic paper. Always leave enough space around the edge of the cell to enable you to remove it.

Install the cap and rotate to the “locked” position. The cap fits very loosely to prevent binding when oxidized. The 4 pins pull into grooves to help them maintain their position while lowering the assembly into and removing it from the furnace.



**Figure 7.** Metal Freeze Point Cell installed in canister

Lower the assembly onto the furnace using the tongs provided. The tips of the tongs fit into the holes inside the top of the cap. Make sure the cap stays in the grooves during the process. Removal of the cell is in reverse of this process.

Install the Heater Liner Top, Thermal Guard, and insulation as shown in [Figure 1 Furnace Core](#) on page 10.

Once the freeze point cell is properly installed, you are prepared to heat the furnace and realize the freeze point for calibration purposes.

## 8.3 How to realize the freezing point of silver

- 1) **Melting the Cell:** Switch on the power to the furnace from the front panel. The temperature ramp rate should not be too high and should be set below the minimum operating temperature of the sodium heat pipe, which is about 500°C. Heating from room temperature to 550°C should take approximately two hours and then another hour from 550°C to 970°C. The ramp rates are programmable from the controller. Use a working high temperature platinum resistance thermometer to monitor the temperature in the cell. When the silver sample begins to melt, the temperature will stop rising and remain almost constant during the melting process. Write down the resistance or temperature indicated by the working thermometer at the melting point for future reference.
- 2) As soon as the silver sample is melted completely, set the furnace at a temperature of 1 to 1.5°C higher than the freezing point. Maintain a stable temperature for twenty minutes. Then let the temperature of the furnace decrease at a rate of 0.2 to 0.3°C per minutes until the temperature indicated by the working thermometer stops decreasing and starts to rise. This indicates that freezing has started. Usually the silver may supercool by an amount approximately one degree Celsius or more before the start of freezing. Take the working thermometer out of the furnace and put the thermometer to be calibrated into the furnace. Meanwhile, maintain the temperature of the furnace at a temperature between 0.5 and 1.0°C lower than the freezing point.
- 3) **Initiating the Freeze and Making Measurements:** The freezing curve usually lasts more than four hours and the temperature in the first half of the freezing curve is usually stable within 0.2 mK or 0.3 mK. If the temperature of the furnace is closer to the freezing point, a longer freezing curve can be obtained. It is not difficult to get a freezing curve longer than ten hours or more if the temperature of the furnace is carefully controlled.

The first thermometer to be calibrated should not be preheated. The cold thermometer will enhance the rate of freezing at the beginning of freezing, i.e. it will “induce” the freezing.

Take the average of several readings of a thermometers resistance over a period of about ten minutes. This is the resistance at the freezing point of silver  $R_{Ag}$ . It is possible to calibrate several thermometers during one freezing curve.

Since a cold thermometer absorbs a large amount of heat, which shortens the freezing curve greatly, it is suggested that the subsequent thermometers to be calibrated be preheated to a temperature very near the freezing point before inserting it into the silver cell. Another advantage of preheating is the equilibrium time in the cell may be shortened by nearly one-half, i.e. from about twenty minutes to ten.

Preheat the thermometers for twenty minutes or so near the freezing point. Do not preheat thermometers for too long, it is unnecessary and the thermometer sensors could possibly be contaminated if preheated for a long period of time.

- 4) **SPRT Annealing:** The rapid cooling from the freezing point of silver to room temperature will introduce extra crystal defects-vacancies in the platinum wire of the thermometer-resulting in a noticeable increase in resistance at the triple point of water ( $R_{tp}$ ). Sometimes a change larger than the equivalent of 30 mK can be observed. An appropriate annealing will get rid of these defects and will return the  $R_{tp}$  to the equilibrium value. Anneal the thermometer at 700°C for two hours in a clean furnace and then cool it from 700°C to 450°C over three hours. An alternative annealing procedure is to anneal at 970°C for 30 minutes and then cool at a constant rate to 500°C over a period of four hours. After annealing the thermometer, take it out of the furnace and cool it down to room temperature in air. Measure the  $R_{tp}$  and calculate the resistance ratio  $R_{Ag}$ :

$$W_{Ag} = \frac{R_{Ag}}{R_{tp}}$$

## 8.4 Safety Precautions



***CAUTION: Sealed cells for freezing points are delicate devices and the quartz shell is prone to be broken. THE CELL MUST BE HANDLED WITH EXTREME CARE.***

Maintain the cell in vertical orientation for safety. Although putting the cell in horizontal orientation for a short period of time may not cause any damage, transporting the cell by any means while in this position is dangerous. Transporting a cell by common carrier is also dangerous. The cell should be hand carried from one place to another. Keep the surface of the cell clean.

**Never touch the cell with bare hands. When handling the cell, wear gloves.**

## 9 Calibration Procedure



**CAUTION:** *The vertical gradient needs to be checked before calibrating the furnace. Checking the vertical gradient insures that the sodium heat-pipe is working properly.*

At times the user may want to calibrate the unit to improve the temperature set-point accuracy. Calibration is done by adjusting the controller probe offset adjustments CE1, CE2, and CE3 so that the temperature of the unit, as measured with a standard thermocouple, agrees more closely with the set-point. The thermometer used must be able to measure the well temperature with higher accuracy (at least 4:1) than the desired accuracy of the unit.

### 9.1 Calibration Points

In calibrating the unit, CE1, CE2, and CE3 are adjusted to minimize the set-point error at each of three different well temperatures. Any three reasonably separated temperatures may be used for the calibration. However, the temperatures selected should cover the entire user selected range of the furnace, e.g., if the furnace will be used for a silver cell, CE3 should be set at 957°C minimum. Improved results can be obtained for shorter ranges when using temperatures that are just within the most useful operating range of the unit. The farther apart the calibration temperatures, the larger will be the calibration range but the calibration error will also be greater over the range.

### 9.2 Calibration Procedure

1. Choose three set-points to use in the calibration of the CE1, CE2, and CE3 parameters. These set-points are generally CT1 = 600°C, CT2 = 800°C and CT3 = 962°C but other set-points may be used if desired or necessary.

If the factory set-points of CT1, CT2 and CT3 are used, leave the values of CE1, CE2 and CE3 at the current settings (see the supplied Report of Calibration).

If the normal set-points are not used, initialize CT1, CT2, and CT3 to the desired set points and CE1, CE2, and CE3 to 0, where CT1 is the low-set point and CT3 is the high set-point.

2. Set the unit to the low set-point. When the unit reaches the set-point and the thermometer reference display is stable (e.g.,  $< 0.1^{\circ}\text{C}$  change in 15 minutes), take a reading from the thermometer. Repeat step 2 for the other two set-points recording them as Tm1, Tm2 and Tm3 respectively.
3. Retrieve the offset adjustments from the unit (CE1, CE2 and CE3).

4. Calculate the new CE1, CE2, and CE3 offset adjustments using the following formula:

$$T_m(n) - CT(n) + CE(n) = CE(m)$$

Where,

$T_m(n)$ =The measured temperature

$CT(n)$ =The set-point temperature

$CE(n)$ =The old value for the offset adjustment

$CE(m)$ =The new value for the offset adjustment

$n=1, 2$  or  $3$

$m=1, 2$  or  $3$

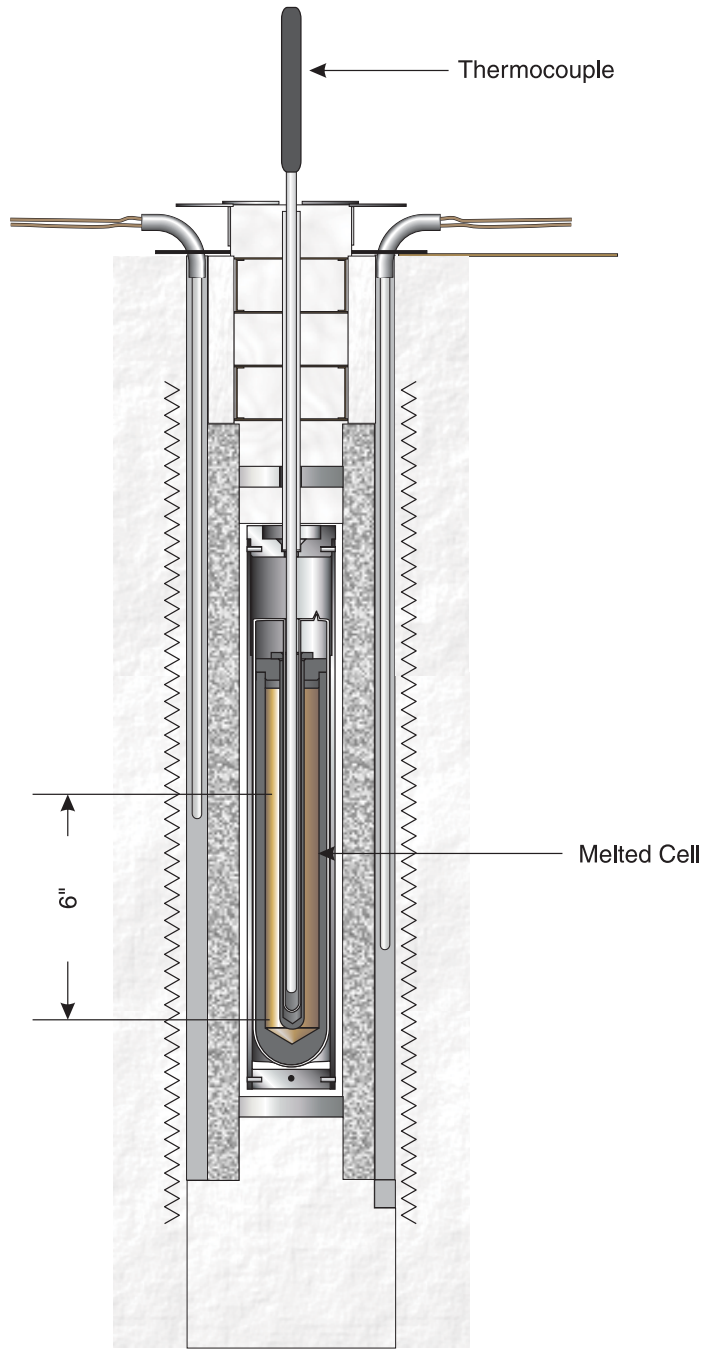
5. Enter new  $CE(m)$  values in the calibration parameter menu using either the keypad or through the serial port.
6. Repeat steps 2 through 5 if required accuracy is not obtained.



# 10 Maintenance

The calibration instrument has been designed with the utmost care. Ease of operation and simplicity of maintenance have been a central theme in the product development. Therefore, with proper care the instrument should require very little maintenance. Avoid operating the instrument in an oily, wet, dirty, or dusty environment.

- If the outside of the instrument becomes soiled, it may be wiped clean with a damp cloth and mild detergent. Do not use harsh chemicals on the surface which may damage the paint.
- Be sure that the well of the furnace is kept clean and clear of any foreign matter. **DO NOT** use fluids to clean out the well.
- If a hazardous material is split on or inside the equipment, the user is responsible for taking the appropriate decontamination steps as outlined by the national safety council with respect to the material.
- If the mains supply cord becomes damaged, replace it with a cord of the appropriate gauge wire for the current of the instrument. If there are any questions, call Hart Scientific Customer Service for more information.
- Before using any cleaning or decontamination method except those recommended by Hart, users should check with Hart Scientific Customer Service to be sure that the proposed method will not damage the equipment.
- If the instrument is used in a manner not in accordance with the equipment design, the operation of the furnace may be impaired or safety hazards may arise.
- The over-temperature cut-out should be checked every 6 months to see that it is working properly. In order to check the user selected cut-out, follow the controller directions ([Section 6](#)) for setting the cut-out.
- **Adjustment of Temperature Uniformity:** Vertical uniformity should be measured in a freeze point cell with the metal melted. The vertical temperature uniformity in the cell should be within  $\pm 0.05^{\circ}\text{C}$  for a distance of six inches upwards from the bottom of the central well (See Figure 8 on page 48). A periodic check of the temperature uniformity using a Type R or Type S thermocouple is recommended at least once every year.
- **Check of the Controller Set-point Accuracy:** This test is carried out in a metal freeze point cell where the metal has been completely melted. Prepare the furnace in the same fashion as though a freeze plateau would be conducted up to the point that the metal sample is melted. This example illustrates measurements made near the silver point.
- Set the temperature of the furnace at  $964^{\circ}\text{C}$  and allow it to stabilize as would be done in preparation for a freeze. Measure the EMF of a thermocouple inserted into the cell. Compare the measured EMF to one taken at the M.P. or F.P. The actual temperature,  $t$ , in the cell can be calculated by using the following equation:



**Figure 8.** Testing Uniformity

$$t = 961.78^{\circ}\text{C} + \frac{E_1 - E_0}{0.0114\text{mV}/^{\circ}\text{C}}$$

where  $E_1$  is the measurement EMF and  $E_0$  is the EMF at the M.P. (961.78°C is the M.P. temperature of copper for this example) and 0.0114mV/°C is the sensitivity of a Type S thermocouple near the M.P. of silver.

For example, the measured EMF  $E_1=9.1502$  mV, the EMF at the M.P.  $E_0=9.1481$  mV, the actual temperature in the furnace.

$$t = 961.78 + \frac{9.1502 - 9.1481}{0.0114} = 961.96^{\circ}\text{C}$$

Since  $t=964.0^{\circ}\text{C}$  = the actual set-point, the error, if any, is very small. If the error is larger than 1°C, you can make an adjustment to the set-point.

# 11 Troubleshooting

If problems arise while operating the 9115, this section provides some suggestions that may help you solve the problem. A wiring diagram is also included.

## 11.1 Troubleshooting

Below are several situations that may arise followed by suggested actions to take for fixing the problem.

Problem	Causes and Solutions
Incorrect temperature reading	Power the unit on and watch the display. If the first number displayed is less than “-0005-”, the unit has been re-initialized. The unit may need to be reprogrammed for CT1, CT2, CT3, CE1, CE2, and CE3. See Section 6.11, Calibration Parameters, on page 33. These numbers can be found on the Report of Calibration that was shipped with the unit.
The unit heats slowly	Check the Scan and Scan Rate setting. The Scan may be on with the Scan Rate set low.
If the display flashes any of the following: “err 1-err 5”	<p>“err 1” – There is a RAM error.            “err 2” – There is a NVRAM error.            “err 3” – There is a RAM error.            “err 4” – There is a ADC set up error.            “err 5” – There is a ADC ready error.</p> <p>Initialize the system by performing the <b>Factory Reset Sequence</b>. If the unit repeats the error code, contact an Authorized Service Center for a return authorization and for instructions on returning the unit.</p> <p><b>Factory Reset Sequence</b> - Hold the “SET” and “EXIT” keys down at the same time while powering up the unit. When the screen displays “-i n i t-” release the keys. The screen then displays the model number and the version of the software. The unit may need to be reprogrammed for CT1, CT2, CT3, CE1, CE2, and CE3 in the calibration menu. See Section 6.11. These numbers can be found on the Report of Calibration that was shipped with the unit.</p>
If the display flashes “err 6”	There is a sensor error. The sensor is disconnected or shorted. Please contact an Authorized Service Center for further instructions.
If the display flashes “err 7”	There is a HtrCTL error. Initialize the unit by performing the <b>Factory Reset Sequence</b> as described above. If the unit repeats the error code, turn the unit off and allow the unit to sit at least one-half hour. Turn the unit back on. If the unit repeats the error code, turn off the unit and contact an Authorized Service Center for assistance.
If the display flashes “err 8”	There is a Soft Cut-out error. Initialize the unit by performing the master reset sequence as described above. The <b>Factory Reset Sequence</b> resets the Soft Cut-out Temperature to the default of 1025°C. If the unit repeats the error code, turn the unit off and allow the unit to sit at least one-half hour. Turn the unit back on. If the unit repeats the error code, turn off the unit and contact an Authorized Service Center for assistance.

<b>Problem</b>	<b>Causes and Solutions</b>
Power Up	The unit is equipped with internal operator accessible fuses. If a fuse blows, it may be due to a power surge or failure of a component. Replace the fuse once. <b>DO NOT</b> replace the fuse with one of a higher current rating. Always replace the fuse with one of the same rating, voltage, and type. If the fuse blows a second time, it is likely caused by failure of a component or part. Contact an Authorized Service Center (see Section 1.4) for assistance.