

Model K52

Argon Triple Point Cell
Operation & Maintenance Manual

by

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Argon Triple Point Cell

1. Background

This report documents the installation, operating and maintenance procedures for the Argon Triple Point Cell, Pond Engineering Model Number K52. Information contained in this manual is proprietary to Pond Engineering and is provided for use by the purchaser exclusively for instructional and maintenance purposes; any other use is prohibited.

2. General Information and Operating Principles

This Liquid Nitrogen-cooled apparatus utilizes a sealed cryogenic triple point cell with an integrated microprocessor-controlled maintenance system to realize and maintain the argon triple point. When properly used, the system is capable of realizing the argon triple point with an uncertainty better than ± 0.00035 K.

Figure 1 shows the general configuration of the Argon Triple Point Cell to provide the reader a better visualization of its appearance, as well as a point of reference for location and use of the control switches and service points discussed later in this manual.



Figure 1 - System General Layout

The system controller's display panel is located on the sloping front surface of the Argon Triple Point Cell. The cell is designed to provide a stable and uniform temperature environment for the realization and maintenance of the argon triple point and is intended for use in calibration of primary temperature standards. The system accommodates one capsule- or long-stem Standard Platinum Resistance Thermometer (SPRTs) at a time in the well in the center of the cell. Liquid Nitrogen, used as an expendable refrigerant and stored in an integrated 10 liter storage dewar, is used to cool the cell. The argon cell itself is positioned inside the storage dewar and is suspended below a second, smaller coolant cup. A single thermowell passes through the liquid nitrogen in the upper cup and down into the cell, providing good thermal guarding between the cell and the outside environment. Multiple thermal shields surround the sealed cell, which is suspended in vacuum for excellent thermal isolation. Active temperature control of the thermal shields provides an isothermal environment within which the argon sample can be held in a condition to realize the triple point.

Both the cell and the external sample storage vessel are constructed of cleaned, polished type 304 stainless steel - to withstand the approximate 40 atmospheres pressure of the gas sample at room temperature as well as preserve the purity of the sample. The .999 999 pure argon sample in the cell ensures consistent, flat temperature plateaus.

Standard features include an RS-232 or USB interface to allow operation of the Argon Triple Point Cell as part of an integrated automatic calibration system. IEEE-488 interface capability is available as a factory installable option.

A removable lower back panel is attached to the cabinet frame with button head screws. By removing this panel, one may gain access to the inner system components. Both side panels are also removable by actuation of the panel latches once the lower back panel is removed. The cover panels may be removed for inspection and service without disconnecting any system cabling.

Three front panel switches and a 2-line by 24-character Liquid Crystal Display (LCD) located in the front panel of the Argon Triple Point Cell provide the operator interface shown in **Figure 2**.

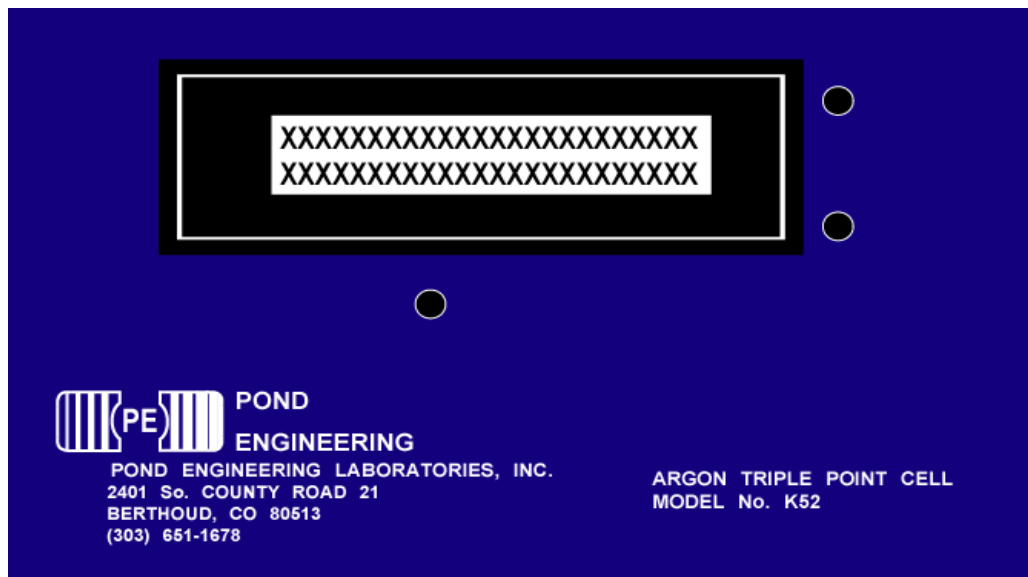


Figure 2 - Front Panel Layout

In the normal operating mode, this display presents the current temperature setpoint for the controller and the measured GUARD temperature on the first and second lines of the display respectively. Measurements are taken at approximately 3-second intervals and the lower line of the display updated following each measurement.

As liquid nitrogen is added to the internal storage dewar, the apparatus is cooled. An internal pump moves liquid nitrogen to the upper cup, allowing the argon cell body and sample gas to be cooled by conduction through the copper shunt below the upper cup. Liquid nitrogen is also pumped onto a wicking sleeve fitted to the outside of the cell vacuum envelope to minimize the effects of the varying liquid levels in the internal storage dewar. As the cooling progresses, argon will begin to condense in the cell. Within a relatively short period of time, the majority of the argon gas sample will have condensed into the cell and reduced the pressure in the sample storage vessel to less than one atmosphere. At this point, further cooling of the cell thermal guarding shields (referred to throughout this manual as "GUARD") causes the liquid argon sample contained in the cell to freeze. A more rapid cooling rate can be achieved by flooding the center well of the system with liquid nitrogen by starting a "freeze cycle". Instructions on how to use the freeze cycle can be found in **Section 2.1.2.3** of this manual. Following the operating principles for cryogenic fixed points outlined in NIST Technical Note 1265, the triple point is realized in this apparatus by controlling the temperature of thermal shields ("GUARD") surrounding the cell to a temperature very close to the triple point temperature and applying heat pulses directly to the cell using a separate heater in thermal contact with the outer cell envelope. As each heat pulse is applied to the otherwise adiabatic cell containing a completely frozen argon sample, a considerable temperature increase will be observed. When the cell

reaches the triple point, a small amount of solid argon is melted around the periphery of the cell with each pulse of heat, but no temperature increase will be observed in the center well. Once a number of heat pulses have been applied to the cell, the "shell" of solid argon will be deteriorated to the point where some transient effects of the heat pulse will be observed as sensible temperature changes in the center well.

An internal microprocessor-based digital controller provides controller functions to facilitate preparation of the cell, control the heat pulses, present diagnostic information, calibrate sensors, etc. Control functions are accessed through the front panel switches and messages are presented by the LCD display as outlined in the following sections.

2.1 System Controller

In an effort to maintain simplicity while providing flexibility to accommodate optional features, the operator interface is based largely on user-interactive software control. The software prompts the user through a set of "COMMAND FUNCTIONS", including "CHANGE SETPOINT TEMPERATURE" and "ADJUST SYSTEM VARIABLES". The user-interactive software displays messages on a 2-line by 24-character Liquid Crystal Display (LCD), prompting the user as necessary to perform all command functions through the use of three front panel switches surrounding the LCD. An outline of the user interface is provided in the following text and figures. It will provide a detailed description of the prompts and user actions necessary to set up the system edit configurations and operate the system manually.

Because of the multitude of functions that each switch will be called upon to perform, all "labeling" of the switches is provided from the software program presented to the user via the LCD display. (The labels attached to the switches in **Figure 3** are for the benefit of the reader in understanding the documentation herein. Such labels do not appear on the device front panel.)

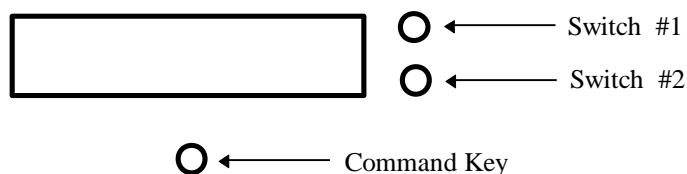


Figure 3 - Front Panel Switch Layout

2.1.1 Normal Operating Mode

The Argon Triple Point Cell requires approximately 10 liters of liquid nitrogen to realize the Triple Point and maintain the plateau for several hours. Normally, the Dewar will need to be filled once to cool the system from room temperature to the working temperature and once more to maintain the plateau through the pulsing period. Maintenance of the plateau for extended periods of time requires only periodic addition of liquid nitrogen to the internal storage dewar.

Pour the liquid nitrogen directly into the internal storage dewar via the large hole in the aluminum bezel located at the top of the system, shown in **Figure 4** below.

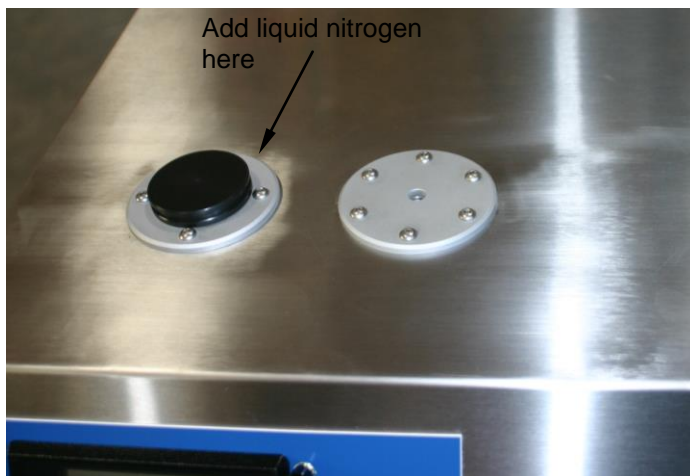


Figure 4 - Manual Liquid Nitrogen Fill Port

In normal operation, the Argon Triple Point Cell controller will continuously display the current temperature setpoint and the measured cell shield temperature as shown in **Figure 5**. Measurements are taken at approximately 3-second intervals and the lower line of the display is updated following measurement. This information is removed from the display when the user presses and holds the Command Key for a period of 2 to 3 seconds, accessing the "COMMAND FUNCTIONS" portion of the program.

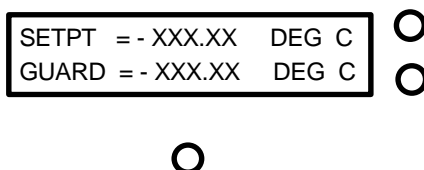


Figure 5 - Normal Operating Mode Display

2.1.2 The "Command Functions"

In order to perform the many functions necessary for operation, the user must leave the normal operating mode and enter the "COMMAND FUNCTIONS" mode. To do this, the user should depress and hold the Command Key. As the key is pressed, the words "COMMAND FUNCTIONS" will immediately appear on the lower line of the display. If the key is released, the normal operation screen will again be displayed and normal operation will continue. In order to proceed to Command Functions, the operator must press and *hold* the Command Key until the words "COMMAND FUNCTIONS" disappear. The operator should then release the key.

****Note****

The system has been designed to operate normally even when the user is accessing the control functions. However, the display of system conditions and remote communication will be suppressed until the system returns to the normal operating mode.

2.1.2.1 Change Setpoint Temperature

This function allows the user to change the setpoint temperature of the system controller to any temperature within the range of 50.000 to -198.000 °C. As shown in **Figure 6**, the label "YES" is adjacent to the top switch, which we identified earlier in **Figure 3** as "Switch #1". The "NO" is adjacent to the switch identified as "Switch #2". If the "NO" selection is made, the program will proceed to access the next function. However, if the user selects "YES" by pressing the corresponding switch, the display shown in **Figure 7** will be presented on the LCD.

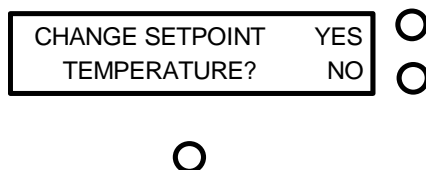


Figure 6 - Setpoint Access Prompt

The system controller is programmed with three memory "SETPOINT" temperatures. The user may select the first memory setpoint temperature by pressing the corresponding "YES" switch. A "NO" selection will advance the system to the next memory setpoint. A "NO" response to all of the three memory setpoint temperatures will allow the user to change the setpoint manually as shown in **Figure 8**.

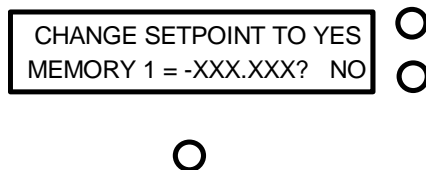


Figure 7 - Memory Setpoint Change Prompt

To adjust the "SETPOINT" manually, use the two switches on the side corresponding to the labels "UP" and "DN" displayed on the LCD. Each depression of a switch increments or decrements the selected digit by one unit. The "COMMAND" switch, located directly below the LCD, advances the cursor to each of the digits. The number presented on the display will be used as the current setpoint by the system controller even while in the command functions mode.

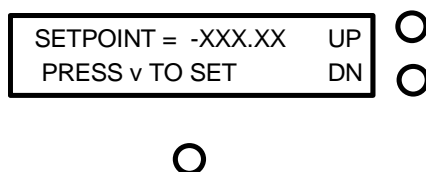


Figure 8 - Manual Setpoint Change Prompt

As the cursor is scrolled past the last digit, setpoint adjustment is complete and the user advances to the next command function, described in the following section.

2.1.2.2 Apply Heat to Cell

This function allows the user to apply a specific amount of heat to the cell. This function is only available when the temperature is at or below -40 degrees C. This function warms the cell once it is completely frozen. Responding "YES" to the prompt shown in **Figure 9** will apply the specified amount of heat to the cell and, thus, begin warming the solid argon in the cell. A "NO" response will move to the next command function.

ADD XXX JOULES OF	YES	<input type="radio"/>
HEAT TO CELL?	NO	<input type="radio"/>

☐

Figure 9 - Apply Heat to Cell Prompt

2.1.2.3 Freeze Cell Cycle

The Freeze Cell function gives the user the ability to automatically freeze and prepare the cell to realize the triple point. This function causes the internal liquid nitrogen pump to fill the upper dewar to the point that the thermowells are flooded, rapidly cooling and freezing the argon sample. Responding "YES" to the prompt in **Figure 10** will begin pumping liquid nitrogen, a "NO" response will move to the next command function.

START CELL	YES	<input type="radio"/>
FREEZE CYCLE?	NO	<input type="radio"/>

☐

Figure 10- Freeze Cell Cycle Prompt

When set up to complete a "FREEZE CYCLE", the controller will immediately begin to pump liquid nitrogen into the upper dewar and flood the thermowells. Flooding the thermowells will rapidly cool the cell, condensing and eventually freezing the argon sample. Flooding continues until the core temperature drops below the value established by the system variable FREEZE THRESHOLD for a period of time established by the system variable FREEZE TIME. While in the FREEZE CYCLE, alternating displays will alert the user that the wells are being flooded. **Figure 11** shows the prompt that is displayed during the cycle. The parameters controlling the freeze cycle are adjustable and are maintained on the VARS ARRAY discussed later in this manual.

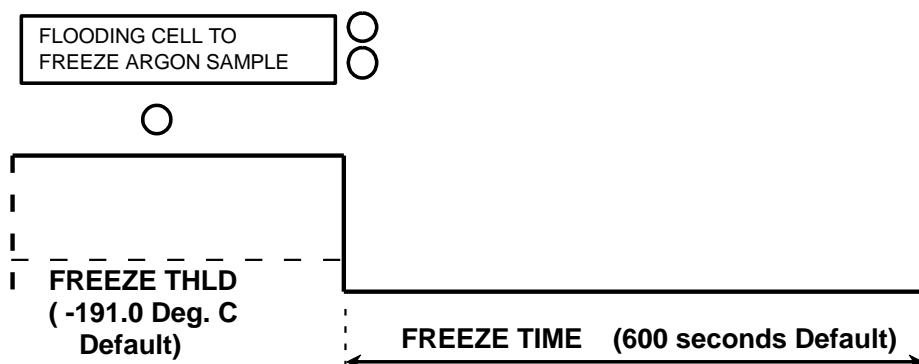


Figure 11 - Freeze Cycle Diagram

If one reenters the “COMMAND FUNCTIONS” and the freeze cycle is active a new prompt, **Figure 11**, will be displayed. This prompt allows the user to cancel the auto pulsing routine. A “YES” response will cancel the auto pulsing routine and a “NO” response will not cancel the routine and move to the next command function.

CANCEL FREEZE	YES	<input type="radio"/>
CYCLE?	NO	<input type="radio"/>

☐

Figure 11 - Cancel Freeze Cycle Prompt

2.1.2.4 Disable Auto Filling from External Dewar

The “DISABLE AUTO FILLING FROM EXTERNAL DEWAR” function gives the user the ability to disable the drive signal to the external fill solenoid valve, forcing the system to use only the LN2 in the internal storage dewar. Responding “YES” to the prompt in **Figure 12** will disable the auto-fill feature. The LCD will display a brief message confirming the action, and will move to the “ADJUST SYSTEM VARIABLES” command function. A “NO” response will not disable the auto-fill, and will move to the next command function

DISABLE AUTO FILLING	YES	<input type="radio"/>
FROM EXTERNAL DEWAR?	NO	<input type="radio"/>

☐

Figure 12 – Auto Fill Disable Prompt

If one reenters the “COMMAND FUNCTIONS” and the auto-fill is disabled, a new prompt, **Figure 13**, will be displayed. This prompt allows the user to cancel the auto pulsing routine. A “YES” response will enable the auto-fill feature and a “NO” response will not enable the feature and move to the next command function.

ENABLE AUTO FILLING	YES	<input type="radio"/>
FROM EXTERNAL DEWAR?	NO	<input type="radio"/>



Figure 13 - Auto Fill Enable Prompt

2.1.2.5 Force Start of Fill from External Dewar

This function allows the user to force the system to fill the internal storage dewar. Under normal operation, the internal storage dewar is only refilled once the level drops below the lower level sensor. Responding "YES" to the prompt in **Figure 14** will cause the system to fill the internal storage dewar using the auto filling feature. Responding "NO" to this prompt will cause the system to continue operating normally and move to the next command function.

FORCE START OF FILL	YES	<input type="radio"/>
FROM EXTERNAL DEWAR?	NO	<input type="radio"/>



Figure 14 - Force Fill Prompt

NOTE: If the Auto Filling feature is disabled, this prompt will not be available, and the system will skip to the next command function. To enable this prompt, Auto Filling must be enabled first.

2.1.2.6 Adjust System Variables

This command function allows the user to examine and adjust the system variables used by the controller as well as the calibration variables for the system core temperature measurement.

ADJUST SYSTEM	YES	<input type="radio"/>
VARIABLES?	NO	<input type="radio"/>



Figure 15 - System Variable Access Prompt

Pressing the switch by the display "YES", in **Figure 15**, will allow access to the first system variable by presenting the messages shown in **Figure 16**, responding with a "NO" command will terminate the function and advance to the next command function.

2.1.2.7 Specify Active Communications Port

The first system variable accessed is the active communications port. Unless the system is ordered with an IEEE-488 interface, it is equipped with both USB and RS-232 communications ports. The factory default configuration is to use the USB port. Upon accessing this variable, the LCD displays a prompt as shown in **Figure 16**. Responding "YES" to this prompt will immediately set the RS-232 port as the active communications port. Responding "NO" will move to the next system variable.

SET RS232 PORT AS	YES	<input type="radio"/>
ACTIVE PORT?	NO	<input type="radio"/>

☐

Figure 16 - RS-232 Select Prompt

The active communications port can be changed back by reentering the command functions and adjusting the system variable. If the RS-232 port is active, the LCD will display a prompt as shown in **Figure 17**.

SET USB PORT AS	YES	<input type="radio"/>
ACTIVE PORT?	NO	<input type="radio"/>

☐

Figure 17 - USB Select Prompt

2.1.2.8 Adjust Memory Setpoint Temperatures

The second variable accessed by the "Adjust System Variables" function is the "Memory 0", shown in **Figure 186**. The "Memory 0" variable is the system startup setpoint. Responding "YES" to the prompt will allow the user to adjust the setpoint, a "NO" response will move to the next memory setpoint.

ADJUST SYSTEM	YES	<input type="radio"/>
MEMORY 0 = XXX.XXX?	NO	<input type="radio"/>

☐

Figure 18 - Adjust Setpoint Memory Prompt

The adjustment of MEMORY 0 may be accomplished using the two switches on the side corresponding to the labels "UP" and "DN" displayed on the LCD. The "COMMAND" switch, located directly below the LCD, is used to advance the cursor to each of the digits.

As the cursor is scrolled past the last digit, memory setpoint adjustment is completed and the user is advanced to the next memory setpoint. When all memory setpoint adjustments have been presented, the system advances to the next System Variable, as described in the following section.

2.1.2.9 Adjust Cell Heat Pulse

The next variable accessed by this function is ADJUST CELL HEAT PULSE, the number of joules that will be applied by the cell heater per heat pulse. To adjust this variable, respond YES to the prompt in **Figure 197**.

ADJUST CELL HEAT	YES	<input type="radio"/>
PULSE = XXXX JOULES?	NO	<input type="radio"/>

☐

Figure 19 - Adjust Heat Pulse Access Prompt

Responding "YES" to the prompt will allow the user to modify the variable as described below, a "NO" response will allow access to the next system variable as described in the following section.

PULSE = <u>XXXX</u> JOULES UP	<input type="radio"/>
PRESS v TO SET DN	<input type="radio"/>

☐

Figure 20 - Heat Pulse Adjust Prompt

Modification of the variable is accomplished by pressing the two switches on the right of the LCD. The command key located directly below the LCD is used to advance the cursor to each of the digits. Pressing the switch adjacent to the "UP" prompt will cause the value of the selected digit to be incremented by one, while pressing the switch adjacent to the "DN" decrements it by one. The range of the variable is from 0 to 2000 joules. As the cursor passes the last digit, the value is stored as a temporary system variable.

2.1.2.10 (Optional) Adjust GPIB Address

If the system is equipped with an IEEE-488 interface, the next variable presented is "ADJUST GPIB ADDRESS". This is the parameter that determines the address at which the system can be accessed over the IEEE-488 interface.

ADJUST GPIB	YES	<input type="radio"/>
ADDRESS = XX	NO	<input type="radio"/>

☐

Figure 21 - IEEE-488 Address Adjust Prompt

The user may examine or adjust this variable by responding "YES" to the prompt, while a "NO" response will allow access to the next Command Function.

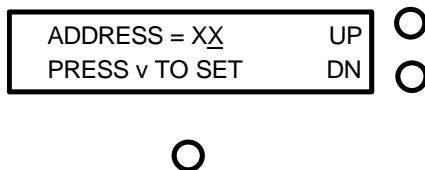


Figure 22 - Address Adjust Prompt

Here the user can modify the IEEE-488 address of the instrument as shown in Error! Reference source not found.22. The adjustment of address may be accomplished by using the two switches on the side corresponding to the labels "UP" and "DN" in Error! Reference source not found.22. This variable may be set within the range of 1 to 30. The Command Key, located directly below the LCD, is used to advance the cursor to each of the digits. Pressing the switch adjacent to the "UP" prompt will cause the value of the selected digit to increment one, while pressing the switch adjacent to the "DN" will decrement it by one. As the cursor passes the last digit, the value is stored as a temporary system variable.

2.1.2.11 Calibrate Shell Temperature Sensor

The next variable accessed by this function is the calibration variable for the shell sensor. The user is given access to these variables by responding "YES" to the prompt shown in **Figure 233**. A "NO" response will allow access to the next system variable in the sequence, as described in the following section.

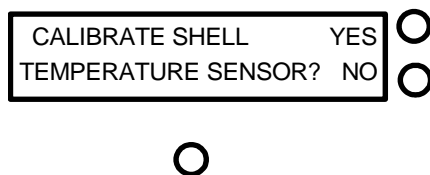


Figure 23 - Calibration Access Prompt

The first display presented allows the user to calibrate the system's shell temperature sensor. This calibration procedure adjusts the offset of the system's shell sensor to match the reading from an external SPRT. The calibration adjustment should be made near the argon triple point, and after the external SPRT and the system have reached stability. The calibration adjustment is accomplished by changing the offset variable for the shell sensor.

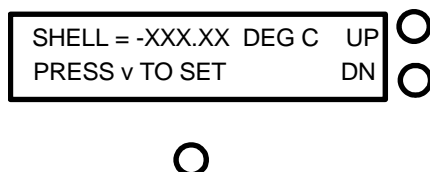


Figure 24 - Shell Sensor Calibration Prompt

The number presented in **Figure 244** is the current shell temperature as calculated by the measured shell temperature plus a temporary shell offset. This temporary shell offset is initially set to the current value of the shell offset being used by the control routines. Adjusting this temporary variable will have no effect on the measured shell temperature in the control routines until this function is completed.

Modification of the shell temperature, through adjustment of the offset value, is accomplished by pressing the two switches on the right of the LCD. Pressing the switch adjacent to the "UP" prompt will cause the apparent measured shell temperature to increase and pressing the switch adjacent to the "DN" will cause the apparent measured shell temperature to decrease. It is important to note that the "UP" switch actually increase the temporary shell offset variable and the "DN" switch decreases the temporary shell offset variable. The system will continually update the measured sensor temperature and displays the corrected temperature value so all changes are reflected on the display as they occur.

After the desired value has been established, by incrementing or decrementing the temporary offset variable, pressing the Command Key causes the value to be stored as a shell offset. Then the user is given the opportunity to examine or modify the shell offset value by the following prompt shown in **Figure 255**.

****Note****

At this point the value that is displayed as the **SHELL OFFSET** is being used in the control routines.



Figure 25 - Manual Shell Offset Access Prompt

It is recommended that the user record any new Shell Offset resulting from a calibration in a log similar to the log provided at the end of this manual. The prompts shown allow the user to manually adjust the offset value, by pressing "MAN", or "USE" this value as the calibrated offset. In the event that the user chooses to "USE" this value the system will skip the manual adjustment function discussed in the following section, while a choice to "MAN", or manually adjust the system, will display the prompt shown in **Figure 266**.

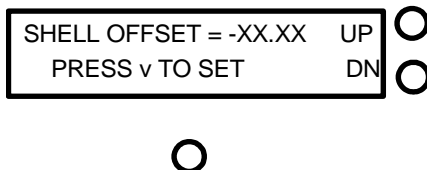


Figure 26 - Manual Shell Offset Adjustment Prompt

Manual adjustment of Shell Offset may be accomplished using the two switches on the side corresponding to the labels "UP" and "DN" displayed on the LCD. The Command Key, located directly below the LCD, is used to advance the cursor to each of the digits.

As the cursor is scrolled past the last digit, Shell Offset adjustment is completed and the system sets the variable to the newly established value and advances to the next System Variable, as described in the following section.

2.1.2.12 Access Vars Array

This function allows access to variables stored in the system's memory. It is strongly recommended that before responding YES to the prompt in **Figure 27**, one carefully reviews what each variable represents

(see chart below). It is also strongly recommended that only experienced users familiar with this system and its limitations exercise this option.

****DO NOT ADJUST VARS ARRAY WITHOUT FIRST CONSULTING FACTORY!!!** Doing so can cause severe damage to the system and could be very dangerous! If the message UNABLE TO LOAD VARIABLES should appear on the front panel display call Pond Engineering [(303)-651-1678] immediately for service.

ACCESS VARS	YES	<input type="radio"/>
ARRAY?	NO	<input type="radio"/>



Figure 27 - Access Variables Array Prompt

Responding YES to the prompt in **Figure 27** will cause the prompt in **Figure 28** to appear. This is to warn the user of the danger if this function is not used properly. Press Switch #1 to continue or Switch #2 to exit to the next function.

WARNING	CONTINUE	<input type="radio"/>
REFER TO MANUAL	EXIT	<input type="radio"/>



Figure 28 - Warning Prompt

If "CONTINUE" is selected, variables will be presented in series as shown in **Figure 29** and can be changed as shown below. The variables are presented by number and are not text labeled. Please refer to the following chart for information about each variable.

VAR XX = XXX.XXXX	UP	<input type="radio"/>
PRESS v TO SELECT	DN	<input type="radio"/>



Figure 29 - Variable Adjustment Prompt

To scroll through the variables, press either of the two switches to the right of the LCD, the switch adjacent to UP increments the variable number and the switch adjacent to DN decrements it. The command key located directly below the LCD is used to start the adjustment of the selected variable by advancing the cursor through the digits.

Once a variable has been selected, pressing the switch adjacent to the UP prompt shown in **Figure 29** will cause the value of the selected digit (of the variable value) to increment one, while pressing the switch adjacent to the DN will cause it to decrement by one. As the cursor passes the last digit the value is stored as a temporary system variable.

****WARNING** Software limit checking is not done for these entries and inputting values outside the recommended ranges can result in permanent damage to the system if utilized for operating the system.**

The following is a list of commonly accessed system variables and their locations.

#	Variable \ Description	Default Values	Recommended Range
0	Current Setpoint Temperature	-189.6 degrees C	-197 to -189 degrees C
1	Memory 0 Setpoint Temperature (Power ON)	-191.0 degrees C	-197 to -189 degrees C
2	Memory 1 Setpoint Temperature	-189.2 degrees C	-197 to -189 degrees C
3	Memory 2 Setpoint Temperature	-197.0 degrees C	-197 to -189 degrees C
4	Memory 3 Setpoint Temperature	-189.0 degrees C	-197 to -189 degrees C
6	GPIO Primary Address	6.0000	1.0000-30.0000
9	Cell Heater Enabled		1- Enabled, 0 - Disabled
14	TPW value for core sensor – Ohms	100.0	See Calibration Log
15	ITS-90 A Coefficient Below 0.01	0.0	See Calibration Log
16	ITS-90 B Coefficient Below 0.01	0.0	See Calibration Log
17	Automatic freeze cycle control	0.0	0 = normal 1 = Initiate automatic freeze cycle
18	Guard Offset - Deg. C	0.0	-10.0 to 10.0
20	ACCESS CODE for protected variables (variables 21 and up)	0.0000	Contact Factory
27	Proportional Gain	8.0	4.0 to 16.0
28	Integral Gain	0.1	0.05 to 0.20
31	Derivative Gain	-70.0	-35.0 to -140.0
34	Remote Fill Control	1.0	0 = Inhibit Fill 1 = Normal Fill Mode 2 = Force Fill (Once full reverts to 1)
39	Measured core sensor PRT resistance – Ohms	Current reading	
40	Measured Core Temperature– Deg. C	Current reading	
41	Averaged Core Temperature– Deg. C	Current reading	

2.1.2.13 Save changes to Variables

The prompt in **Figure 30** provides the opportunity to save the newly established system variables as the power-up default values. As mentioned earlier, the newly established system variables are stored as temporary system variables at this point. Responding "YES" to this prompt stores the variables in the non-volatile memory within the system controller. The variables will then be utilized as the systems power-on defaults.

SAVE CHANGES TO YES ☐
 VARIABLES? NO ☐



Figure 30 - Save Variables Prompt

Responding "NO" to this prompt causes the system to exit the function without saving the variables to non-volatile memory. This allows the user to temporarily establish new values for the system variables without permanently altering the default values. If the power were to be shut off or interrupted before the new information was stored in the non-volatile memory, however, the newly established values would be lost and the system would utilize the default values. Following verification of performance of the newly established variable values, they may be stored by entering the "ADJUST SYSTEM VARIABLES" routine, stepping directly through the prompts and responding "YES" to the "SAVE CHANGES" prompt.

2.1.2.14 Diagnostics Display Mode Select

Here the user is given the opportunity to choose which set of information is presented by the display during operation. Two options are available, "NORMAL DISPLAY" and "DIAGNOSTIC INFORMATION". The "NORMAL DISPLAY" mode is always presented upon system power up and is changed through this control function to allow display of "DIAGNOSTIC INFORMATION" by answering "YES" to the command function prompt.

DISPLAY DIAGNOSTIC	YES	<input type="radio"/>
INFORMATION?	NO	<input type="radio"/>

☐

Figure 31 - Diagnostics Mode Display Prompt

If the system is currently displaying "DIAGNOSTIC INFORMATION", the prompt will be modified as shown below to allow the user to toggle back to the "NORMAL DISPLAY" mode by answering "YES" to the command function prompt.

CHANGE TO NORMAL	YES	<input type="radio"/>
DISPLAY?	NO	<input type="radio"/>

☐

Figure 32 - Normal Mode Display Prompt

The "DIAGNOSTIC INFORMATION" display presents an array of numeric information for system diagnostics for the "CORE" zone and the "GUARD" zone. An example of the core diagnostic information is shown below in Error! Reference source not found.30.

300.0	300.0	993	==>C	<input type="radio"/>
192.0	1.00	1200	G	<input type="radio"/>

☐

Figure 33 - Core Diagnostics Display

The information presented by the CORE DIAGNOSTICS display is as follows. Top row from left to right: target joules at end of pulse cycle, joules per cycle, seconds to start next pulse. Bottom row left to right: number of joules currently added, heater relay status (1=picked or ON, 2=unpicked or OFF), number of seconds between pulse periods.

Pressing switch #2 will allow the user to toggle between the core and guard diagnostics display. The arrow indicates the currently presented diagnostic display.

-189.30	0.01	-0.10	==>G
-189.30	1.50	1.41	C



Figure 34 - Guard Diagnostics Display

The information presented by the GUARD DIAGNOSTICS display is as follows. Top row left to right: guard zone setpoint temperature (degrees C), guard proportional signal, guard derivative signal. Bottom row left to right: current guard temperature, guard integrator signal, current guard zone heater voltage.

****Note****

Once the "DIAGNOSTIC INFORMATION" mode has been entered the display will only present the diagnostic information; normal display mode can be obtained through the "COMMAND FUNCTIONS" options as described above.

After completing all of the command functions as described above, the system will again return to the normal operating mode, presenting the normal display or diagnostic display information as selected by the user.

2.2 Remote Interface Commands

If the K52 Argon Triple Point has been equipped with the optional IEEE-488 Remote Interface, any host computer which can be set up as a system controller may be used to operate the K52 over the interface. Pond Engineering has software available for interfacing with the system variables using an IBM compatible or Macintosh computer with a National Instruments 488 interface card. Custom software packages are also available to integrate the K52 as part of an automated lab. The system variables which can be accessed over the 488 interface can be found in section 3.2.2.5. *Access Vars Array*. Also included on the CD-ROM is a copy of Pond Engineering's GPIB Communication software, see section 5.1 for operation.

2.2.1 Pond Engineering GPIB Software

Included on the CD-ROM is a copy of the Pond Engineering GPIB Communication Software. This utilizes a simple command set by just referencing a variable and setting the variable to read or write.

Installation

To begin the installation insert the CD-ROM into a windows x86 PC, let the auto run start and click on *Install Pond Engineering GPIB Driver*. Alternatively you can go to D:\ PEL GPIB Driver Setup.exe and double click the icon to start the installation where D is your CD-ROM drive. From there follow the prompts to install the software.

Usage

To start the software go to Start – Program Files – Pond Engineering GPIB Driver. The screen should look like this.

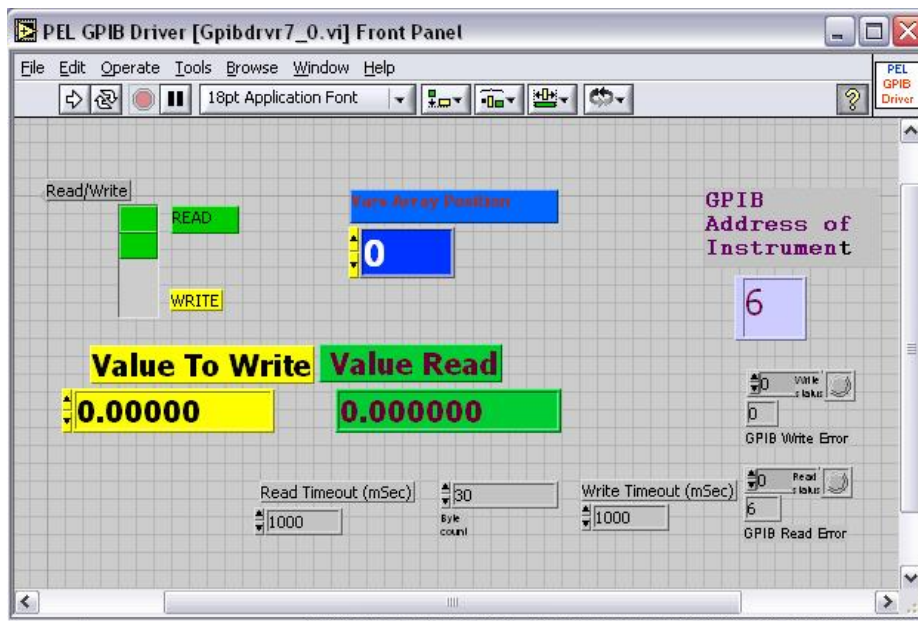


Figure 35 - PEL GPIB Screen

From here you can select the Variable Number (blue) in which you wish to change/read, select Read or Write (Yellow), and run the program to receive or write the value. (Note: the variable numbers are on page 16 of this manual) The values read will show in the green Value Read box, those you wish to write you enter in the yellow Value to Write box. To run the program, push the arrow button in the upper left part of the toolbar. If you need to change the timeout, byte count, or GPIB Address you can do so here in the indicated windows.

2.2.2 K52 Argon Triple Point Command Set

The command set for the system is outlined in the following paragraphs. The commands are issued from the host computer which acts as the controller. The K52 acting as the slave listens when addressed as a listener, talks when addressed as a talker, but does not issue a service request(SRQ).

Read System Variable

The "READ" command, called by sending an "R" (upper or lower case) followed by the two digit address of the variable, enables the user to read or interrogate the current values for system variables. The syntax for the read command is "Rxx", where xx is the address of the variable to interrogate. The address must be two digits in length, therefore addresses less than 10 MUST HAVE LEADING ZEROS. After receiving a read command, the system will wait to be addressed as a talker to return the data over the bus. The system has a one second write time-out, thus the controller in charge must read from the system within one second after sending the "R" command to receive data. For example: If the user wants to see the value stored as the system's alarm temperature, the user would send the character string: R05. The controller in charge would then address the system as a talker and wait for the data to be returned.

****Note:** Control of the K52 remains the highest priority even when the system is in remote mode and may put off responding to 488 commands for as long as 500ms.

For Example, having been addressed as a talker, the K52 will return the text string +4.300000e+02(space)05 as soon as it is not performing control functions. This indicates that the value 430 (°C) is the current value for Variable 05 (the Alarm Temperature). Values returned are always in the above scientific format followed by a space and address number for the given value.

Write System Variable

The "WRITE" command, executed by sending a "W" (upper or lower case) followed by the two digit address, a comma, and the desired new value, enables the user to write or set the values for system variables. The syntax for the write command is "Wxx, (value)", where xx is the address of the variable and (value) is the new desired value for the variable. The format for (value) must be decimal with at least seven significant digits, not to exceed 15 digits in length.

****Note:** As variables are written to the system the new value is immediately used by the system. However this new value is not stored as a permanent system variable until the user saves the variable using the "Save variables" command through the front panel.

IMPORTANT! SAVING CHANGED VARIABLES WILL PERMANENTLY CHANGE THE SYSTEMS VARIABLES. Pond Engineering strongly recommends the users keep a log of any changed variables. Also, Pond Engineering is in no way responsible for any damage caused by the failure to use these commands properly. **DO NOT** set a variable outside the recommended range. In order to maintain flexibility the only variables that are limit checked are the Setpoint variables and the alarm variable. All other variables can be set to any value received over the 488. Extreme caution must be used when setting any variable over the 488. The user should read back any variable after setting it to insure the variable was received by the system correctly. This is especially important when setting the RTPW's and calibration coefficients, since an errant RTPW or coefficient may cause the system to go out of calibration.

Remote Mode

After receiving a command over the 488 Buss, the system enters remote mode. In remote mode the LCD will display the message shown in Figure 33.

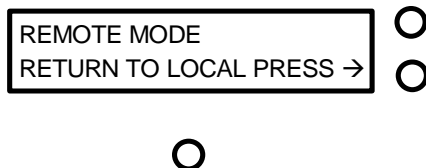


Figure 36 - Remote Mode Display

In remote mode, the system will only respond to the return to local switch (switch #2). After returning to local control the system will respond normally to all switches.

3. Normal Operating Procedures

Outlined below are recommended operating procedures for realizing the argon triple point utilizing the model K52 apparatus with liquid nitrogen as the coolant.

3.1 Preparing the System

Connect the system to an appropriate power source consistent with the input power designation on the system Serial Number placard (Normally 85 to 265 VAC at 2 Amps) and the compressed air source. Also connect the Liquid Nitrogen source (if equipped with the automatic fill option). Switch on the power and verify proper operation of the controller and normal display in accordance with the controller description earlier in this manual. Establish a system setpoint of -189.6°C or below.

Automatic Fill - Units need only to have LN2 available at the LN2 connection port (liquid transfer line connected and pressurized liquid dewar supply valve open) to begin the cool down process and fill the internal storage dewar. This process should be completed automatically within approximately 30 minutes.

Manual Fill - Filling the internal storage dewar (approx. liquid capacity 10 liters) with approximately 3 liters of liquid nitrogen will begin the cool-down process. Take the usual cautions to accommodate the escaping nitrogen gas and limit the rate of filling until the dewar surfaces cool down close to liquid nitrogen temperatures. Once the rapid boiling subsides, the dewar can be filled to within 7" (18 cm) of the top of the cabinet and the system allowed to cool unattended.

Once the shell temperature has cooled to -150°C or below, starting the FREEZE CELL CYCLE will cause the system controller to increase the upper dewar fill level and allow liquid nitrogen to enter the cell thermometer wells through the liquid ports, flooding the thermowells. The liquid nitrogen is then able to remove heat directly from the cell center well and cool the system much more rapidly and completely freeze the argon sample. While this cycle is in process, the front panel will alternately display FLOODING CELL TO FREEZE ARGON SAMPLE and the normal display (or diagnostic display if selected). During this process it is normal for the shell temperature to be cooled to well below the system controller setpoint since the presence of LN2 in the thermowells overwhelms the controller heater output by a large margin.

NOTE: Some liquid nitrogen may escape from the system at the top of the cabinet during this process.

Keeping the system setpoint between -189.2 and -189.8 during this process will allow the system to begin warming the cell toward the argon triple point temperature as soon as the cycle ends. After the cycle is completed (approximately 15 to 30 minutes, depending on the system temperature when the cycle was started) the internal shields and cell will have been cooled to the point where the argon gas sample will have condensed in the cell envelope and been frozen.

Once the FREEZE CELL CYCLE completes, (the FLOODING CELL message no longer is displayed), it is recommended that a room temperature thermometer or glass rod be fully inserted (and immediately withdrawn) into each of the wells to displace and boil off any liquid nitrogen remaining in the wells. **Be careful to protect yourself from the cold gas and liquid that will escape from the thermowell as the nitrogen is displaced.** A check thermometer (inserted near room temperature if desired) may be used to monitor the temperature of the cell at this time and the system allowed to equilibrate to a temperature close to the argon triple point (a setpoint of -189.6°C is recommended). Heat pulses may be applied using the system controller functions if desired to decrease the time necessary to complete the warming process.

NOTE: A 300 joule pulse will increase the temperature of the cell and solid sample approximately 0.6°C when the sample is completely frozen.

At this point the liquid nitrogen level in the storage dewar should be checked and the storage dewar refilled if necessary. Liquid nitrogen may be added to the storage dewar at any time without disturbing the thermal equilibrium of the system.

3.2 Pulsing the Cell onto the Plateau

When the sample is confirmed to be completely frozen, and has been allowed to equilibrate for a minimum of 20 minutes at a temperature between -190.0°C and -189.5°C , the system setpoint should be changed to -189.30°C , and the user should initiate delivery of a 300 to 1000 Joule pulse of heat to the cell; allowing the system to move up onto a plateau at the argon triple point as indicated by a check thermometer. This procedure should form an “outer melt” or layer of liquid argon adjacent to the inner wall of the stainless steel cell wall.

NOTE: Additional heat pulses may be applied during the life of the plateau to verify that the system is on plateau. A 300 Joule heat pulse will typically result in less than a 0.0003°C spike in the sensed temperature of a check thermometer, with the spike lasting less than 10 minutes. Application of additional heat pulses will shorten the useable duration of a plateau.

For best results, an “inner melt” should be also be formed at this point and may be easily accomplished by removing the check thermometer and allowing it to warm. After the thermometer is near room temperature, it can be reinserted into the cell to form the inner melt.

NOTE: It is recommended that the user periodically (weekly to quarterly depending on conditions) verify that the calibration of the internal guard control sensor is within $\pm 0.05^{\circ}\text{C}$ using a check thermometer. It is critical that the sample be completely frozen when verifying calibration or calibrating the internal sensor. During this check, supporting the check thermometer approximately 12 cm above the bottom of the cell well places the sensing element of the thermometer in much better thermal contact with the guard shells and results in a much shorter time constant in the equilibration process. With the thermometer at the bottom of the well, several hours time are typically required to equilibrate the system.

3.3 Calibrating Multiple Thermometers

Multiple thermometers may be calibrated on a single plateau by inserting chilled thermometers into one of the thermowells.

NOTE: The storage dewar fill port may conveniently be used to chill thermometers by directly inserting them into the fill port.

Readings taken with a check thermometer before and after calibration of additional thermometers should agree within 0.0005°C .

NOTE: The setpoint temperature may be varied while the system is on plateau to verify proper operation of the system. A system setpoint change of 0.05°C typically results in a sensible change on the order of 0.00012°C when measured by a check thermometer when the system is on plateau.

3.4 Hydrostatic Head Correction

Triple point conditions are maintained inside the cell at the vapor /liquid /solid interface which occurs only at the upper surface of the argon. Since there is hydrostatic head pressure imposed by the column of liquid argon at the depth of immersion of this cell, a correction must be applied to the theoretical triple point temperature to account for the static head pressure present at the sensing point. For all practical purposes, the liquid surface of the argon is 17.7 cm above the bottom of the thermometer well resulting in an increase of 0.0004 degrees C and variability between cells is negligible. This results in a temperature, at the center of the sensing element of a SPRT inserted in the thermowell, of -189.3438 degrees C (ITS-90). Data concerning cell construction is provided in Section 7 of this manual for reference and to allow independent verification of hydrostatic head corrections applied.

4. Handling and Maintenance

4.1 System Maintenance

The Argon Triple Point Cell is designed and fabricated to require minimal periodic maintenance. The following information is provided to guide the user in maintaining the system. Care should be taken to avoid accumulation of excessive moisture in the thermometer well or in the system dewar. After each use or when significant moisture accumulation is observed, the system setpoint should be set to 50.0 °C and the system allowed to remain stable at this temperature for 12 hours or more to drive off excessive moisture.

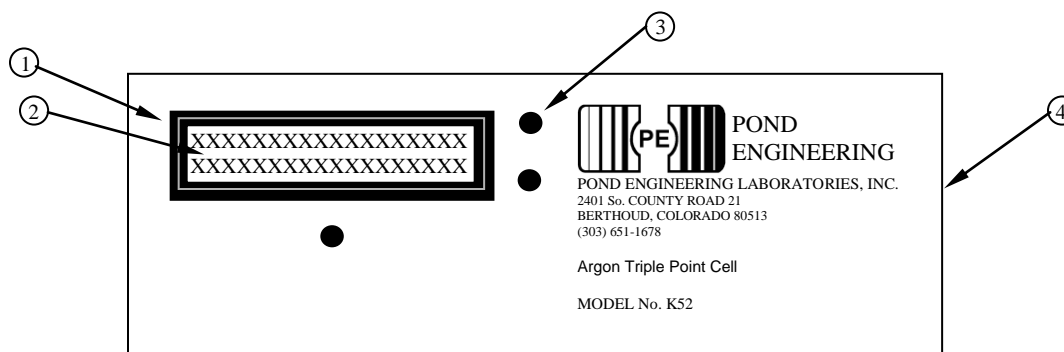
4.1.1 Cleaning

Exterior surfaces of the cabinet are coated with a durable polyurethane coating which is not affected by most solvents and cleaning agents. It is recommended that exterior surfaces be cleaned with alcohol or a mild commercial cleaning agent.

5. System Hardware Description

A brief description of system hardware is provided in this section as a reference to aid the user in periodic maintenance of the system. In the event significant maintenance or repair is required, it is recommended that Pond Engineering be contacted prior to replacing or modifying major system components.

5.1 Front Panel



<u>REF.#</u>	<u>NAME</u>	<u>MAKE</u>	<u>MODEL</u>
1	Die Cast Metal Bezel	JMJ Technical Products	1-458
2	24X2 Dot Matrix LCD Module DMC Series	Optrex	DMC20261
3	Miniature Push-button panel Mount SPDT Switches - 3 each - with Caps for Miniature Switches	Eaton Cutler-Hammer	PS1-100Q W-KN-17
4	Engraved Laminate Front Panel	Pond Engineering	K38-FP

5.2 Rear Panel



1/2" O.D Flare
for Liquid
Nitrogen fill

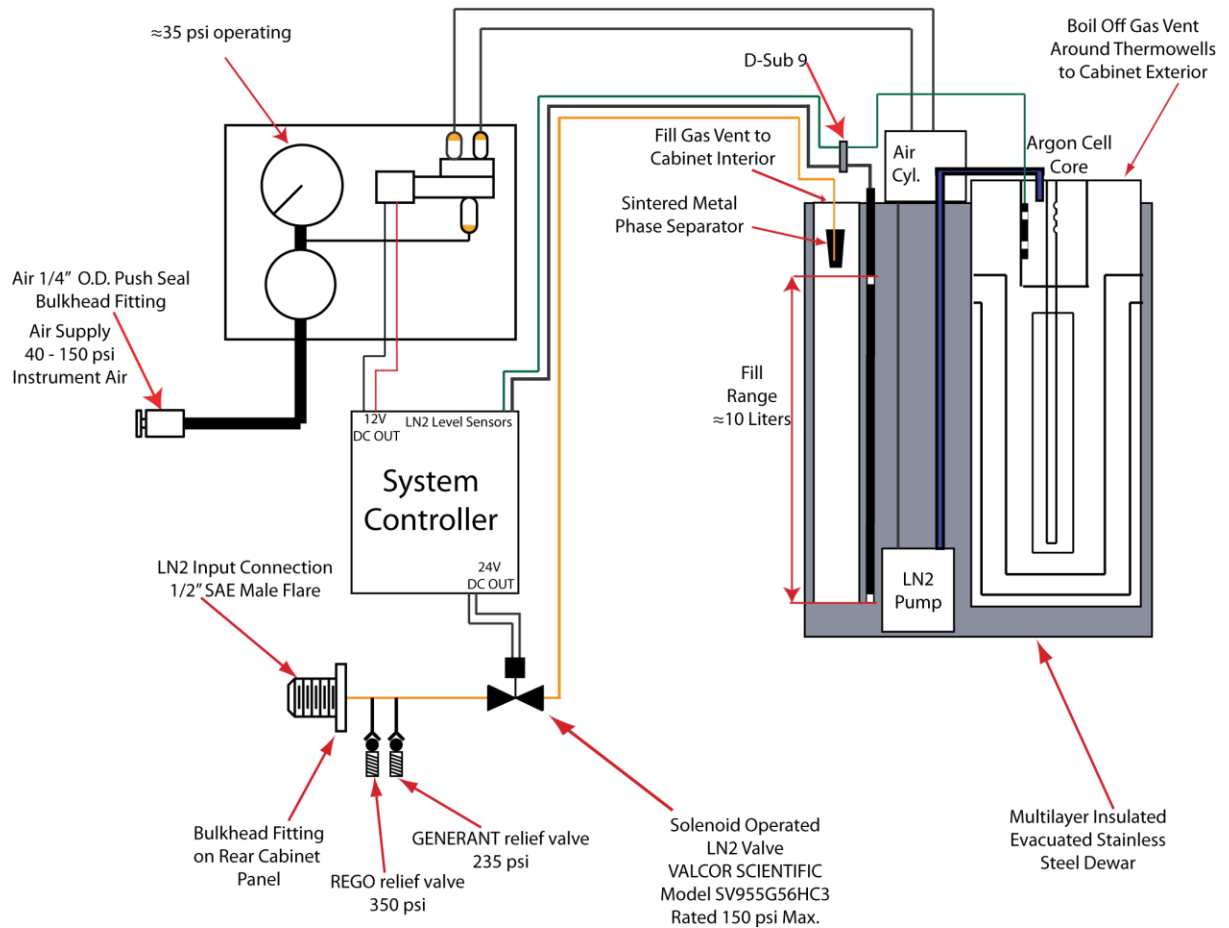
1/4" Push-To-Connect
Air Fitting

Communications Port
(IEEE-488 shown)

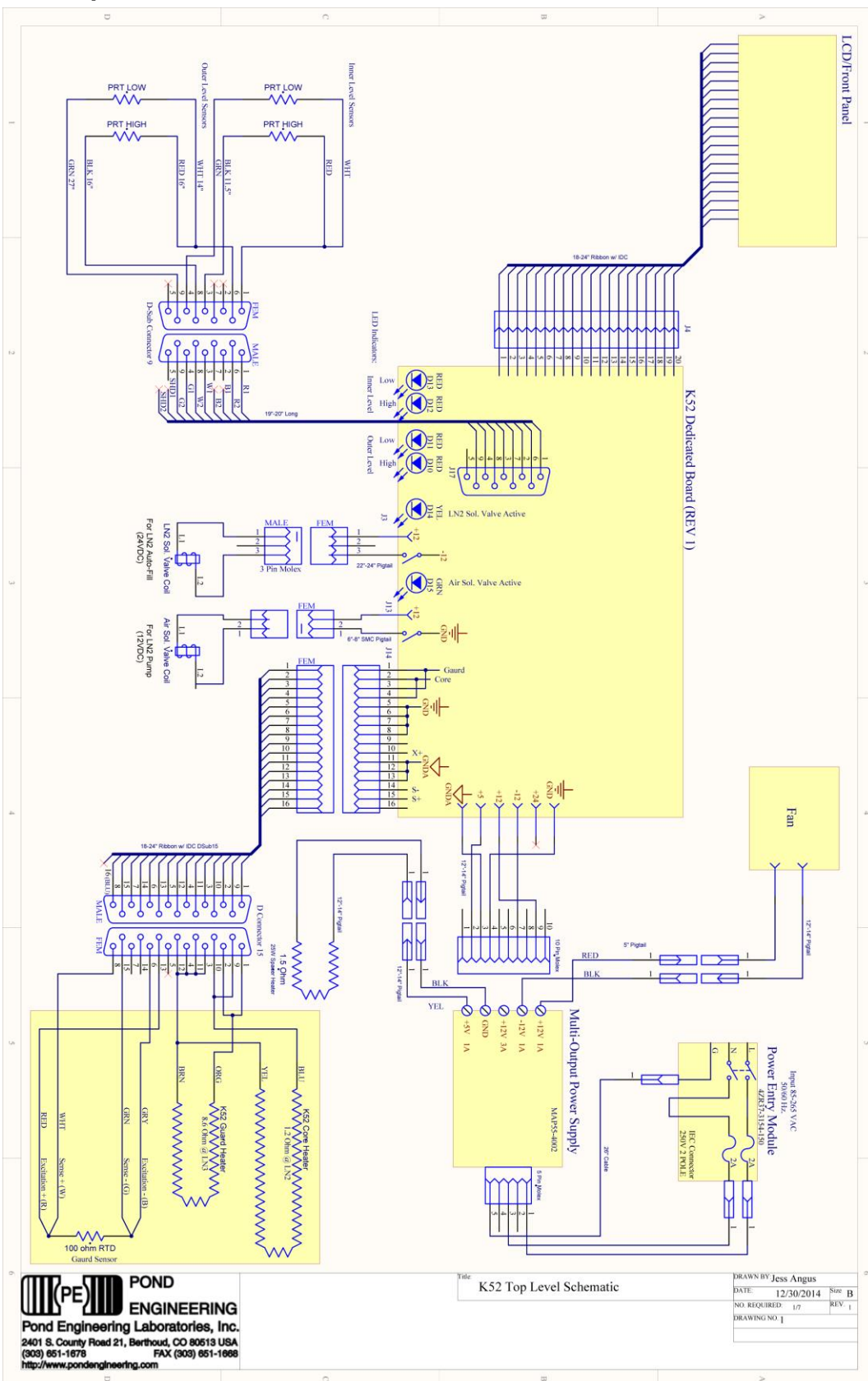
Power Entry Module

For push-to-connect fitting:
To insert hose, press into fitting
To remove hose, press on bulkhead and gently pull on hose

5.3 LN2 Control System Block Diagram



5.4 Top Level Schematic





POND ENGINEERING

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Factory Calibrations:

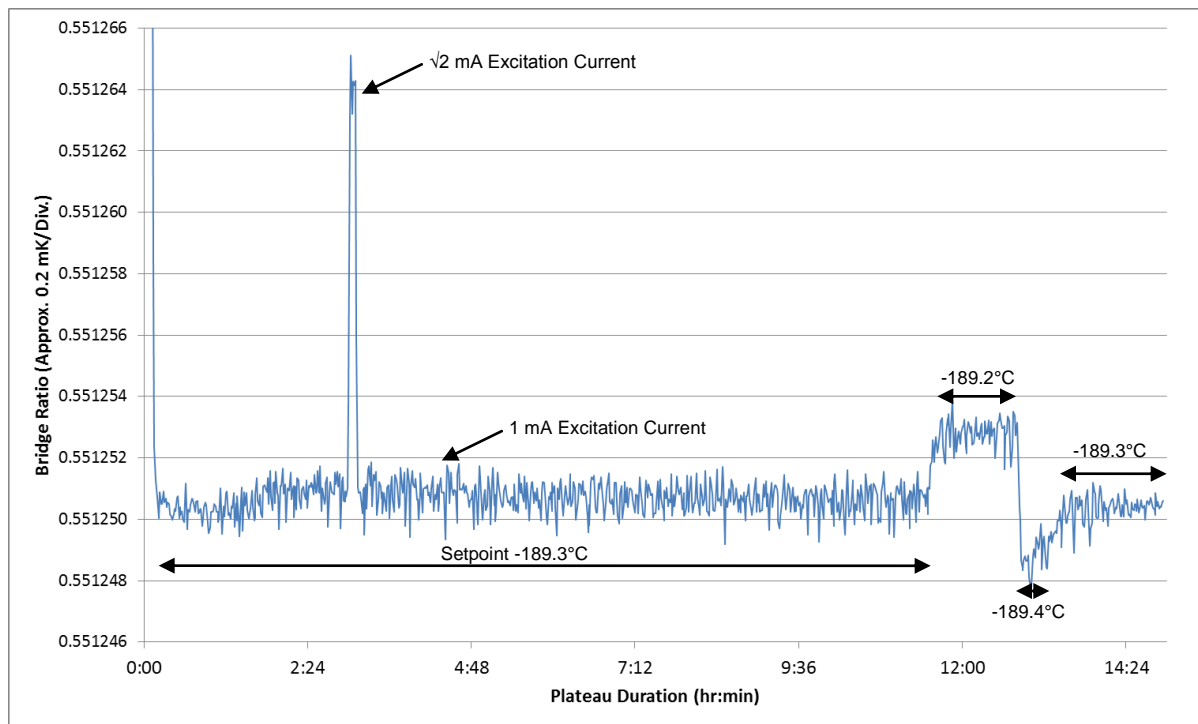
RTPW:

Coefficient A:

Coefficient B

[illegible]

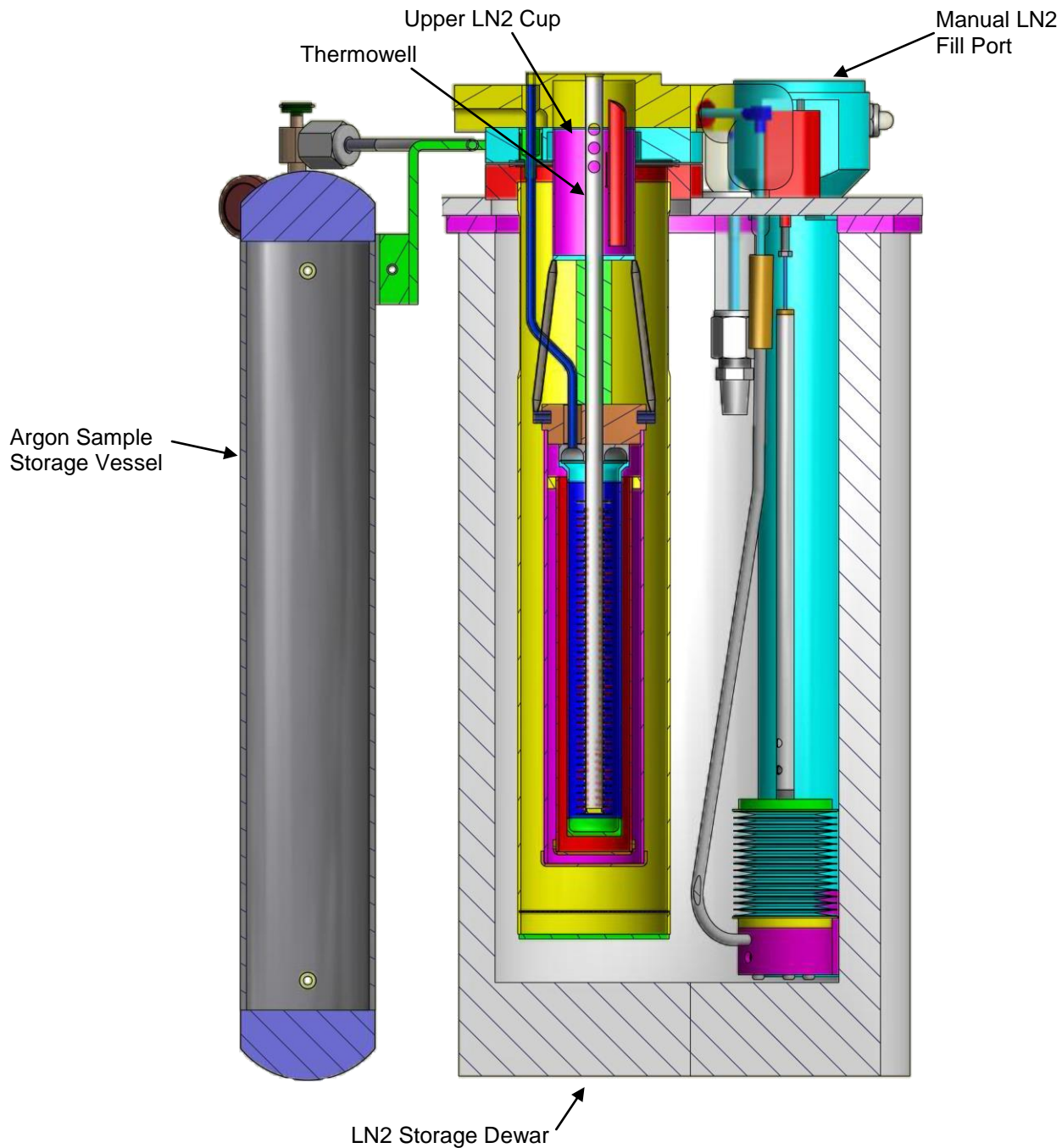
6. Graph of a Typical Plateau



YSI 8167 vs 10 Ω standard in oil as measured by ISOTECH Micro-K 100

7. Cross-Sectional View of the K52

Following is a cross-sectional view of the Argon Triple Point Cell to assist the user in understanding the operation of the system.



8. Fixed Point Cell Certificate of Sample Purity

The following page contains the Fixed Point Cell Certificate of Purity.