

FLUKE®

Hart Scientific®

9230

*Gallium Cell
Maintenance System
User's Guide*

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1 Before You Start

1.1 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
	AC-DC
	Battery
	CE
	DC
	Double Insulated
	Electric Shock
	Fuse
	PE Ground
	Hot Surface
	Read the User's Manual
	Off
	On



Canadian Standards Association



C-TIC Australian EMC Mark

CAT II

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.



The European Waste Electrical and Electronic Equipment (WEEE) Directive (2002/96/EC) mark.

1.2 Safety Information

Use of this instrument other than as specified in this manual may impair the protection provided by the instrument.

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.

1.2.1 WARNINGS

To avoid possible electric shock or personal injury, follow these guidelines.

BURN HAZARD – DO NOT leave the melt heater outside of the unit and energized. The melt heater can reach approximately 110°C if not properly inserted into the unit. Place the melt heater in the storage well located at the back of the instrument when not in use.

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

Use only grounded AC mains supply of the appropriate voltage to power the instrument. Refer to Section 3.1, Specifications, for power details.

DO NOT connect this unit to a non-grounded, non-polarized outlet.

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 the power off and disconnect the power cord.

Always replace the fuse with one of the same rating, voltage, and type.

Overhead clearance is required. **DO NOT** place this instrument under a cabinet or other structure. Additionally, at least 6 inches of space between the instrument and nearby objects is necessary for sufficient air circulation.

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 guide.

DO NOT turn the unit upside down with the cell in place; the cell will fall out of the unit.

DO NOT operate near flammable materials.

DO use a ground fault interrupt device.

Before initial use, after transport, and anytime the dry-well has not been energized for more than 10 days, the calibrator must be energized for a dry-out period of 1 to 2 hours before it can be assumed to meet all of the safety requirements of the IEC1010-1.

Follow all safety guidelines listed in the user's manual.

Calibration Equipment should only be used by Trained Personnel.

When the melt heater switch is in the MANUAL ON position, the melt heater is continuously energized and can get hot!

1.2.2

CAUTIONS

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

DO NOT use fluids to clean out the well.

DO NOT slam the probe stems into the entrant well of the gallium cell. The probe could be damaged.

DO use the cell removal tool to remove the cell from the well of the instrument. Grasp the cell with a hand as soon as the cell is removed far enough from the well.

DO NOT use the removal tool to carry the cell. The removal tool is not designed for transporting the cell.

Never introduce foreign material into the well. Fluids, etc. can leak into the calibrator causing damage.

DO use silicon oil or other appropriate heat transfer medium in the entrant well of the gallium cell. Be sure the transfer medium does not leak out into the well of the instrument.

DO NOT change the value of the calibration constant from the factory set value. The correct setting of this parameter is important to the safety and proper operation of the calibrator.

The Auto Program configuration parameters should not be adjusted under normal use. These parameters directly affect the melt plateau duration time and proper freezing of the gallium cell.

Insert only frozen gallium cells into the 9230. **DO NOT** insert a melted cell into the unit and then initiate the Auto Program. An improper freeze may occur resulting in damage to the cell.

1.3 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 Introduction

The 9230 is a specialized instrument for the realization of the gallium melting point (29.7646°C). The instrument works with Hart's 5943 stainless steel Ga cell to provide melting plateaus that can last a week (or longer). This instrument is intended for Calibration Laboratory use and not for field applications. Internal programming of the micro-processor controller provides preprogrammed scan rates, set-points for each step in the process, dwell timing, and indication that the next step is ready. The Model 9230 is available in 100-230 VAC ($\pm 10\%$), 50-60 Hz.

Built in programmable features include:

- Temperature scan rate control
- Fixed-point programming with default settings for the gallium cell
- Eight set-point memories
- Adjustable readout in °C or °F

The temperature is accurately controlled by Hart's special proportional-integral-derivative (PID)/digital controller. The controller uses a platinum RTD as a sensor and controls the well temperature with thermoelectric Peltier devices.

After properly inserting a frozen Ga cell and powering the unit on, the Model 9230 controller sets the block temperature to 25°C and the LED front panel display shows $\mu\text{E}0$. The user then makes sure that the inner melt heater is attached to the back of the unit, the melt heater switch is in the Auto position, and inserts the melt heater into the entrant well of the gallium cell. When the user selects $\mu\text{E}0$ by pressing the SET button, the Model 9230 Auto Program begins a pre-determined program to initiate and maintain a gallium melt plateau. While in the Auto Program mode, the display shows the current process in the gallium melt cycle. During this mode the user may not change those parameters, which could negatively impact the gallium melt. At the end of the plateau the unit properly refreezes the cell and returns the temperature to 25°C (standby) to await the next user initiated melt.

While in the manual mode of operation, the LED front display panel continuously shows the current well temperature. The temperature may be easily set with the control buttons to any desired temperature within the specified range. Certain parameters of the Auto Program itself may be adjusted in the manual mode of operation.

The Model 9230 was designed for high accuracy fixed-point calibration methods and for ease of operation. Through proper use, the instrument will continuously provide accurate calibration of temperature sensors and devices. The user should be familiar with the safety guidelines and operating procedures of the instrument as described in this User's Guide.

3 Specifications and Environmental Conditions

3.1 Specifications

Operating Range	15°C to 35°C (59°F to 95°F)
Gallium Cell Uncertainty	±0.08mK, typical
Gallium Melting Plateau Duration	5 days, typical
Display Accuracy	±0.05°C at 29.76°C
Stability	±0.02°C
Display Resolution	0.01°C or °F (0.001°C or °F in program mode)
Display Scale	°C or °F, switchable
Preheat Wells	2 wells (8.2 mm diameter)
Immersion Depth	152 mm (6 inches) in gallium cell
Vertical Gradient	< 0.03°C over 6 inches during cell maintenance
Fault Protection	Firmware protection
Communications	RS-232 included
Ambient Temperature	18°C to 28°C (65°F to 82°F)
Power Requirements	115 VAC (±10%), 60 Hz, 1.0 A or 230 VAC (±10%), 50 Hz, 0.65 A, 175 W maximum
Exterior Dimensions	8.75" W x 10.25" D x 19.25" H (22 x 26 x 49 cm)
Weight	18 lb (8.2 kg) without cell

3.2 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: 18–28°C (65–82°F)
- ambient relative humidity: 15-50%
- pressure: 75kPa – 106kPa
- mains voltage within ±10% of nominal
- vibrations in the calibration environment should be minimized
- altitudes less than 2,000 meters

3.3 Warranty

Hart Scientific, Inc. (Hart) warrants this product to be free from defects in material and workmanship under normal use and service for a period as stated in the current product catalog from the date of shipment. This warranty extends only to the original purchaser and shall not apply to any product which, in Hart's sole opinion, has been subject to misuse, alteration, abuse or abnormal conditions of operation or handling.

Software is warranted to operate in accordance with its programmed instructions on appropriate Hart products. It is not warranted to be error free.

Hart's obligation under this warranty is limited to repair or replacement of a product which is returned to Hart within the warranty period and is determined, upon examination by Hart, to be defective. If Hart determines that the defect or malfunction has been caused by misuse, alteration, abuse or abnormal conditions or operation or handling, Hart will repair the product and bill the purchaser for the reasonable cost of repair.

To exercise this warranty, the purchaser must forward the product after calling or writing a Hart Scientific Authorized Service Center for authorization. Hart assumes NO risk for in-transit damage.

For service or assistance, please contact a Hart Scientific Authorized Service Center (see Section 1.3).

THE FOREGOING WARRANTY IS PURCHASER'S SOLE AND EXCLUSIVE REMEDY AND IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO ANY IMPLIED WARRANTY OR MERCHANTABILITY, OR FITNESS FOR ANY PARTICULAR PURPOSE OR USE. HART SHALL NOT BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL, OR CONSEQUENTIAL DAMAGES OR LOSS WHETHER IN CONTRACT, TORT, OR OTHERWISE.

4 Quick Start in Auto Program Mode

The user should read this user's guide and be familiar with the functional performance of the Model 9230 before using the instrument to achieve a gallium melt plateau.

4.1 Unpacking

Unpack the instrument carefully and inspect it for any damage that may have occurred during shipment. If there is shipping damage, notify the carrier immediately. Verify that the following components are present:

- Model 9230 Gallium Maintenance Apparatus
- Melt heater
- Power cord
- User's Guide
- Top Insulation Plugs
- Cell Removal Tool

If you are missing any item, please call a Hart Scientific Authorized Service Center.

4.2 Set Up

Place the instrument on a flat surface with at least 6 inches of free space around and 18 inches above the instrument. Avoid drafty areas. Install the power cord into the power entry module on the back of the instrument. Plug the power cord into a grounded mains outlet. Verify that the nominal voltage corresponds to that indicated on the back of the instrument.

Using the cell removal tool, carefully insert the frozen gallium cell, prepared as described in Section 9, Melting Point of Gallium Realization, into the well. (DO NOT drop it into the well.) The well must be clear of any foreign objects, dirt and grit before the cell is inserted.

Firmly place the two top insulation plugs into the well such that they rest on top of the cell. Make sure that the inner melt heater is connected to the inner melt heater connector on the back of the instrument and the melt heater switch is in the *OFF* position. Carefully insert the melt heater through the hole in the top insulation plugs and into the entrant well of the frozen gallium cell. Make sure the inner melt heater extends to the bottom of the gallium cell entrant well.

4.3 Power

Plug the instrument power cord into a mains outlet of the proper voltage, frequency and current capability. Refer to Section 3.1, Specifications, for power

details. Turn the instrument ON using the power switch on the back of the unit. The instrument turns on and begins to approach 25°C. The front panel LED display shows $\mu\epsilon\sigma$.

4.4 Auto Program

The Model 9230 is designed to automate the gallium cell melt plateau. This is accomplished through the use of the Auto Program; a microprocessor-controlled program that determines set temperatures, scan rates, duration times, Peltier module configurations, and melt heater controls. By controlling all of these parameters in the proper order, a temperature profile can be maintained in the block assembly that assures a uniform melt, maintains the melt over long periods of time, properly refreezes the cell and keeps the cell ready for the next melt cycle. The Auto Program is illustrated in Figure 1.

4.4.1 Starting the Auto Program

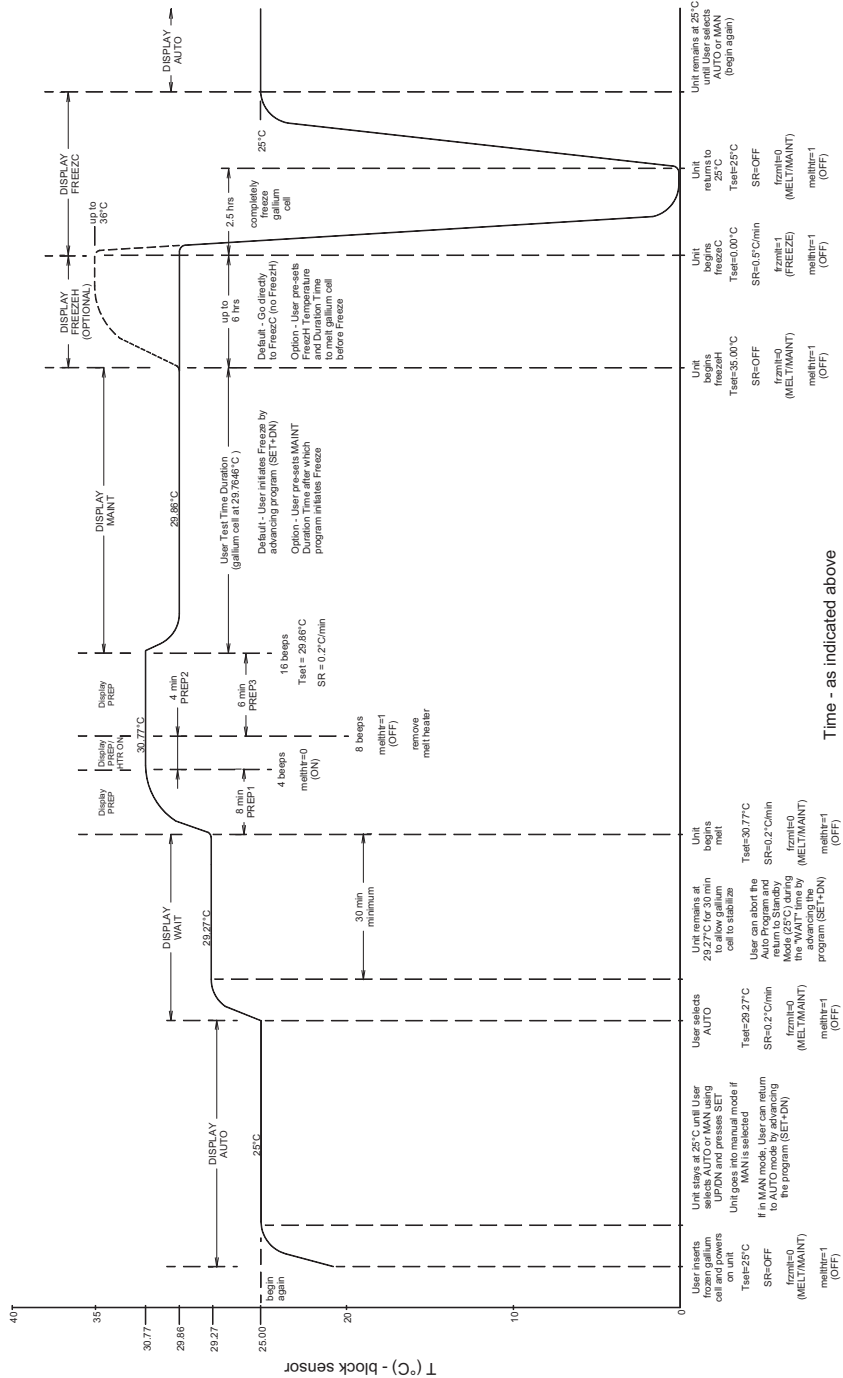
After the frozen gallium cell is properly installed in the instrument (See Section 9, Melting Point of Gallium Realization, for more details) and the unit is powered ON, the front panel LED display shows $\mu\epsilon\sigma$. The instrument goes to 25°C and remains there until the SET button on the front panel is pressed which starts the Auto Program. The display changes to $\nu\mu\mu\epsilon$ and the Auto Program begins. The unit resets to 29.27°C at a scan rate of 0.2°C/minute in accordance with the Auto Program (see Figure 1).

4.4.2 Generating the Melt Plateau

Once the unit has stabilized at 29.27°C, it waits for a minimum of 30 minutes to ensure that the gallium cell has equalized in temperature. At the end of the equalization time, the Auto Program begins to initiate the melt. The display changes to $PREP$ as the set temperature of the block is changed to 30.77°C. This initiates a melt on the outer surfaces of the gallium within the cell. A few minutes into the program, the unit beeps four times and the display changes to a message that flashes between $PREP$ and $HEATER ON$ to indicate that the inner melt heater has been turned ON. The inner melt heater remains ON for a few minutes and then shuts off as the display changes back to $PREP$ and the unit beeps eight times. The user should remove the inner melt heater at this time. **Note:** No damage occurs to the gallium cell if the heater is not removed, however, a shortening of the melt duration may result.

After the inner melt heater is powered OFF, the instrument remains at 30.77°C for a few more minutes to complete the outer melt of the gallium cell. The unit then beeps 16 times, the display changes from $PREP$ to $INNER M$ and the block temperature resets to 29.86°C to maintain the melting gallium cell.

The user may then insert a preheated monitor thermometer to monitor the temperature of the gallium cell. Due to the heat applied to the cell by the inner melt heater, the temperature of the cell's entrant well is higher than the gallium melting point (29.7646°C) for a short period of time. Generally, after 30 to 60 minutes, the temperature of the cell's entrant well is sufficiently stable to begin



Time - as indicated above

Figure 1 Auto Program Diagram

calibrations of thermometer sensors. The duration of the melt plateau varies depending on usage, but lengths of 5 days or longer are achievable.

4.4.3 Refreezing the Gallium Cell

At the end of the melt plateau (or when the user wishes to end the plateau), the Auto Program is advanced by pressing the SET and DOWN buttons (press the SET button and keep it pressed and then press the DOWN button and release both) on the front panel (program advance). The display will flash “*ADU*” and display “*nnnnn*.” Use the UP and DOWN buttons to select the program state “*FREEZE*” then press the ENTER button. See Section 6.21.2, Maintain Time-out, for optional user defined auto time-out. Gallium, like water, expands in volume when freezing. Therefore, the cell must be frozen from the bottom up or there is a potential for damage to the cell. The Auto Program process ensures that the cell is properly refrozen.

The Auto Program initiates the freeze and the display shows *FREEZE* at this point. The Peltier devices are reconfigured and controlled such that a large temperature gradient is generated between the top and bottom of the block. The instrument slowly scans to 0°C to maintain the gradient over several hours. See Section 6.21.4, Freeze Hot Temperature, for optional user defined freeze hot temperature.

4.4.4 Return to Standby

Upon completion of the freeze, the Auto Program returns the unit to 25°C, the front panel LED display shows *Sto* and the unit remains there until the user initiates the Auto Program again by pressing the SET button.

5 Parts and Controls

The user should become familiar with the Model 9230 parts. Successful use of the instrument is dependent upon knowledge of important components and their proper use.

5.1 Bottom Panel

On the bottom panel are the fan and feet. See Figure 2.

1. The cooling fan inlet is at the bottom of the unit. The cooling air circulating through the instrument keeps the electronics and the chassis cool. Keep the area immediately around the instrument clear to allow adequate ventilation.
2. Three feet support the chassis permitting air space for the fan.

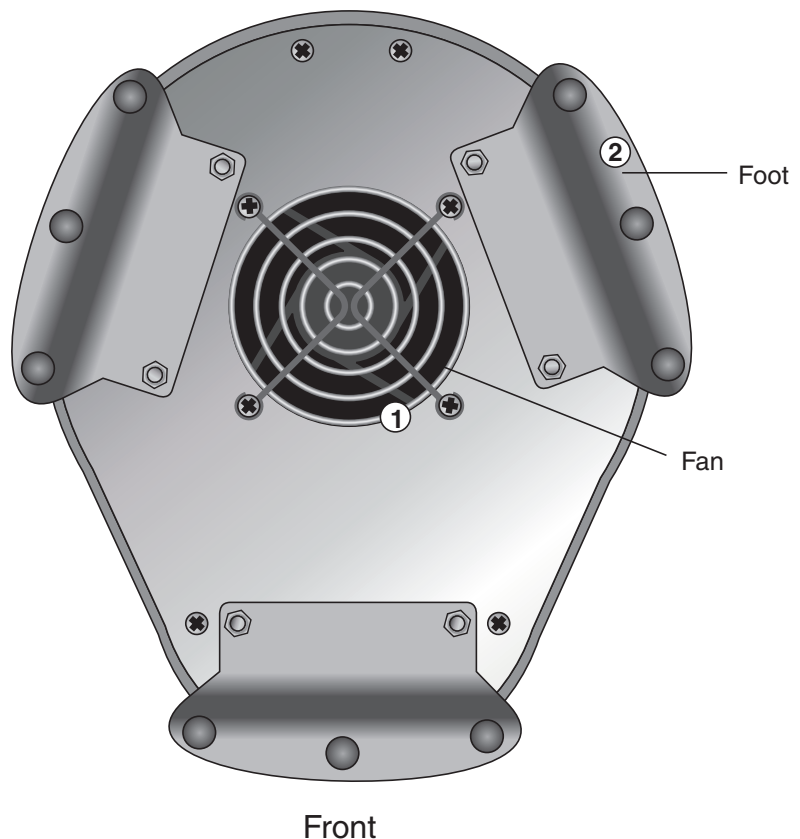


Figure 2 Bottom Panel

5.2 Front Panel

The front panel contains the digital display and the controller keypad. See Figure 3.

1. The digital display is an important part of the temperature controller because it not only displays set and actual temperatures but also displays various instrument functions, settings, and constants. The display shows temperatures in units according to the selected scale °C or °F. When in the Auto Program mode, the display indicates the current status of the unit in the gallium melt process.

2. The four button controller keypad allows easy setting of the set-point temperature. The control buttons (SET, DOWN, UP, and EXIT) are used to set the temperature set-point, access and set other operating parameters, and access and set calibration parameters.

Setting the control temperature is done directly in degrees of the current scale and can be set to 0.01 of a degree Celsius or Fahrenheit.

During the Manual mode of operation, the functions of the buttons are as follows:

SET – Used to display the next parameter in the 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 a function and to skip to the next function. Any changes made to the displayed value are ignored. Holding the EXIT button for about ½ second returns control to the main display.

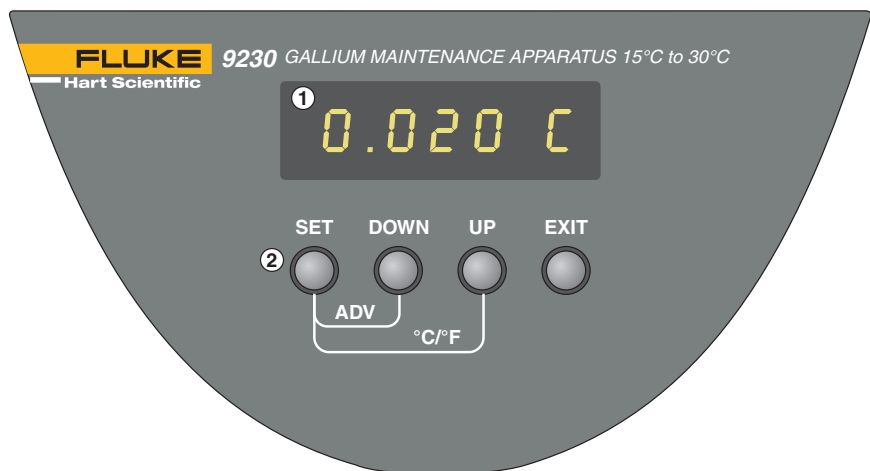


Figure 3 Front Panel

5.3 Top Panel

The primary feature of the top of the unit is to provide access to the temperature controlled block. The top panel consists of the constant temperature block assembly; two preheat wells, top insulation plug, cooling air vents, the melt heater and melt heater storage well. See Figure 4.

1. The constant temperature block assembly is where the gallium cell is inserted. Peltier devices surround the cell in order to provide a uniform temperature. These devices can be configured by the controller to provide a large vertical gradient during the freezing of the gallium cell, or a very uniform temperature when maintaining a melting cell.

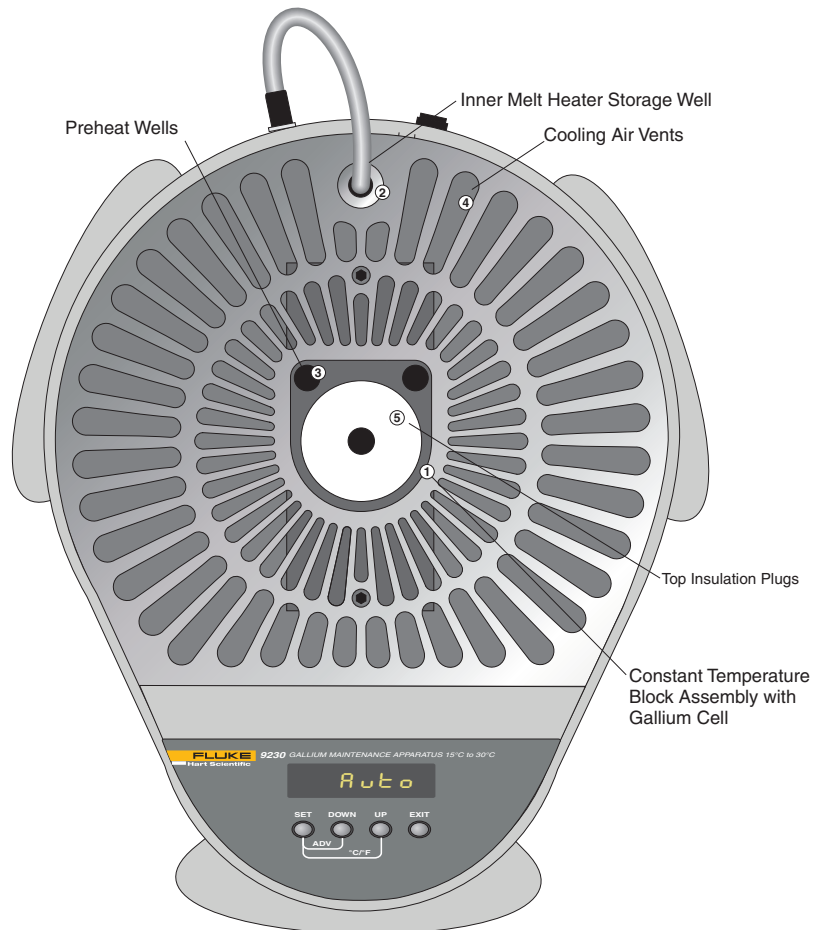


Figure 4 Top Panel

2. The inner melt heater is a low-power heater that creates an inner liquid layer next to the reentrant tube of the gallium cell during the melting process. This inner melt heater is controlled automatically by the microprocessor when using the Auto Program mode, or may be used manually with the switch in back. When not in use, the inner melt heater is stored in the well at the back of the unit to prevent it from causing any damage.
3. The thermometer preheat wells are located at the back of the block access well. Thermometers are preheated in these wells prior to insertion into the cell in order to conserve its latent energy.
4. The cooling air vents in the top of the unit permit heated air to exit the unit.
5. The top insulation plugs fit into the well over the gallium cell. See Section 8.5, Inserting the Top Insulation Plugs.

5.4 Rear Panel

The rear panel consists of the power connection, power switch, melt heater switch, inner melt heater connection, and serial port. See Figure 5.

1. The power connection is where the power cord plugs into the back of the instrument.
2. Set the power switch “ON” to operate the instrument and “OFF” to disconnect the power.
3. The melt heater plugs into the rear of the instrument into the inner melt heater connector provided. Be sure it is plugged in during operation.
4. The melt heater switch can be set to MANUAL ON position or AUTO position. The instrument’s microprocessor has control when the switch is in the AUTO position.



Warning: *When the melt heater switch is in the MANUAL ON position, the melt heater is continuously energized and can get hot!*

5. The serial port is a DB-9 male connector for interfacing the instrument to a computer or terminal RS-232 communications.

5.5 Thermal Block Assembly

The thermal block assembly holds the gallium cell and heats or cools to maintain the proper temperature. The thermal block assembly consists of the thermal block, thermoelectric Peltier devices, the gallium cell, and the top insulation plugs.

1. The thermal block is specifically designed to contain the gallium cell. The thermal block also has 2 preheat wells for the test thermometers.

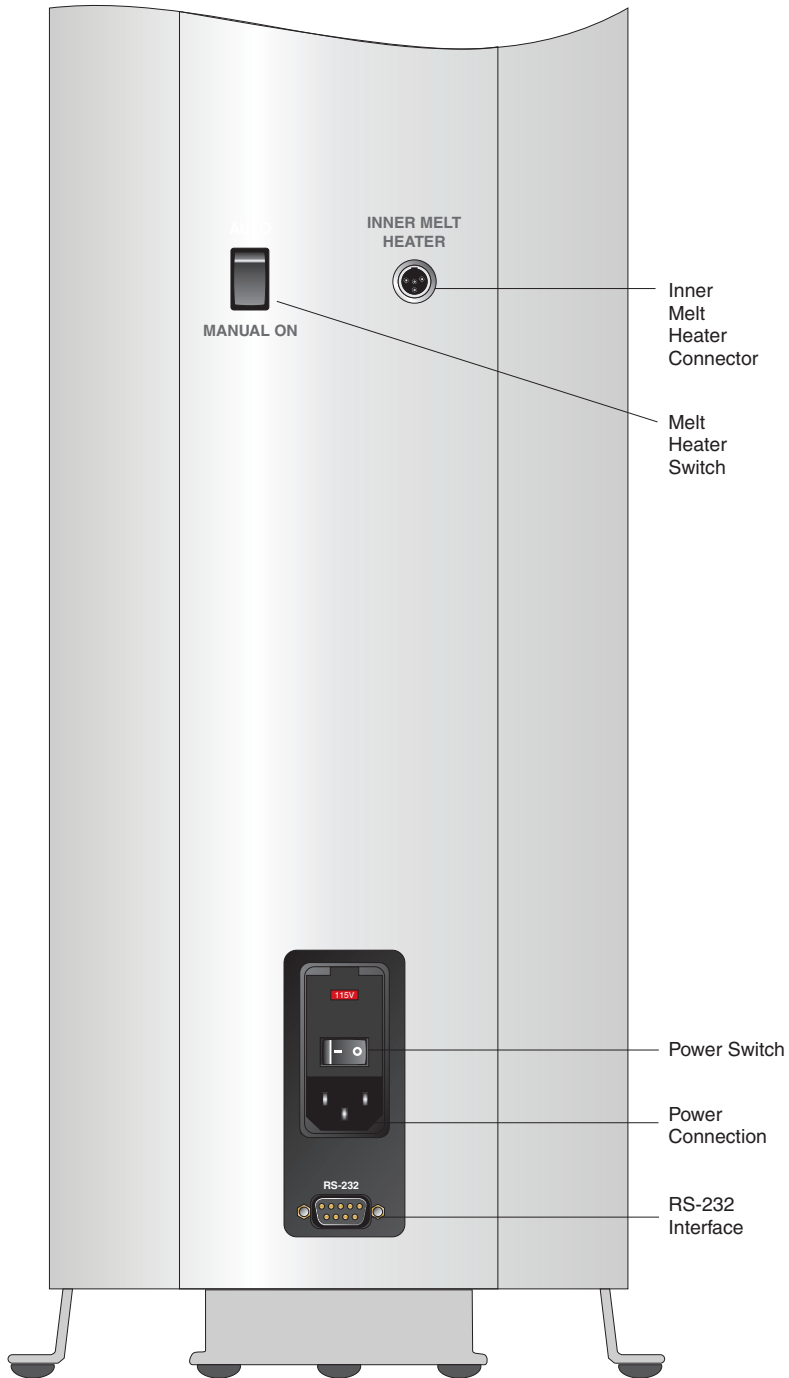


Figure 5 Rear Panel

2. The block assembly is heated or cooled as necessary to maintain the proper temperature with thermoelectric Peltier devices.
3. The gallium cell is placed into the block. Use the removal tool to carefully insert or remove the cell.



WARNING: *DO NOT use the removal tool to carry the cell.*



4. The top insulation plugs are placed over the gallium cell to help maintain the proper temperature. See Section 8.5, Inserting the Top Insulation Plugs.

6 Controller Operation

This section discusses in detail how to operate the instrument temperature controller using the front control panel. By using the front panel key-switches and LED display, the user may initiate the Auto Program mode; a microprocessor-controlled program that determines set temperatures, scan rates, duration times, Peltier module configurations, and melt heater controls. By controlling all of these parameters in the proper order, a temperature profile can be maintained in the block assembly that assures a uniform melt, maintains the melt over long periods of time, properly refreezes the cell and keeps the cell ready for the next melt cycle. After selecting the Manual mode of operation, the user may monitor the well temperature, adjust the set-point temperature in degrees C or F, monitor the Peltier device output power, adjust the controller proportional band, and program the operating parameters, program parameters, serial interface configuration, and the controller calibration parameters. Operation of the functions and parameters is shown in the flowchart in Figure 6 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.

When two buttons are shown to initiate an action, for example,

 +  *Press and hold the first button (e.g., SET) and then press the second button (e.g., DOWN) and release both*

6.1 Standby

After the instrument is powered ON or at the end of a gallium cell refreeze process, the unit automatically is set to 25°C and the digital LED display reads:

 *Ready to Start Auto Program*

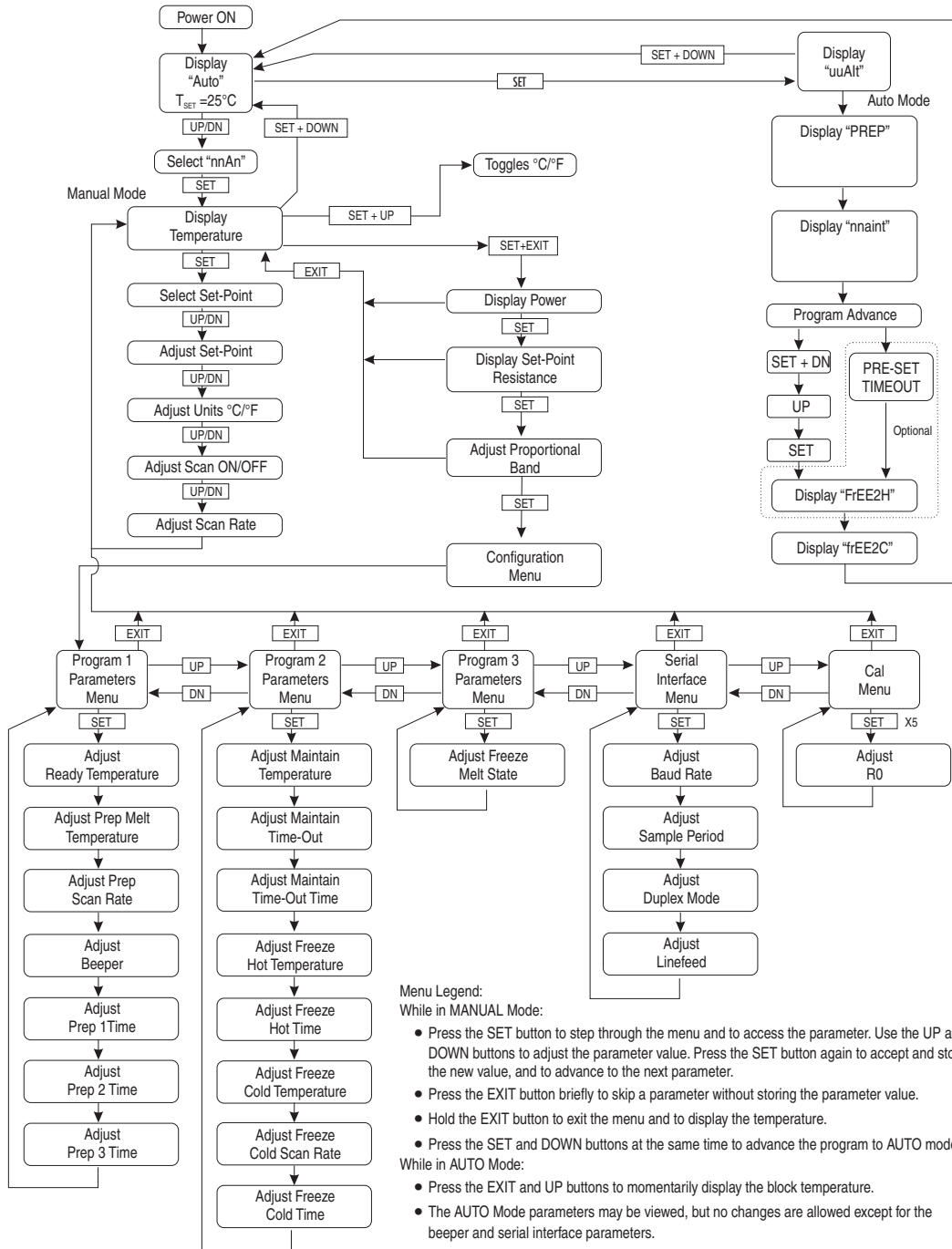
The unit is in a Standby condition and remains at 25°C indefinitely until the user initiates a change by pressing a button sequence.

6.2 Starting the Auto Program

The user begins the Auto Program by pressing the SET button.

 *Start the Auto Program*

 *WAIT - Auto Program started*



(see Figure

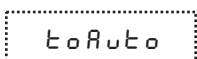
(see Figure

Figure 6 Controller Flowchart


At this point the Auto Program takes control of the instrument and initiates a melt in the gallium cell in accordance with Figure 1 on page 13 and Figure 6 on page 22.

At any time the user may press the program advance button sequence to access the program advance mode.

 +  *Access program advance*

 *Exit from auto mode*


From here, the set button can be pressed to go back to the Auto state or press the up or down buttons to choose a different program state. Once the desired program state is chosen press SET to accept the chosen state.

 *Accept the program state*

The chosen program state will now start.

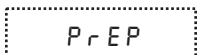
6.3 Auto Program – Initiating and Maintaining the Gallium Melt Plateau

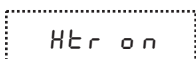
Approximately 55-65 minutes after starting the Auto Program, the gallium cell reaches 29.27°C and stabilizes enough to start the melt plateau. The display shows:

 *PrEP – gallium cell melt initiated*

The Auto Program takes the block assembly to 30.77°C, which begins melting the outer surface of the gallium inside the cell.

After approximately 8 minutes, the Auto Program energizes the inner melt heater inside the gallium cell entrant well. The instrument sounds an audible beep 4 times and the display message alternates as indicated:

 *Alternating message between PrEP and Htr on*



The inner melt heater is energized for approximately four minutes after which the Auto Program turns it OFF and sounds an audible beep eight times. At this point the display indicates:

 *Inner melt heater OFF*

The user should remove the inner melt heater at this time. **Note:** No damage will occur to the gallium cell if the heater is not removed, however, a shorten-

ing of the melt plateau may result. The inner melt heater may be stored in the melt heater well at the back of the unit.

After the inner melt heater has been powered OFF, the instrument remains at 30.77°C for a few more minutes to complete the outer melt of the gallium cell. The unit then sounds an audible beep 16 times, the display changes from *P R E P* to *n n R i n t* and the block temperature is reset to 29.86°C to maintain the melting gallium cell.

n n R i n t *Maintain – instrument maintaining melt plateau*

The user may then insert a preheated monitor thermometer to monitor the temperature of the gallium cell. Due to the heat applied to the cell by the inner melt heater, the temperature of the cell's entrant well is higher than the gallium melting point (29.7646°C) for a short period of time. Generally, after 30 to 60 minutes, the temperature of the cell's entrant well is sufficiently stable to begin calibrations of thermometer sensors. The duration of the melt plateau varies depending on usage, but lengths of 5 days or longer are achievable.

6.4 **Auto Program – Ending the Gallium Melt Plateau and Refreezing the Cell**


At the end of the melt plateau (or when the user wishes to end the plateau), the Auto Program is advanced by pressing the program advance button sequence.


 +  *Access program advance*

Display will flash advance for 1 second then show the current program state.

A d U *Program advance*

n n R i n t *Maintain - instrument maintaining melt plateau*

 *Advances to next program state*

 *Accepts new program state*

F r E E Z C *Start freezing the gallium cell*

Gallium, like water, expands in volume when freezing. Therefore, the cell must be frozen from the bottom up or there is a potential for damage to the cell.

The Peltier devices are reconfigured and controlled such that a large temperature gradient is generated between the top and bottom of the block. The instrument slowly scans from 29.86°C to 0°C to maintain the gradient over several hours thus ensuring the cell freezes properly, beginning at the bottom and continuing up to the top of the cell.

See Section 6.21.2, Maintain Time-out, for optional user defined auto time-out (an optional method of ending the melt plateau and initiating the refreeze process). See Section 6.21.4, Freeze Hot Temperature, for optional user defined freeze hot temperature (a method to totally melt the cell before freezing it).

6.5 Return to Standby

Upon completion of the freeze, the Auto Program returns the unit to 25°C, the digital LED display shows:


 *Instrument returning to 25°C*

After reaching 25°C, the unit remains there until the user initiates the Auto Program again by pressing the SET button or goes into the Manual mode of operation.

6.6 Program Advance


From the manual mode, the program advance function allows the user to return to the standby condition.

 +  *Access program advance*

 *“ADU” flashes and then displays program mode*

 *Program mode*

Press the UP or DOWN button to toggle between the Auto Program and Manual mode.


 *Accepts the new operation*

At any time during the auto program the user may press the program advance button sequence to access the program advance function.

 +  *Access program advance*

The current program state will be displayed.

 *Return to main menu*

 *Initiate cell melt with melt heater*



 *Maintain melt plateau*

 *Initiate total melt*

or

 *Start freezing the cell*

From the current program state, the up or down buttons can be pressed to choose a different program state.

 or  *Choose the desired program state*

Once the desired program state is chosen, press “SET” to accept and start that program.

 *Accept new program state and start the program*

To get to the wait state the user must select the Auto state then press “set” to start the program in the wait state.

6.7 Manual Mode


6.8 Starting the Manual Mode

After the instrument is powered ON or at the end of a gallium cell refreeze process, the unit automatically is set to 25°C and the digital LED display reads:


 *Ready to start Auto Program*

The unit is in a Standby condition and remains at 25°C indefinitely until the user initiates a change by pressing a button sequence. To begin the Manual mode of operation, the user presses the UP or DOWN buttons to change the display reading as follows:

 or  *Toggle display between Auto and Manual*

 *Ready to go into Manual mode*

By pressing the SET button while this is displayed, the instrument goes into the Manual mode and begins to display the well temperature:

 *Accept Manual mode*

 *Well temperature in degrees Celsius*

Once in the Manual mode, the temperature display function may be accessed from any other function by pressing and holding the EXIT button a few seconds.

6.9 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 temperature limit of any device inserted into the well.


Setting the temperature involves selecting one of the eight (8) set-points in memory and then adjusting the set-point value.

6.9.1 Programmable Set-points

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


To set the temperature, first select the set-point memory. This function is accessed from the temperature display function by pressing the SET button. 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.

 Well temperature in degrees Celsius

 Access set-point memory

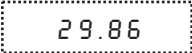
 Set-point memory 1, 25.0°C currently used

To change the set-point memory to another preset value press the UP or DOWN button.

 New set-point memory 4, 30.0°C

Press the SET button to display the new selection and access the set-point value.

 Display selected set-point memory

 New set-point value 29.86°C

6.9.2 Set-point Value

If the set-point value is correct, hold the EXIT button to accept the new value and resume displaying the well temperature. Otherwise, press the UP or DOWN button to adjust the set-point value.

 *New set-point value*


When the desired set-point value is reached press the SET button to accept the new value and to access the temperature scale units. If the EXIT button is pressed, any changes made to the set-point are ignored.

 *Accept new set-point value*

The default programmable set-points (factory set) are given in Table 2.

Table 2 Default Programmable Set-points (Factory Set)

Set-point Memory Number	Default Set-point Temperature (°C)
1	25.00
2	29.27
3	30.77
4	29.86
5	35.00
6	00.00
7	10.00
8	20.00

 **NOTE:** Setpoint 1 will always be set back to 25.00°C after the power has been cycled off then on.

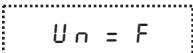
6.10 Temperature Scale Units

The temperature scale units of the controller can be set by the user to degrees Celsius (°C) or Fahrenheit (°F). The units are used in displaying the well temperature, set-point, scan rate, and proportional band.

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

 *Scale units currently selected*

Press the UP or DOWN button to change the units.

 *New units selected*

The units can also be changed quickly from the main display by pressing the SET and UP buttons.

6.11 Scan

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

6.11.1 Scan Control

The scan is controlled with the scan ON/OFF function that appears in the main menu after the set-point and unit's functions.

 *Scan function OFF*

Press the UP or DOWN button to toggle the scan ON or OFF.

 *Scan function ON*

Press the SET button to accept the present setting and continue.

 *Accept scan setting*

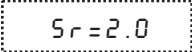
6.12 Scan Rate

The next function in the main menu is the scan rate. The scan rate can be set from 0.1 to 5.0°C/minute (0.2 to 9.0°F/min). The maximum scan rate, however, is actually limited by the natural heating or cooling rate of the instrument. This is typically less than 2°C/minute.

The scan rate function appears in the main menu after the scan control function. The scan rate units are in degrees per minute, degrees C or F depending on the selected units.

 *Scan rate in °C/min*

Press the UP or DOWN button to change the scan rate.

 *New scan rate*

Press the SET button to accept the new scan rate and continue.

 *Accept scan rate*

The display returns to the current well temperature.

6.13 Temperature Scale Units

To toggle between °C and °F, press and hold the SET button and then press the UP button when the temperature is displayed.

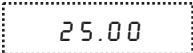


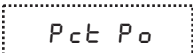
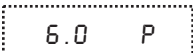
6.14 Secondary Menu

Functions used less often are accessed within the secondary menu. Press and hold the SET and EXIT buttons to access the secondary menu. The first function in the secondary menu is the power display. (See Figure 6 on page 22.)

6.15 Peltier Device Power

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

The device power display is accessed in the secondary menu. Press and hold the SET and EXIT buttons. The Peltier device power is displayed as a percentage of full power.

	<i>Well temperature</i>
	<i>Access Peltier device power in secondary menu</i>
	<i>Flashes and then percent power flashes</i>
	<i>Flashes prior to displaying Peltier device power</i>
	<i>Peltier device power in percent</i>

To exit out of the secondary menu press the EXIT button and hold for a brief moment. To continue on to the proportional band setting function, press the EXIT button momentarily or the SET button.


6.16 Set-point Resistance

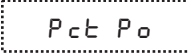
The set-point resistance is the resistance associated with the set-point temperature. Allow the temperature to stabilize at the desired set-point before using the

displayed resistance value. The set-point resistance function is available for diagnostic purposes.


Press the SET and EXIT buttons to enter the secondary menu and show the heater power. Then press the SET button twice to access the set-point resistance.

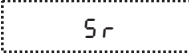
 +  *Access Peltier device power in secondary menu*

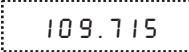
 *Flashes and then percent power flashes*

 *Flashes prior to displaying Peltier device power*

 *Peltier device power in percent*

 *Access set-point resistance*

 *Flashes "5r" (Set-point Resistance) and then displays the setting*

 *Resistance in ohms*

6.17 Proportional Band


In a proportional controller such as this, the Peltier device output power is proportional to the well temperature over a limited range of temperatures around the set-point. This range of temperature is called the proportional band. At the bottom of the proportional band, the Peltier device output is $\pm 100\%$. At the top of the proportional band, the Peltier device output is 0. Thus as the temperature rises the Peltier device power is reduced, which consequently tends to lower the temperature back down. In this way the temperature is maintained at a fairly constant value.

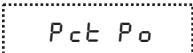
The temperature stability of the well and the response time depend on the width of the proportional band. If the band is too wide, the well temperature deviates excessively from the set-point due to varying external conditions. This deviation is because the power output changes very little with temperature and the controller does not respond 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 8.0°C . The proportional band width may be altered by the user 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 the SET and EXIT buttons to enter the secondary menu and show the device power. Then press the SET button twice to access the proportional band.

 +  *Access Peltier device power in secondary menu*

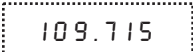
 *Flashes "SEC" and then percent power flashes*


 *Flashes prior to displaying Peltier device power*

 *Peltier device power in percent*

 *Access set-point resistance*

 *Flashes "5r" (Set-point Resistance) and then displays the setting*

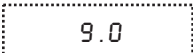
 *Resistance in ohms*

 *Access proportional band*


 *Flashes "Pr o P" and then displays the setting*

 *Proportional band setting*

To change the proportional band press the UP or DOWN button.

 *New proportional band setting*

To store the new setting press the SET button. Press the EXIT button to continue without storing the new value.

 *Accept the new proportional band setting*

6.18 Controller Configuration, Operating, and Calibration Parameters

The controller has a number of configuration, operating, and calibration parameters that are programmable via the front panel. The first set of parameters is associated with the Auto Program and is located under the configuration submenus called *Pr o G 1*, *Pr o G 2*, and *Pr o G 3*. The next set of parameters is

associated with the serial RS-232 interface and is located under the configuration submenu called *SERIAL*. The last set of parameters is associated with the calibration of the instrument and is located under the configuration submenu called *CAL*.

6.19 Auto Program Configuration Parameters



CAUTION: *The Auto Program configuration parameters should not be adjusted under normal use. These parameters directly affect the melt plateau duration time and proper freezing of the gallium cell.*

The first set of parameters is associated with the Auto Program (see Figure 1, page 13). The Auto Program is a microprocessor-controlled program that determines set temperatures, scan rates, duration times, Peltier module configurations and melt heater controls. By controlling all of these parameters in the proper order, a temperature profile can be maintained in the block assembly that assures a uniform melt, maintains the melt over long periods of time, properly refreezes the cell and keeps the cell ready for the next melt cycle. The de-

fault programmable set-points (factory set) are given in Table 3, along with minimum and maximum set values.

Table 3 Auto Program Parameters (Prog 1, Prog 2, Prog 3)

Parameter Name	Parameter	Low Value	Default Value*	High Value	Units	Prog	Set Resolution
Ready Temperature (WAIT)	<i>r d u t</i>	29.000	29.270	29.300	°C	1	xx.xxx
Prep Melt Temperature (PREP)	<i>P r E P t</i>	30.000	30.770	35.000	°C	1	xx.xxx
Prep Scan Rate (PREP)	<i>P r E P S r</i>	0.1	0.2	0.5	°C/min	1	x.x
Beeper (PREP)	<i>b E E P E r</i>	-	ON	-	-	1	ON/OFF
Prep 1 Time (PREP)	<i>d u r 1</i>	360	480	600	sec	1	xxx
Prep 2 Time (PREP)	<i>d u r 2</i>	120	240	360	sec	1	xxx
Prep 3 Time (PREP)	<i>d u r 3</i>	240	360	480	sec	1	xxx
Maintain Temperature (MAINT)	<i>n n a i n t</i>	29.790	29.860	35.000	°C	2	xx.xxx
Maintain Time-out (MAINT)	<i>t i o u t</i>	-	OFF	-	-	2	ON/OFF
Maintain Time-out Time (MAINT)	<i>d u r 4</i>	1	7200	43,200	min	2	xx,xxx
Freeze Hot Temperature (FREEZH)	<i>F r E E Z H</i>	29.860	29.860	36.000	°C	2	xx.xxx
Freeze Hot Time (FREEZH)	<i>d u r 5</i>	0	0	360	min	2	xxx
Freeze Cold Temperature (FREEZC)	<i>F r E E Z C</i>	-1.000	0.000	10.000	°C	2	xx.xxx
Freeze Cold Scan Rate (FREEZC)	<i>F r E E Z r</i>	0.4	0.5	0.6	°C/min	2	x.x
Freeze Cold Time (FREEZC)	<i>d u r 6</i>	120	150	180	min	2	xxx
Freeze Melt State (Manual Mode Only)	<i>F r n E L t</i>	-	MELT	-	-	3	MELT/ FREEZE

*Factory Set

6.20 Prog 1

The parameters within the Prog 1 submenu are accessed from the secondary menu after the proportional band function by pressing the SET button. *C o n F I G* flashes and then the name of the first Auto Program parameter menu *P r o G 1* is displayed:

8 . 0 Proportional band setting

SET Select the configuration menus

C o n F I G Flashes before entering configuration menus

Prog 1

Prog 1 submenu

Pressing the SET button accesses the parameters within the Prog 1 submenu. Pressing the UP button accesses Prog 2. The well temperature is displayed by pressing the EXIT button.

6.20.1 Ready Temperature

The Ready Temperature parameter is the temperature at which the instrument stabilizes before initiating a melt in the gallium cell. The default value is 29.27°C and can be seen on Figure 1 as the temperature that the well goes to when the display shows *rdut*. The instrument holds the well at this temperature for 30 minutes minimum to assure that the gallium cell has stabilized, before allowing the melt to proceed.

When Prog 1 is displayed, press the SET button to access the Ready Temperature parameter. The display flashes *rdut* and then displays the current Ready Temperature value:

Prog 1

Prog 1 submenu



Access the ready temperature parameter

rdut

Flashes before entering ready temperature

29.270

Current ready temperature

Use the UP or DOWN buttons to adjust the value.

29.300

New ready temperature

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.



Accept new ready temperature value and continue to Prep Melt Temperature

6.20.2 Prep Melt Temperature

The Prep Melt Temperature parameter is the temperature at which the instrument begins to melt the outer surfaces of the gallium within the gallium cell. The default value is 30.77°C and can be seen on Figure 1 as the temperature that the well goes to when the display shows *PREP*. The instrument holds the well at this temperature for approximately 18 minutes (the sum of Prep 1, Prep 2 and Prep 3 duration times) to begin melting the outer surfaces of the gallium.

After accessing the Ready Temperature parameter, the display flashes *P R E P t* and displays the current value of the Prep Melt Temperature.

P R E P t *Flashes before entering prep melt temperature parameter*

30.770 *Current prep melt temperature*

Use UP or DOWN buttons to adjust the value.

35.000 *New prep melt temperature*

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.

 *Accept new Prep Melt Temperature value and continue to the Prep Scan Rate*

6.20.3 **Prep Scan Rate**

The Prep Scan Rate parameter is the rate in °C/minute or °F/minute at which the instrument scans from the Ready Temperature to the Prep Melt Temperature. The default value is 0.2°C/minute and can be seen on Figure 1, where the unit begins the melt.

After accessing the Prep Melt Temperature parameter, the display flashes *P R E P S r* and displays the current value of the Prep Scan Rate.


P R E P S r *Flashes before entering prep scan rate parameter*

S r = 0.2 *Current prep scan rate*

Use the UP or DOWN buttons to adjust the value.

S r = 0.5 *New prep scan rate*

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.

 *Accept new prep scan rate value and continue to the inner melt heater Beeper*

6.20.4 **Beeper**

The Beeper parameter is used to enable or disable the audible warnings that may be generated during the initiation of the melt. The default value is ON.

As seen on Figure 1, the Auto Program sounds an audible beep 4 times when it energizes the inner melt heater and the display shows an alternating message from *PREP* to *HEAT ON*. After approximately 4 minutes, the Auto Program sounds an audible beep 8 times and turns the inner melt heater OFF (the display returns *PREP*). After approximately 6 more minutes, the Auto Program sounds an audible beep 16 times and displays *ONLINE*, an indication that the gallium melt has begun.

Changing the Beeper parameter to OFF disables the beeper during this phase of the Auto Program.

After accessing the Prep Scan Rate parameter, the display flashes *BEEPER* and displays the current value of the Beeper parameter.

BEEPER *Flashes before entering beeper parameter*

On *Current beeper setting (enabled)*

Use the UP or DOWN buttons to adjust the value.

OFF *New beeper setting*

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.

 *Accept new beeper setting and continue to the prep 1 time*

6.20.5 Prep 1 Time

After beginning the gallium melt, see Figure 1, the Auto Program waits a short time before energizing the inner melt heater. The Prep 1 Time parameter specifies this time in seconds. The default value is 480 seconds (8 minutes). At the end of this time, the Auto Program energizes the inner melt heater and sounds 4 audible beeps.

After accessing the Beeper parameter, the display flashes *dur 1* and displays the current value of the Prep 1 Time parameter.

dur 1 *Flashes before entering the prep 1 time parameter*

SEC480 *Current prep 1 time value (8 minutes)*

Use the UP or DOWN buttons to adjust the value.

SEC600 *New prep 1 time value (10 minutes)*

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.

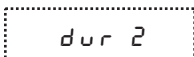


Accept new prep 1 time value and continue to prep 2 time

6.20.6 Prep 2 Time

After energizing the inner melt heater during the initialization of the gallium melt, see Figure 1, the Auto Program waits a short time before turning OFF the inner melt heater. The Prep 2 Time parameter specifies this time in seconds. The default value is 240 seconds (4 minutes). At the end of this time, the Auto Program turns the inner melt heater OFF and sounds 8 audible beeps.

After accessing the Prep 1 Time parameter, the display flashes `dur 2` and displays the current value of the Prep 2 Time parameter.



Flashes before entering the prep 2 time parameter



Current prep 2 time value (4 minutes)

Use the UP or DOWN buttons to adjust the value.



New prep 2 time value (6 minutes)

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.

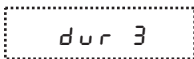


Accept new prep 2 time value and continue to prep 3 time

6.20.7 Prep 3 Time

After turning OFF the inner melt heater during the initialization of the gallium melt, see Figure 1, the Auto Program waits a short time before beginning the maintenance process of the gallium melt plateau. During this time the instrument remains at 30.77°C to complete the melting of the outer surfaces of the gallium in the cell. The Prep 3 Time parameter specifies this time in seconds. The default value is 360 seconds (6 minutes). At the end of this time, the Auto Program begins the maintenance process and sounds 16 audible beeps.

After accessing the Prep 2 Time parameter, the display flashes `dur 3` and displays the current value of the Prep 3 Time parameter.

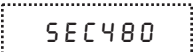


Flashes before entering the prep 3 time parameter




Current prep 3 time value (6 minutes)

Use the UP or DOWN buttons to adjust the value.

 *New prep 3 time value (8 minutes)*

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and to return to the Prog 1 submenu.

 *Accept new prep 3 time value and return to the prog 1 submenu*


 *Prog 1 submenu*

Press the UP button to access Prog 2. The well temperature is displayed by pressing the EXIT button. Press the SET button to access the parameters within the Prog 1 submenu.

6.21 Prog 2

Prog 2 is displayed by pressing the UP button while the display shows Prog 1. Pressing the SET button then accesses the parameters within the Prog 2 submenu.

 *Prog 1 submenu*

 *Select the Prog 2 submenu*

 *Prog 2 submenu*

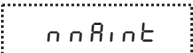
Pressing the DOWN button may access Prog 1. Pressing the UP button may access Prog 3. The well temperature is displayed by pressing the EXIT button. Pressing the SET button accesses the parameters within the Prog 2 submenu.

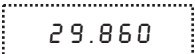
6.21.1 Maintain Temperature

The Maintain Temperature parameter is the temperature at which the instrument stabilizes and remains during the melt plateau in the gallium cell. The default value is 29.86°C and can be seen on Figure 1 as the temperature that the well goes to when the display shows *maint*. The instrument holds the well at this temperature until the melt plateau is ended.

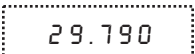
 *Prog 2 submenu*

 *Access the maintain temperature parameter*


 *Flashes before entering the maintain temperature parameter*

 *Current maintain temperature value*

Use the UP or DOWN buttons to adjust the value.

 *New maintain temperature value*

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.

 *Accept new maintain temperature value and continue to the maintain time-out*


6.21.2 Maintain Time-out

The Maintain Time-out setting is used to enable a preset delay time after which the gallium melt plateau is terminated. The default setting is OFF and at this setting the instrument remains at the Maintain Temperature until the user advances the Auto Program by depressing the SET and DOWN buttons and then selecting the FREEZE program state. A refreeze of the gallium cell is then started.

If the Maintain Time-out setting is ON, the Auto Program automatically ends the gallium melt plateau after the preset delay time has been reached and refreezes the gallium cell. It can be seen on Figure 1 under the Maintain portion of the Auto Program.

After accessing the Maintain Temperature parameter, the display flashes ϵ , \circ \circ ϵ and displays


 *Flashes before entering the maintain time-out parameter*

 *Current maintain time-out setting*

Use UP or DOWN buttons to adjust the value.

 *New maintain time-out setting*

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.

 *Accept new maintain time-out setting and continue to the maintain time-out time*

6.21.3 Maintain Time-out Time

The Maintain Time-out Time parameter is the time that the instrument maintains the gallium cell at the Maintain Temperature before terminating the gal-

lium melt plateau. The default value is 7200 minutes (5 days). It is only used if the Maintain Time-out parameter is set to ON. If the Time-out parameter is set to ON, the instrument ends the Maintain portion of the Auto Program after the Maintain Time-out Time has been reached and refreezes the gallium cell. It can be seen on Figure 1 under the Maintain portion of the Auto Program.

After accessing the Maintain Time-out parameter, the display flashes *d u r 4* and displays the current value of the Maintain Time-out Time.

d u r 4 Flashes before maintain time-out time parameter

7200 Current maintain time-out time value (7200 minutes)

Use the UP or DOWN buttons to adjust the value.

1440 New maintain time-out time value (1440 minutes)

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.



Accept new maintain time-out time value and continue to the freeze hot temperature

6.21.4 Freeze Hot Temperature

After the melt plateau is finished or the user has ended the melt, the instrument normally proceeds to refreeze the cell. If desired, the Freeze Hot Temperature and Freeze Hot Time parameters may be used to preset the instrument to totally melt the cell before refreezing.

The Freeze Hot Temperature parameter is the temperature at which the instrument totally melts the gallium in the cell. The value can be set up to 36.00°C and can be seen on Figure 1 as the temperature that the well goes to when the display shows *F r E E 2 H*. The instrument holds the well at this temperature for up to 6.0 hours to ensure that all of the gallium has been melted before refreezing the cell.

After accessing the Maintain Time-out Time parameter, the display flashes *F r E E 2 H* and displays the current value of the Freeze Hot Temperature.

F r E E 2 H Flashes before entering the freeze hot temperature parameter

29.86 Current freeze hot temperature value

Use the UP or DOWN buttons to adjust the value.

36.00

New freeze hot temperature value

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.



Accept new freeze hot temperature value and continue to the freeze hot time

6.21.5 Freeze Hot Time

The Freeze Hot Time parameter is the time that the instrument maintains the gallium cell at the Freeze Hot Temperature to totally melt the gallium in the cell. The value can be set up to 360 minutes (6 hours) and can be seen on Figure 1 as the time duration when the display shows *FR EEZH*. The instrument remains at the Freeze Hot Temperature for this time to ensure that all of the gallium has been melted before refreezing the cell.

Note: The default program does not use the Freeze Hot option and thus the Freeze Hot time is set to 0 seconds.

After accessing the Freeze Hot Temperature parameter, the display flashes *dur 5* and displays the current value of the Freeze Hot Time.

dur 5

Flashes before entering the freeze hot time parameter

0

Current freeze hot time value (0 minutes)

Use the UP or DOWN buttons to adjust the value.

360

New freeze hot time value (360 minutes)

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.



Accept new freeze hot time value and continue to the freeze cold temperature

6.21.6 Freeze Cold Temperature

The Freeze Cold Temperature parameter is the temperature to which the instrument scans during the refreezing of the gallium cell. The default value is 0.00°C and can be seen on Figure 1 as the temperature the unit scans to while the display shows *FR EEPC*.

After accessing the Freeze Hot Time parameter, the display flashes *FR EEPC* and displays the current value of the Freeze Cold Temperature.

FREEZE

Flashes before entering the freeze cold temperature parameter

0.000

Current freeze cold temperature value

Use the UP or DOWN buttons to adjust the value.

5.000

New freeze cold temperature value

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.

SET

Accept new freeze cold temperature value and continue to the freeze cold scan rate

6.21.7 Freeze Cold Scan Rate

The Freeze Cold Scan Rate parameter is the rate in °C/minute or °F/minute at which the instrument scans from the Freeze Hot Temperature to the Freeze Cold Temperature. The default value is 0.5°C/minute and can be seen on Figure 1 where the unit begins to refreeze the gallium cell. This parameter is very important for the proper gradient to be maintained during the refreeze process.

After accessing the Freeze Cold Temperature parameter, the display flashes **FREEZE** and displays the current value of the Freeze Cold Scan Rate.

FREEZE

Flashes before entering the freeze cold scan rate parameter

SR = 0.5

Current freeze cold scan rate

Use the UP or DOWN buttons to adjust the value.

SR = 0.5

New freeze cold scan rate

If no changes are desired, press the EXIT button to continue to the next parameter. Press the SET button to accept the new value and continue on to the next parameter.

SET

Accept new freeze cold scan rate value and continue to the Freeze Cold Time

6.21.8 Freeze Cold Time

The Freeze Cold Time parameter is the time that the instrument scans to and maintains the gallium cell at the Freeze Cold Temperature to totally refreeze the gallium in the cell. The default value is 150 minutes (2.5 hours). At the end of

this time, the Auto Program returns the well to 25°C to wait for the next melt process.

After accessing the Freeze Cold Scan Rate parameter, the display flashes `d u r 5` and displays the current value of the Freeze Cold Time parameter.

`d u r 5` *Flashes before entering the freeze cold time parameter*

`150` *Current freeze cold time value (150 minutes)*

Use the UP or DOWN buttons to adjust the value.

`180` *New freeze cold time value (180 minutes)*

If no changes are desired, press the EXIT button to Prog 1 submenu. Press the SET button to accept the new value and continue on to the next parameter.


 *Accept new freeze cold time value and continue to the Prog 2 submenu*

Prog 1 may be accessed by pressing the DOWN button. Prog 3 may be accessed by pressing the UP button. The well temperature is displayed by pressing the EXIT button. Pressing the SET button accesses the parameters within the Prog 2 submenu.

6.22 Prog 3

`Prog 3` is displayed by pressing the UP button while the display shows `Prog 2`. Pressing the SET button then accesses the parameters within the Prog 3 submenu.

`Prog 2` *Prog 2 submenu*

 *Select the Prog 3 submenu*

`Prog 3` *Prog 3 submenu*

Prog 2 may be accessed by pressing the DOWN button. The serial interface menu may be accessed by pressing the UP button. The well temperature is displayed by pressing the EXIT button. Pressing the SET button accesses the parameters within the Prog 3 submenu.

6.22.1 Freeze Melt State

The Freeze Melt State parameter is used to control the configuration of the Peltier devices and is only usable in the manual mode of operation. During normal operation of the instrument this parameter should not be altered.

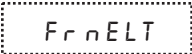
During the melt and maintain processes, the Peltier devices are configured to apply a uniform heat source to the well. This minimizes vertical gradients in the well and gallium cell to maximize the length of the melt plateau. During a Freeze process, the Peltier devices are configured to generate a large vertical gradient in the well to ensure the proper refreezing of the gallium cell.

The default setting for this parameter is MELT.

The Freeze Melt State parameter is accessed from the Prog 3 submenu.

 *Prog 3 submenu*

 *Access the Freeze Melt State parameter*

 *Flashes before entering the freeze melt state parameter*

 *Current freeze melt state setting*

Use the UP or DOWN buttons to adjust the setting.

 *New freeze melt state setting*

If no changes are desired, press the EXIT button to exit the Prog 3 submenu. Press the SET button to accept the new value and return to the Prog 3 submenu.

 *Accept new Freeze Melt State setting*

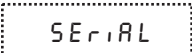
The display then shows *Prog 3* and returns to the Prog 3 submenu.

 *Prog 3 submenu*

Prog 2 may be accessed by pressing the DOWN button. The serial interface menu may be accessed by pressing the UP button. The well temperature is displayed by pressing the EXIT button. Pressing the SET button accesses the parameters within the Prog 3 submenu.

6.23 Serial Interface Parameters

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

 *Serial RS-232 interface parameters menu*

The serial interface parameters menu contains parameters, which determine the operation of the serial interface. The parameters in the menu are – BAUD rate, sample period, duplex mode, and linefeed.

6.23.1 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 of the serial communications may be programmed to 300, 600, 1200, 2400, 4800, or 9600. The default setting is 2400 baud.

`BAUD` *Flashes before displaying the serial baud rate setting*

`2400 b` *Current BAUD rate*

Use the UP or DOWN buttons to adjust the setting.

`1200 b` *New BAUD rate*

Press the SET button to store the new setting as the baud rate or press the EXIT button briefly to skip to the next parameter without storing the new setting.

6.23.2 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 instrument transmits the current measurement over the serial interface approximately every five seconds. The automatic sampling is disabled with a sample period of 0.

`SPER` *Flashes before displaying serial sample period setting*

`SP=1` *Current sample period (seconds)*

Use the UP or DOWN buttons to adjust the setting.

`SP=60` *New sample period*

Press the SET button to store the new setting as the sample period or press the EXIT button briefly to skip to the next parameter without storing the new setting.

6.23.3 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 instrument 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.

`DUP` *Flashes before displaying serial duplex mode setting*

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

Use the UP or DOWN buttons to adjust the setting.

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

Press the SET button to store the new setting as the duplex mode or press the EXIT button briefly to skip to the next parameter without storing the new setting.

6.23.4 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.

`LF` *Flashes before displaying serial linefeed setting*

`LF = 0 n` *Current linefeed setting*

Use the UP or DOWN buttons to adjust the setting.

`LF = 0 F F` *New linefeed setting*

Press the SET button to store the new setting as the linefeed.

Prog 3 may be accessed by pressing the DOWN button. The calibration menu may be accessed by pressing the UP button. The well temperature is displayed by pressing the EXIT button. Pressing the SET button accesses the parameters within the serial interface menu.

6.24 Calibration Parameters

The operator of the instrument has access to one of the calibration constants, R0. This value is set at the factory and must not be altered. The correct value is important to the accuracy and proper and safe operation of the instrument. This parameter should not be adjusted except in the event the controller's memory fails. The user may then restore this value to the factory setting. This constant is supplied to the user on the Report of Calibration with the manual.



CAUTION: *DO NOT change the value of the instrument calibration constant from the factory set value. The correct setting of this parameter is important to the safety and proper operation of the instrument.*

`C R L` *Calibration parameters menu*

Press the SET button five times to enter the menu.

6.24.1 R0


The calibration parameter R0 characterizes the resistance temperature relationship of the platinum control sensor. This parameter may be adjusted by an experienced user to improve the accuracy of the instrument.

To properly maintain the gallium cell during the melt plateau and during the refreezing process, the accuracy of the Model 9230 near the gallium melting point (29.7646°C) must be very precise, $\pm 0.05^\circ\text{C}$ or better. The value of R0 for this instrument refers to the resistance of the control probe at 0°C. For calibration purposes, the value of R0 is adjusted to compensate for the error at 29.27°C. The value of this parameter is set at the factory for best instrument accuracy. This parameter may be adjusted by an experienced user to improve the accuracy of the instrument (see Section 10, Calibration Procedure).

From the CAL submenu, pressing the SET button 5 times accesses the R0 parameter:

 *Calibration parameters menu*

Press the SET button five times to enter the menu.

 *Flashes before displaying the R0 setting*

 *Current R0 value (ohms)*

Use the UP or DOWN button to adjust the setting.

 *New R0 value (ohms)*

If no changes are desired, press the EXIT button to return to the CAL submenu. Press the SET button to accept the new value.

The display then shows CAL and returns to the CAL submenu. The serial interface menu may be accessed by pressing the DOWN button. The well temperature is displayed by pressing the EXIT button. Pressing the SET button five times accesses the R0 parameter within the CAL submenu.

7 Digital Communication Interface

The Model 9230 is capable of communicating with and being controlled by other equipment through the 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. Communications commands are summarized in Table 4 on page 52.

7.1 Serial Communications

The instrument 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 7, Controller Operation with the exception of the BAUD rate setting. Additionally, when the instrument is in Standby mode (well at 25°C and display shows $\text{R} \cup \text{E} \cup$), the user is required to use the front panel buttons, to enter Manual mode or to initiate the Auto Program.

The serial interface operates with eight data bits, one stop bit, and no parity.

7.1.1 Wiring

The serial communications cable attaches to the instrument through the DB-9 connector at the back of the instrument. Figure 7 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 (DB-9) and the shield. If the instrument is used in a heavy industrial setting, the serial cable must be limited to one meter in length.

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 pro-

RS-232 Cable Wiring for IBM PC and Compatibles

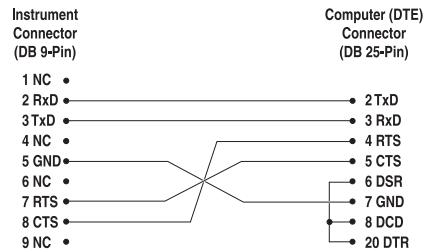
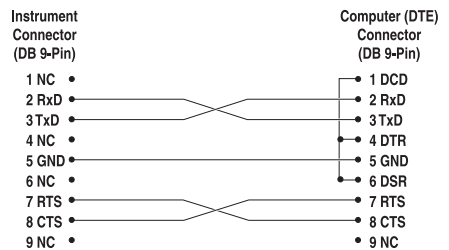


Figure 7 Serial Cable Wiring


grammed within the serial interface menu.

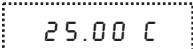
To enter the serial interface menu from the standby mode do the following:

 *Instrument in standby mode*

Use the UP or DOWN buttons to select the manual mode.

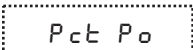
 *Instrument in manual mode*

 *Enter the manual mode*


 *Well temperature displayed*

 +  *Enter the secondary menu*

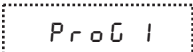
 *Flashes and then*

 *Flashes prior to displaying Peltier device power*

 *Peltier device power in percent*


 *Continue pressing the SET button until the display shows C o n F I G*

 *Flashes configuration menu*

 *Program 1 submenu*

 *Press the UP button three times until S E R I A L is displayed*

 *Serial interface menu*

 *Access the serial interface parameters*

7.1.2.1 Baud Rate

The baud rate is the first parameter in the menu. The display prompts with the baud rate parameter by showing “b A U d”. Press the SET button to choose to set the baud rate. The current baud rate value is displayed. The baud rate may be programmed to 300, 600, 1200, or 2400 baud. The baud rate defaults to 2400 baud.

Use the UP or DOWN buttons to change the baud rate value. Press the SET button to set the baud to the new value or the EXIT button 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 “*SPEr*”. 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 instrument transmits the current measurement over the serial interface approximately every five seconds. The automatic sampling is disabled with a sample period of 0. Press the SET button to choose to set the sample period. Adjust the period with the UP or DOWN button and then use the 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 “*DUP L*”. The duplex mode may be set to half duplex (“*HALF*”) or full duplex (“*FULL*”). With full duplex any commands received by the instrument 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 default setting is full duplex. The mode may be changed using the UP or DOWN buttons and pressing the SET button.

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 the UP or DOWN buttons and pressing the SET button.

7.1.3 Serial Operation

Once the cable has been attached and the interface set up properly the controller immediately begins transmitting temperature readings at the programmed rate. The set-point and other commands may be sent to the instrument via the serial interface to set the temperature set-point and to view or program the various parameters. The interface commands are discussed in Section 8.3, Interface Commands.

The serial interface operates with eight data bits, one stop bit, and no parity.

7.2 Interface Commands

The various commands for accessing the instrument functions via the digital interface are listed in this section. 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 an “=” character. For example, an “s”<cr> returns the current set-point and an “s=28.00”<cr> sets the set-point to 28.00 degrees.

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

Table 4 *Serial Interface Commands*

Command Description	Command Format	Command Example	Returned	Returned Example	Acceptable Values
Display Temperature					
Read current set-point	s[etpoint]	s	set: 9999.99(C or F)	set: 25.00 C	
Set current set-point to n	s[etpoint]=n	s=28			-5.00C to 40.00C
Read units	u	u	u:{C or F}	u: C	
Read temperature	t	t	t: 9999.99(C or F)	t: 29.86 C	
Set temperature units:	u[nits]=c/f				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	
Set scan function:	sc[an]=on/off[f]				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: 0.5C/min	0.1C/min to 5.0C/min
Set scan rate to n degrees per minute	sr[ate]=n]	sr=5			
Read program state	adv	adv	adv:{OFF, WAIT, PREP, MAINTAIN, FREEZHOT, FREEZCOLD}		
Advance program state	adv={adv/auto}	adv=adv			ADV, AUTO
Secondary Menu					
Read heater power (duty cycle)	po[wer]	po	po: 999.9	po: 12.3	

Command Description	Command Format	Command Example	Returned	Returned Example	Acceptable Values
Read proportional band	pr[op-band]	pr	pb: 999.9	pb: 8	
Set proportional band	pr[op-band]=n	pr=8.83			0.1C to 100C
Read set-point resistance	*sr	*sr	999.999	111.153	
Configuration Menu					
Program 1					
Read ready temp	rd[y]	rd	readytemp :99.99(C or F)	readytemp:29.2 7 C	
Set ready temp	rd[y]=n	rd=29.27			28.0 to 29.30 C
Read prep melt temperature	me	me	Preptemp:999.99(C or F)	Preptemp:30.77 C	
Set prep melt temperature	me=n	me=30.77			30.0 to 35.0 C
Read prep scan rate	ps[ra]	ps	Prepsrate :99.9(C or F)/min	Prepsrate:0.2 C/min	
Set prep scan rate	ps[ra]=n	ps=0.2			0.1 to 0.5 C/min
Read beeper setting	bee[p]	bee	beep: ON/OFF	beep: ON	
Set beeper	bee[p]=on/off[f]	bee=off			ON or OFF
Read prep 1 duration	prea	prea	Prep1dur :999 sec	Prep1dur :360 Sec	
Set prep 1 duration	prea=n	prea=400			360 to 600 sec
Read prep 2 duration	preb	preb	Prep2dur :999 sec	Prep2dur :360 Sec	
Set prep 2 duration	preb=n	preb=300			120 to 360 sec
Read prep 3 duration	prec	prec	Prep3dur :999 sec	Prep3dur :360 Sec	
Set prep 3 duration	prec=n	prec=400			240 to 480 sec
Program 2					
Read maintain temperature	ma	ma	ma:99.999(C or F)	ma:29.86 C	
Set maintain temperature	ma=n	ma=29.86			29.79 to 35.00 C
Read maintain duration	dm	dm	dm:99999	dm:7200	
Set maintain duration	dm=n/OFF	dm=1000			OFF,1 to 43200 min
Read freeze hot temperature	freh	freh	freezHtemp:99.99(C or F)	freezHtemp:99. 99(C or F)	
Set freeze hot temperature	freh=n	freh=35.00			29.86 to 36.00 C
Read freeze hot duration	dfrh	dfrh	freezHdur : 999 min	freezHdur : 150	
Set freeze hot duration	dfrh=n	dfrh=300			0 to 360 min
Read freeze cold temperature	fr[ec]	fr	freezCtemp :99.99(C or F)	freezCtemp :99.99(C or F)	

Command Description	Command Format	Command Example	Returned	Returned Example	Acceptable Values
Set freeze cold temperature	fr[ec]=n	fr=0.01			-0.01 to 10.00 C
Read freeze cold scan rate	fc[sr]	fc	freezCsrates: 0.2(C or F)/min	freezCsrates: 0.2(C or F)/min	
Set freeze cold scan rate	fc[sr]=n	fc=0.6			0.4 to 0.6 C/min
Read freeze cold duration	d[frc]	dfrc	freezCdur : 140 min	freezCdur : 140	
Set freeze cold duration	d[frc]=n	df=170			120 to 180 min
Program 3					
Read freeze/melt state	frm[t]	frm	FreezeMelt: MELT Mode	FreezeMelt: MELT Mode	
Set freeze/melt state	frm[t]=n	frm=melt			MELT or FREEZE
Serial Interface Menu					
Read serial sample setting	sa[mple]	sa	sa: 9	sa: 1	
Set serial sampling setting to <i>n</i> seconds	sa[mple]=n	sa=0			0 to 10,000
Set serial duplex mode:	du[plex]=f[ull]/h[alf]				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			
Set serial linefeed mode:	lf[eed]=on/off[f]				ON or OFF
Set serial linefeed mode to on	lf[eed]=on	lf=on			
Set serial linefeed mode to off	lf[eed]=off[f]	lf=of			
Calibration Menu					
Read R0 calibration parameter	r[0]	r	r0: 999.999		
Set R0 calibration parameter	r[0]=n	r=100.713			98.0 to 102.0 ohms
These commands are only used for factory testing					
Miscellaneous (not on menus)					
Read firmware version number	*ver[sion]	*ver	ver: 999,9.99	ver.9230,v1.00	
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 Melting Point of Gallium Realization

This section explains the procedure for realizing the melting point of gallium using a Model 5943 Stainless Steel Gallium Cell and the Model 9230 Maintenance System.

8.1 General

The gallium melting point (29.7646°C) is a critical temperature. Thermometers used in life science, environmental monitoring, at room or body temperatures, and many other applications depend on it for accurate calibrations. Laboratory standards rely on it as an ITS-90 check standard and as a means of measuring drift between calibrations. Use of the Model 5943 Stainless Steel Gallium Cell and the Model 9230 Maintenance System provides an easy way to generate and maintain the gallium melting point. Plateaus lasting up to 5 days are typical.

The Model 5943 Stainless Steel Gallium Cell holds a gallium sample that is 99.99999+% pure. The gallium is sealed in a Teflon envelope in a high purity argon atmosphere, which is then sealed inside a stainless steel housing. This double-sealing method reduces leaching of the gallium sample and ensures a life of ten years or longer for the cell. Gallium expands on freezing by 3.1% and, to compensate for this, the inner construction of the cell incorporates a flexible Teflon wall. Although the cell is more rugged than other gallium cells, care must be taken to prevent damage to the cell, particularly during the refreeze process. Because gallium expands on freezing (like water), the cell must be frozen from the bottom up or there is potential for damage to the cell. The Auto Program process of the Model 9230 ensures that the cell is frozen properly. The cell's entrant well dimensions are 8.7" (length) x 0.324" (diameter) with an immersion depth of 6.58" in gallium.

The Model 9230 Gallium Maintenance Apparatus is specifically designed to maintain the Model 5943 Gallium Cell. Once properly installed in the Model 9230, the Auto Program may be started and subsequently initiates the melt plateau, maintains the plateau for up to 5 days typical, and properly refreezes the cell for the next melt. When not actively melting or refreezing a cell, the Model 9230 automatically maintains the cell at 25.00°C (Standby), well below the melting point. To initiate a melt, the inner melt heater is inserted into the cell and the Auto Program is started through the Model 9230's front-panel buttons. The Model 9230 increases its temperature to just above the gallium point and powers the 8-watt inner melt heater. Approximately 30 minutes later, the gallium cell is ready to be used (see Figure 1).

The Model 9230 uses a Proportional Integral Derivative PID controller with specialized analog circuitry to control several Peltier devices. The controlled Peltier devices provide the appropriate amount of heating or cooling to the block assembly into which the gallium cell is inserted. The controller also controls the inner melt heater, cooling fan and beeper alarm. Two preheat wells ensure that thermometers are properly warmed when inserted into the gallium cell. The preheat wells are oversized so thermometers fit loosely and heat loss

from the gallium cell is neither too sudden nor too severe when cold thermometers are initially placed into the wells.

At the end of the melting plateau (or when the user wishes to end the plateau), the Auto Program is advanced. When totally melted, the Peltier devices are re-configured and controlled such that a large temperature gradient is generated between the top and bottom of the block. The instrument slowly scans to 0°C to maintain the gradient over several hours. This ensures that the gallium cell is frozen from the bottom up.

In summary, the following steps are involved in the realization of the melting point of gallium:

- Prepare the Model 9230 to receive the gallium cell.
- Prepare the frozen Model 5943 gallium cell to be installed in the Model 9230.
- Install the frozen Model 5943 Stainless Steel Gallium Cell into the Model 9230.
- Insert the top insulation plugs into the well on top of the cell.
- Insert the inner melt heater into the entrant well of the gallium cell.
- Start the Auto Program.
- Generate the melt plateau and remove the inner melt heater when prompted by the Model 9230 beeper alarm.
- Calibrate thermometers during the gallium melt plateau.
- Advance the Auto Program to refreeze the cell.

The details of operating the Model 9230 to realize the melting point of gallium are explained for each step in the following sections.

8.2 Preparing the Model 9230 to Receive the Gallium Cell

The Model 9230 is designed to be used in a controlled laboratory environment (see Section 3.2, Environmental Conditions). Control the ambient temperature to a fixed temperature within the specified operating range (18°C to 28°C). Rapid changes in ambient temperature of more than $\pm 1.0^\circ\text{C}$ reduces the stability of the instrument. Avoid drafty areas.

Place the Model 9230 on a flat surface with at least 6 inches of free space around and 18 inches above the instrument.

Make sure that the well of the instrument is clean and clear of any foreign objects, debris, dirt or grit.

Connect the inner melt heater to the back of the instrument making sure that it is securely connected. Place the inner melt heater cartridge into the melt heater storage well at the top back of the instrument.

Verify that the POWER switch is in the OFF position (“0”). Verify that the Inner Melt Heater switch is in the AUTO position. Install the power cord into the power entry module on the back of the instrument and plug the cord into a mains outlet of the proper voltage, frequency, and current capability. Turn on the power to the instrument by toggling the switch on the power entry module located on the back of the instrument. The fan should begin blowing air through the instrument, the beeper sounds one beep, and the display illuminates after approximately 3 seconds. After a brief self-test the controller should begin normal operation, the instrument will begin to approach 25°C, and the display will show μC .

8.3 Preparing the Frozen Model 5943 Gallium Cell for Installation in the Model 9230

Maintain the Model 5943 gallium cell at temperatures below the melting point (29.7646°C). The cell must be frozen when installed in the Model 9230.

Make sure that the outer surfaces and entrant well of the frozen Model 5943 gallium cell are clean and that the entrant well of the cell is clear of any foreign objects, debris, dirt or grit. Use pure or reagent grade alcohol and clean, soft cotton cloths to clean all surfaces if necessary. Thoroughly dry the cell before continuing.

Add a small amount of silicone oil 200.05, or equivalent transfer fluid, to the entrant well of the cell. **Do not overfill the entrant well.** The oil level in the well should be approximately the same level (52 mm below the top of the cell) as that of the gallium in the cell when the thermometer is in place in the well.

8.4 Installing the Frozen Model 5943 Gallium Cell into the Model 9230

The instrument comes with a cell removal tool to aid in installing and removing the gallium cell from the Model 9230 well. The tool has a spring-loaded handle with a compressible rubber tip. When the handle is squeezed, thereby compressing the spring, the rubber tip is extended and the tip may be inserted into the entrant well of the cell. Releasing the handle causes the rubber tip to compress and become firmly positioned in the entrant well. Once lodged in the entrant well, the cell can be gently lowered into the Model 9230 well. Wet the rubber tip of the tool with transfer fluid (e.g. silicone oil 200.05) for ease in inserting into the cell’s entrant well.



CAUTION: Do not use the cell removal tool to carry the cell. The tool is to be used only to aid in installing and removing the cell from the Model 9230 well. Do not drop the cell into the well. Damage to the cell and Model 9230 may occur if the cell is dropped.

With one hand holding the outside of the cell, insert the cell removal tool tip into the cell's entrant well as described above and verify that the rubber tip is firmly lodged after releasing the tool handle. Hold onto the removal tool with the other hand and gently lower the cell with the tool the rest of the way while carefully releasing the grip on the outside of the cell. When the cell is securely lowered into the Model 9230 well, compress the removal tool handle to loosen the rubber tip and gently remove the tool from the cell's entrant well. The gallium cell is now installed in the Model 9230.

8.5 Inserting the Top Insulation Plugs

The instrument comes with 4 rubber insulation plugs to be inserted into the Model 9230 well on top of the cell (2 spares). The plugs have a hole in the center through which thermometers can be inserted. The plugs serve two purposes. First, they help ensure an even temperature profile in the block and cell allowing a longer lasting and more accurate melting-point realization. Second, they protect against excessive moisture condensation during the cell refreezing process. Condensation can lead to corrosion over the long term and freezing water can damage the block.

Firmly insert two insulation plugs into the Model 9230 well on top of the cell. Make sure that they are positioned such that the center holes are aligned with the entrant well of the gallium cell.

8.6 Inserting the Inner Melt Heater

The inner melt heater's function is to melt a small volume of gallium around the entrant well of the cell when initiating the melt. The recommended practice for realizing the gallium melt by national standards laboratories requires two solid/liquid interfaces. An inner liquid interface exists at the reentrant well with an outer liquid interface completely surrounding the inner interface at the container wall. For a detailed description of the liquid/solid/liquid interfaces in the realization of the gallium melt see NIST Note 1265.

Verify that the inner melt heater is securely connected to the back of the instrument. Remove the inner melt heater from the storage well and carefully insert it through the holes in the top insulation plugs and into the entrant well of the gallium cell. Make sure that the heater cartridge is completely inserted to the bottom of the cell's entrant well.

8.7 Auto Program

The Model 9230 is designed to automate the gallium cell melt plateau. This is accomplished through the use of the Auto Program, a microprocessor-controlled program that determines set temperatures, scan rates, duration times, Peltier module configurations and melt heater controls. By controlling all of these parameters in the proper order, a temperature profile can be maintained in the block assembly that assures a uniform melt, maintains the melt over long

periods of time, properly refreezes the cell and keeps the cell ready for the next melt cycle. The Auto Program is illustrated in Figure 1.

8.7.1 Starting the Auto Program

After the frozen gallium cell has been properly installed in the instrument and the unit has been powered ON, the front panel LED display shows *Auto*. The instrument goes to 25°C and remains there until the SET button is pressed on the front panel starting the Auto Program. After pressing the SET button, the display changes to *Auto* and the Auto Program begins. The instrument automatically resets to 29.27°C at a scan rate of 0.2°C/minute in accordance with the Auto Program (see Figure 1).

8.7.2 Generating the Melt Plateau and Removing the Inner Melt Heater

Once the unit has stabilized at 29.27°C, it waits for a minimum of 30 minutes to ensure that the gallium cell has equalized in temperature. At the end of the equalization time, the Auto Program begins to initiate the melt. The display changes to *PREP* as the set temperature of the block is changed to 30.77°C. This initiates a melt on the outer surfaces of the gallium within the cell. A few minutes into the program, the unit beeps four times and the display changes to a message that flashes between *PREP* and *HEAT ON* to indicate that the inner melt heater has been turned ON. The inner melt heater remains ON for a few minutes, then shuts off as the display changes back to *PREP*, and the unit beeps 8 times. The user should remove the inner melt heater at this time (**Note:** Damage does not occur to the gallium cell if the heater is not removed, however, a shortening of the melt duration may result).

After the inner melt heater has been powered OFF, the instrument remains at 30.77°C for a few more minutes to complete the outer melt of the gallium cell. The unit then beeps 16 times, the display changes from *PREP* to *ONLINE* and the block temperature resets to 29.86°C to maintain the melting gallium cell.

8.7.3 Calibrating Thermometers

After the melt has been initiated, a preheated monitor thermometer may be inserted to monitor the temperature of the gallium cell. Due to the heat applied to the cell by the inner melt heater, the temperature of the cell's entrant well is higher than the gallium melting point (29.7646°C) for a short period of time. Generally, after 30 to 60 minutes, the temperature of the cell's entrant well is sufficiently stable to begin calibrations of thermometer sensors. The duration of the melt plateau varies depending on usage, but lengths of 5 days or longer are achievable.

The two preheat wells in the block may be used to heat up the thermometers before inserting them into the cell. The preheat wells are slightly oversized so thermometers fit loosely and heat loss from the gallium cell is neither too sudden nor too severe when cold thermometers are initially placed in the preheat wells.

8.7.4 Advancing the Auto Program and Refreezing the Gallium Cell

At the end of the melt plateau (or when the user wishes to end the plateau), the Auto Program is advanced by pressing the SET and DOWN buttons and then using the UP and DOWN buttons to select the next program state. Press SET to start the program. See Section 6.21.2, Maintain Time-out, for optional user defined Auto Time-Out. Gallium, like water, expands in volume when freezing. Therefore, the cell must be frozen from the bottom up or there is a potential for damage to the cell. The Auto Program process ensures that the cell is properly refrozen.

The Auto Program initiates the freeze and the display shows *F R E E Z E* at this point. The Peltier devices are reconfigured and controlled such that a large temperature gradient is generated between the top and bottom of the block. The instrument slowly scans to 0°C to maintain the gradient over several hours.

8.7.5 Return to Standby

Upon completion of the freeze, the Auto Program returns the unit to 25°C, the front panel LED display shows *8 0 E 0*, and the unit remains there until the user initiates the Auto Program again by pressing the SET key.

9 Calibration Procedure

To properly maintain the gallium cell during the melting plateau and during the refreezing process, the accuracy of the Model 9230 near the gallium melting point (29.7646°C) must be very precise, $\pm 0.05^\circ\text{C}$ or better. To achieve this, the instrument must be calibrated near this temperature. The value of R0 for this instrument refers to the resistance of the control probe at 0°C. For calibration purposes, the value of R0 is adjusted to compensate for the error at 29.27°C. The value of this parameter is set at the factory for best instrument accuracy and is located on the Report of Calibration shipped with the instrument. This parameter may be adjusted by an experienced user to improve the accuracy of the instrument. A calibration should be done at regular intervals, such as once a year, to ensure that it is operating properly.

9.1 Calibration Equipment

Calibration of the Model 9230 requires a standard thermometer and readout device that are adequately accurate. The combined accuracy of the thermometer and readout should be $\pm 0.0125^\circ\text{C}$ or better. Recommended equipment includes a laboratory grade PRT or thermistor probe with a length of 230 to 300 mm (9 to 12 inches). The calibration may be performed in a frozen Model 5943 gallium cell or in an aluminum test block that simulates the gallium cell. A small amount of silicone oil 200.05 (or equivalent) should be used in the entrant well for good thermal transfer from the cell to the thermometer.

9.2 Calibration Procedure

The accuracy of the Model 9230 at the gallium melting point (29.7646°C) is determined by the value of the calibration parameter R0. The calibration procedure involves measuring the error between the Model 9230 and the reference thermometer at a temperature near the gallium melting point and adjusting the calibration parameter R0 as necessary to reduce the errors to within acceptable limits. The stated accuracy of the Model 9230 is given in Section 3.1, Specifications. For calibration purposes, the temperature is measured at 29.27°C and R0 is adjusted as applicable. The following steps may be followed to calibrate the Model 9230:

1. Enter the Manual mode of operation and set the temperature of the Model 9230 to 29.270°C with a frozen gallium cell installed. Use a small amount of silicone oil 200.05 (or equivalent) in the entrant well for good thermal transfer from the cell to the thermometer. Be sure to install the 3 top insulation plugs on top of the cell. Insert the reference thermometer and monitor the temperature of the cell with an appropriate readout.
2. Allow the system to stabilize at 29.270°C for approximately 1 to 2 hours. When the stability of the reading is less than ± 0.02 over a 20 to 30 minute period record the average reading of the reference thermometer. This reading gives the error of the Model 9230 and may be used for “as found” data if re-

quired. No further action is required if the unit is found to be within acceptable limits.

3. If step 2 above shows that the Model 9230 is out of specification or acceptable limits, use the following equation to calculate a new R0 value:

$$R_2 = R_1 - (T_2 - T_1) * (0.3850)$$

Where,

R₂ = the new calculated value for R0

R₁ = the old value for R0 currently programmed in the Model 9230 memory

T₂ = the temperature measured in step 2 above

T₁ = the set temperature of the 9230 from step 1 above (29.270°C)

The value of R₁ (old R0) should be obtained from the memory of the Model 9230. Use the following process with the front panel buttons to obtain the currently programmed value of R0:

25.00 C *Instrument in manual mode; well temperature displayed*

SET + EXIT *Press the SET button and then the EXIT button to enter the secondary menu*

SEC *Flashes secondary menu*

Pct Po *Flashes percent power*

18.0P *Peltier device power in percent*

SET *Press the SET button until CONFIG is displayed*

CONFIG *Flashes configuration menu*

Prog 1 *Prog 1 submenu*

UP *Press the UP button until CAL is displayed*

CAL *Calibration parameter menu*

SET *Press the SET button 5 times to access the R0 value*

R0 *Flashes before entering the R0 setting*

99.983

Current R0 value (ohms)

4. Program the new value for R0 (R_2) from step 3 above into the Model 9230 memory (see Section 6.24.1, R0).

5. Repeat steps 1 and 2 to verify that the newly calibrated Model 9230 is now within acceptable limits. If not, perform steps 3, 4, and 5 again as necessary.

Note: If it is known that the Model 9230 is more than 0.5°C out of specification, such that, if the unit was calibrated at 29.270°C the cell would begin melting, then the above procedure should be performed below the 29.270°C set point as applicable (e.g., 28.5°C). After adjusting the unit at the lower temperature (e.g., 28.5°C), and verifying that the error had been reduced to less than 0.5°C, then the above procedure may be safely used.

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.
- It is important to keep the well of the instrument clean and clear of any foreign matter.
- The instrument should be handled with care. Avoid knocking or dropping the instrument.
- Examine the gallium cell for deformities before inserting it in the well. If there is any chance of jamming the cell in the well, file or grind off the protuberance.
- Do not drop the probes into the well. This type of action can cause damage to the entrant well of the gallium cell.
- If a hazardous material is spilled 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 with 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 does not damage the equipment.
- If the instrument is used in a manner not in accordance with the equipment design, the operation of the instrument may be impaired or safety hazards may arise.
- Do not drop the gallium cell into the instrument well. This type of action can cause a shock to the control sensor, thermoelectric modules, or damage the cell.

11 Troubleshooting

This section contains information on troubleshooting, CE Comments, and a wiring diagram.

11.1 Troubleshooting Problems, Possible Causes, and Solutions

In the event that the instrument appears to function abnormally, this section may help to find and solve the problem. Several possible problem conditions are described along with likely causes and solutions. If a problem arises, please read this section carefully and attempt to understand and solve the problem. If the problem cannot otherwise be solved, contact Hart Scientific Customer Service for assistance (1-801-763-1600). Be sure to have the model number and serial number of your instrument available.

Problem	Possible Causes and Solutions
Incorrect temperature reading	<p>Incorrect R0 parameter. Find the value for R0 on the Report of Calibration that was shipped with the instrument (or from subsequent calibrations of the instrument). Reprogram the parameter into the Model 9230 memory (see Section 6.24.1, R0). Allow the instrument to stabilize and verify the accuracy of the temperature reading.</p> <p>Controller locked up. The controller may have locked up due to a power surge or other aberration. Initialize the system by performing the Factory Reset Sequence.</p> <p>Factory Reset Sequence. Hold the SET and EXIT buttons down at the same time while powering on the instrument. After the instrument displays <code>-i n i t-</code>, release the buttons. The display shows <code>-i n i t-</code>, then displays <code>9230</code>, then displays the firmware version, and then displays <code>R u e o</code>. After performing the master reset sequence, all of the configuration parameters are reset to their default values. Reprogram the R0 parameter into the Model 9230 memory (see Section 6.24.1, R0) and any other applicable configuration parameters. Allow the instrument to stabilize and verify the accuracy of the temperature reading.</p>
Blank display after mains power applied	<p>Blown fuse. A fuse may have blown 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 the failure of a component. Always replace the fuse with one of the same rating, voltage, and type. Never replace the fuse with one of a higher current rating.</p>
The Instrument heats or cools too quickly or too slowly	<p>Incorrect scan and scan rate settings. The scan and scan rate settings may be set to unwanted values. Check the Scan and Scan Rate settings. The scan may be off (if the unit seems to be responding too quickly). The scan may be on with the Scan Rate set low (if unit seems to be responding too slowly).</p>

Problem	Possible Causes and Solutions
The display shows any of the following: <i>Err 1</i> , <i>Err 2</i> , <i>Err 3</i> , <i>Err 4</i> , or <i>Err 5</i>	Controller problem. The error messages signify the following problems with the controller. <i>Err 1</i> - a RAM error <i>Err 2</i> - a NVRAM error <i>Err 3</i> - a Structure error <i>Err 4</i> - an ADC setup error <i>Err 5</i> - an ADC ready error Initialize the system by performing the Factory Reset Sequence describe above.
The display shows <i>Err 6</i>	Defective control sensor. The control sensor may be shorted, open or otherwise damaged. Disconnect the power cord from the instrument. Looking at the front of the instrument (display), remove the front panel and right side panel. Locate the analog control board (on the block side of the electronics panel). The Molex connector at the top of the board connects the control probe to the controller (see wiring diagram, connector J4). Remove the connector and measure the resistance of the control sensor. It should be approximately 110 ohms at room temperature. If it measures close to this, make sure that it is securely re-connected to the controller, replace the panels and reapply power to the instrument. If the error message returns, there is a problem with the controller.
The display shows <i>Err 8</i>	Improper freezing. The Auto Program of the instrument is designed to monitor power levels and scan rates during the freezing cycle. If an unacceptable level or rate is detected, the unit will show <i>Err 8</i> and the beeper alarm will sound. To prevent any damage from occurring to the cell, remove it immediately and freeze it manually. To freeze manually, place the lower 1/3 of the cell in an ice bath with the top 2/3 exposed to the ambient air for approximately one hour until the cell freezes. The instrument may be reset by pressing any of the front panel buttons, but if the error message continues, the unacceptable freeze conditions are still present. Power the unit OFF and wait a few minutes. Then power the unit ON and reset the instrument by pressing any of the front panel buttons to see if the problem has been eliminated.
After starting the Auto Program the display shows <i>UBATE</i> and doesn't go into <i>PREP</i> .	Ambient temperature may be changing too rapidly. Rapid changes in the ambient temperature of more than $\pm 1.0^{\circ}\text{C}$ reduce the stability of the instrument. The Auto Program monitors the stability of the block at 29.27°C and if it is too unstable the melt does not proceed. Check the ambient temperature around the instrument for rapid changes. Avoid drafty areas.

11.2 Comments

11.2.1 EMC Directive

Hart Scientifics' equipment has been tested to meet the European Electromagnetic Compatibility Directive (EMC Directive, 89/336/EEC). The Declaration of Conformity for your instrument lists the specific standards to which the unit was tested.

11.2.2 Low Voltage Directive (Safety)

In order to comply with the European Low Voltage Directive (73/23/EEC), Hart Scientific equipment has been designed to meet the IEC 1010-1 (EN 61010-1) and the IEC 1010-2-010 (EN 61010-2-010) standards.