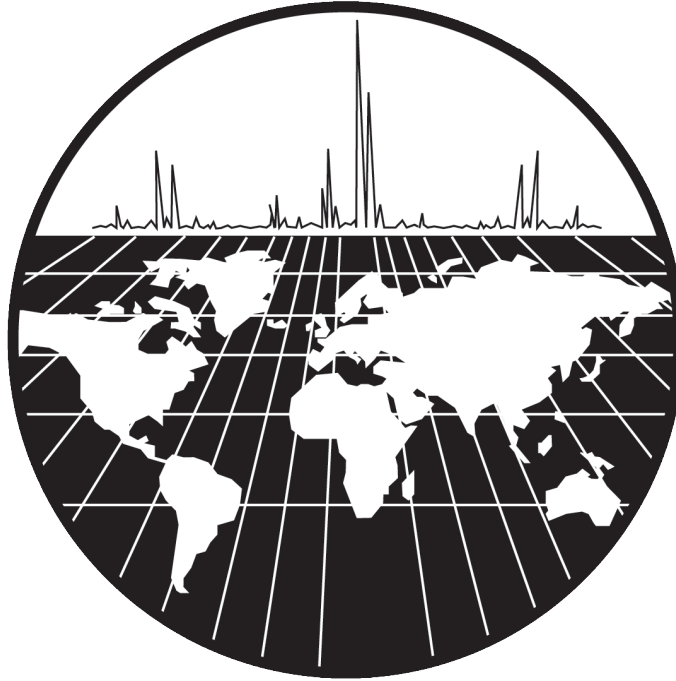


High Temperature Direct Probe Manual

Temperature Controller Model PC-2 (v201411)



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Specifications

Probe

Maximum temperature: 800°C.

Maximum heating rate: exceeds 300°C/min.

Maximum cooling gas pressure: 60 psi.

Flared sample vials recommended: 1.7mm O.D. x 12mm long. SIS part # S17A

Temperature Controller

110 volts AC, 10 amps maximum.

Warnings

WARNING - Probe tip can be heated to 800°C. Be careful when handling probe to avoid injury.

WARNING - Cooling Gas must be on during the entire operation of the probe. Maximum cooling gas pressure is 60 psi.

WARNING - Do not use combustible gas for cooling the probe.

WARNING - Do not remove a probe from the mass spectrometer while it is hot or damage to the inlet seals may occur.

NOTE - The information contained in this document is subject to change without notice. Scientific Instrument Services makes no warranty of any kind with regard to the material contained in this manual, including, but not limited to, the implied warranties of usefulness and fitness of the equipment and techniques therein described for a particular purpose.

Warranty

The probe and controller are warranted against defects in material and workmanship for a period of ninety days from the date of shipment from Scientific Instrument Services, hereafter referred to as the company. The companies liability on this equipment is limited to the cost of correcting the defect in the product. In no case shall the company be liable for consequential or special damages. The company will not correct, under warranty, defects caused by the buyers negligence. The company does not guarantee or warrant the product for any particular purpose. The companies warranty shall end 90 days after shipment.

Operating Procedure for High Temperature Direct Probe Temperature Controller Model PC-2

INTRODUCTION

The Scientific Instrument Services Direct Probe Temperature Controller is designed to operate with the direct probes designed and manufactured by Scientific Instrument Services. Two modes of operation are permitted with the controller: (1) ballistic heating of the probe to a set temperature and (2) multistep temperature programming of the probe from an initial temperature to a higher final temperature via a predetermined ramp rate. In the ballistic mode of operation, the direct probe will heat up rapidly (in excess of 300°C degrees per minute). In the temperature program mode of operation the probe temperature, it is typically ramped at rates from 1 to 200 degrees per minute up to the final temperature.

CONTROLLER DESCRIPTION - FRONT PANEL

Switches - There are two rocker type switches on the front panel of the controller. See Figure 1. The main power switch turns on the power to the controller. The heater switch allows power to the probe heater. The light under the heater switch shows when power is *actually* flowing through the heater (which will blip on/off as the temperature falls below or above the set-point).



Temperature Controller - The temperature controller displays the actual temperature of the probe at top and the set temperature at bottom. Normally these two values are similar unless the heater switch is turned OFF. The two arrow buttons on the temperature controller enable changing the temperature set-point for the controller. Further detail on this module is provided later in this manual.



Remote Start - The remote start pushbutton switch on the front of the controller is used to remotely start the data system of the mass spectrometer for data collection via a contact closure on the back panel. This switch can be activated at any time prior to, or during the operation of the probe.

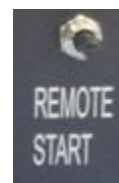




Figure 1: Front Panel

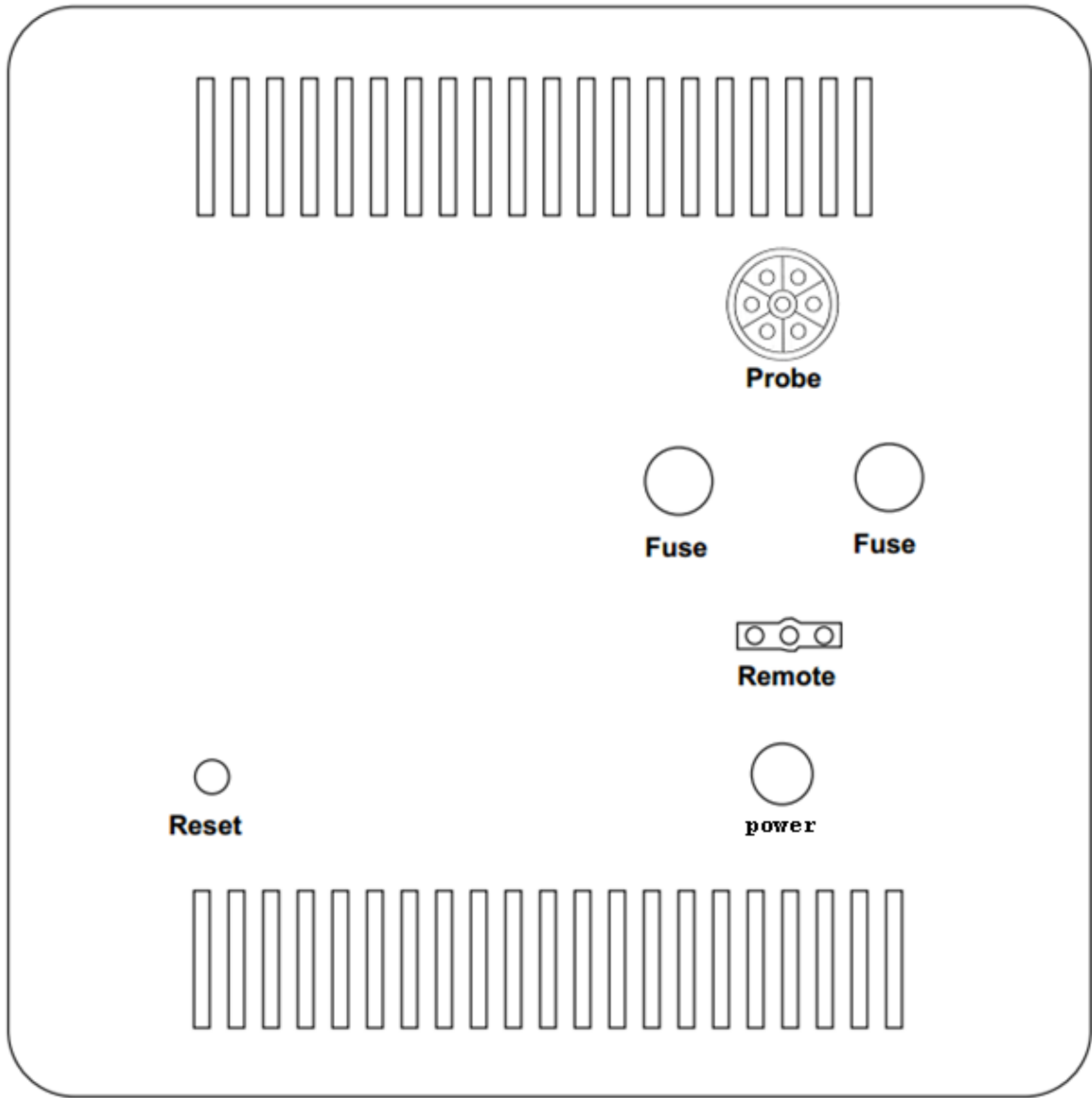
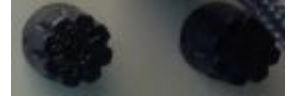


Figure 2: Rear Panel – diagram and picture



CONTROLLER DESCRIPTION - REAR PANEL

Fuses - There are two fuses located on the rear panel of the controller (for main power and heater). See Figure 2. The fuses are both 10 amp rated fuses. When replacing fuses be certain to use the same rated fuses.



Probe Connector – The top-most circular connector on the rear panel of the controller is for insertion of the cable which is connected to the probe (Part # C100)



Remote Start Connector - The bottom connector is for the remote start cable which is included with the probe (Part # 782012). Only the left two pins (contact closure) are used.



Power Cord - The power cord must be connected to a grounded power outlet of the correct voltage for the controller. This is 110 volts AC, unless the controller was specifically ordered for 220 volts.



Reset Button- There is a thermocouple in the shaft of the probe which acts as a safety shut-off switch should the probe shaft exceed 200°C. If the cooling gas is not on during the entire operation of the probe, especially at high temperatures, the shaft temperature will exceed 200°C and the controller will shut down power to the probe heater. Cooling gas to the probe should be turned on immediately to cool the probe shaft. After the probe has cooled the controller must be reset by pushing this reset button.



Schematic

A conceptual electrical schematic of the PC-2 probe is below.

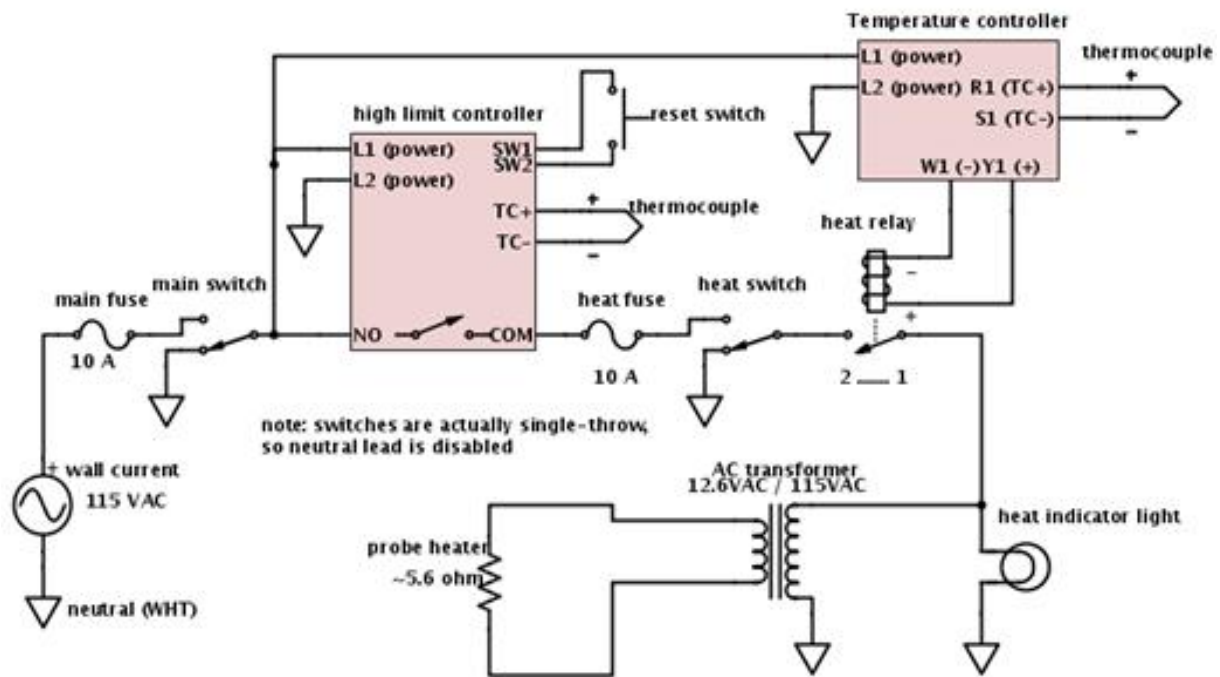


Figure 3: Conceptual schematic

The user interface mainly consists of the Watlow control unit on the front panel (Figure 1), which monitors the temperature of the probe with a thermocouple and applies power to the probe heater. Both front panel switches must be turned ON for heating. There are also internally fuses and a high limit controller (cut-off) for added safety. The heat indicator light on front turns ON when power is sent to the probe heater.

INSTALLATION

To prepare for operation of the probe, plug the controller into an outlet of the correct voltage and connect the cable from the controller to the probe. This cable must be connected or an error message will appear on the controller. If you plan on using the remote start switch on the controller to start the data system you must install the remote start cable. Once these connections are made you are ready to begin operation of the probe. The following goes into detail concerning the two modes of probe operation.

BALLISTIC MODE OF OPERATION

- (1) Turn the Main Power switch ON the controller. Leave the Heater switch OFF.
- (2) Use the two arrow buttons on the temperature controller to set the desired final temperature in the lower display.
- (3) After the probe has been properly inserted into the mass spectrometer and into the source, begin the mass spectrometer scanning operation, via the remote start on the temperature controller or at the data system, and turn ON the Heater switch on the controller. The probe will rapidly heat up to the set temperature at a rate in excess of 300 degrees per minute, and will hold at that temperature until either the heater switch is turned OFF or the set temperature on the lower display is changed via the temperature arrow buttons. These values can be changed while the system is running.
- (4) When finished, turn the Heater switch OFF on the controller. Allow the probe to cool below 100°C before removing completely from the mass spectrometer.

TEMPERATURE PROGRAM MODE OF OPERATION

A multistep temperature program can be used to control the heating and cooling of the direct probe. Normally only one temperature program rate is used to heat the probe, however several temperature program rates and steps are permissible with this system. A typical one rate temperature program is normally comprised of five steps as follows:

- STEP 1 - INITIAL HOLD - hold the current temperature for a set time (HR/MIN/SEC)
 - STEP 2 - TEMPERATURE RAMP - ramp probe temperature at a certain °/min to a final temperature.
 - STEP 3 - TEMPERATURE HOLD - hold final temperature for a set time (HR/MIN/SEC).
 - STEP 4 - COOL DOWN - reset to initial temperature.
 - STEP 5 - END - End the program and initialize it for the next sample.
- Probe temperatures can be set anywhere from 0° C to 800° C maximum.

A typical probe ramp will be as follows: An initial temperature will be room temperature (25 °C) which is held for 5 seconds after which the probe will be programmed from room temperature up to 600 °C at a rate of 200° C/min. After the final temperature of 600 °C is reached, the probe will be held at this temperature for 2 minutes and will then be cooled back down to room temperature to get ready for the next sample.

PROGRAMMING THE TEMPERATURE CONTROLLER

Details on the Watlow EZ-Zone® PM PID (model PM4R2CA-AAAAAAA) are provided separately in the Watlow documentation, which is online [3]. In particular see the User Manuals [1,2]

[1] <http://www.watlow.com/downloads/en/manuals/pmpid.pdf>

[2] <http://www.watlow.com/downloads/en/manuals/PM%20Quick%20Start%20Guide.pdf>

[3] <http://www.watlow.com/products/controllers/ez-zone-pm-controller.cfm?famid=19>



Figure 4: Temperature controller user interface (left) and other controls (right).

In typical usage, the up/down arrows control the set-point temperature. The red EZ1/EZ2 buttons optionally provide quick access to pre-programmed profiles. The infinity key returns to the home page or goes back. The blue cycle button advances through parameter settings. **There are many more functions though, so see the Watlow manual for details.**

The HEATER switch on the front panel overrides the temperature controller. Heating will only occur if the HEATER switch is ON.

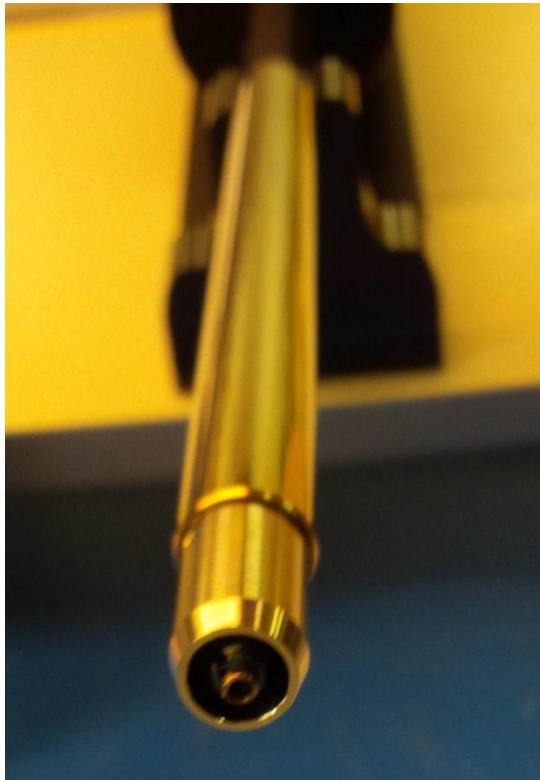
DIRECT PROBE DESCRIPTION

Figure 5 shows the direct probe including an enlarged cut-away of the tip of the probe. The probe is designed such that the heater is located at the tip of the probe and is in direct contact with the sample vial. This design provides the very fast ramp rates available on this probe. The spring clip, as shown in the drawing, holds the sample vial in place during the analysis.

A detailed drawing of the bottom of the probe handle is shown in Figure 5. The following is a description of the fittings and valve on the back of the probe handle.

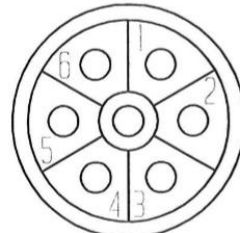
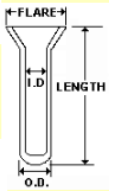
Probe Cable Connection - This is the electrical connection for plugging in the probe cable from the temperature controller.

Cooling Gas Hookup - This is a 1/8" quick connect fitting for connection of the cooling gas line. The quick connect fitting has an automatic shut-off feature which will stop the cooling gas flow when the fitting is disconnected. Typically compressed air is used as the cooling gas for this probe; however, nitrogen can also be used. In no case should combustible gas be used to cool the probe. The probe can also be cooled using a liquid cooling fluid. The unthreaded nipple on the back of the probe handle is the cooling gas exit. Cooling gas must be circulating through the probe during the entire probe operation.



S17 Series - SIS Probe Flared Type Sample Vials

O.D.	1.7 mm
I.D.	0.8 mm
Length*	12.0 mm
Lip	1.5 mm
Flare	3.0 mm



- 1 - Heater Load
- 2 - Heater Load
- 3 - K+ Yel thermocouple
- 4 - K- Red thermocouple
- 5 - J+ White thermocouple (high limit)
- 6 - J- Red thermocouple (high limit)

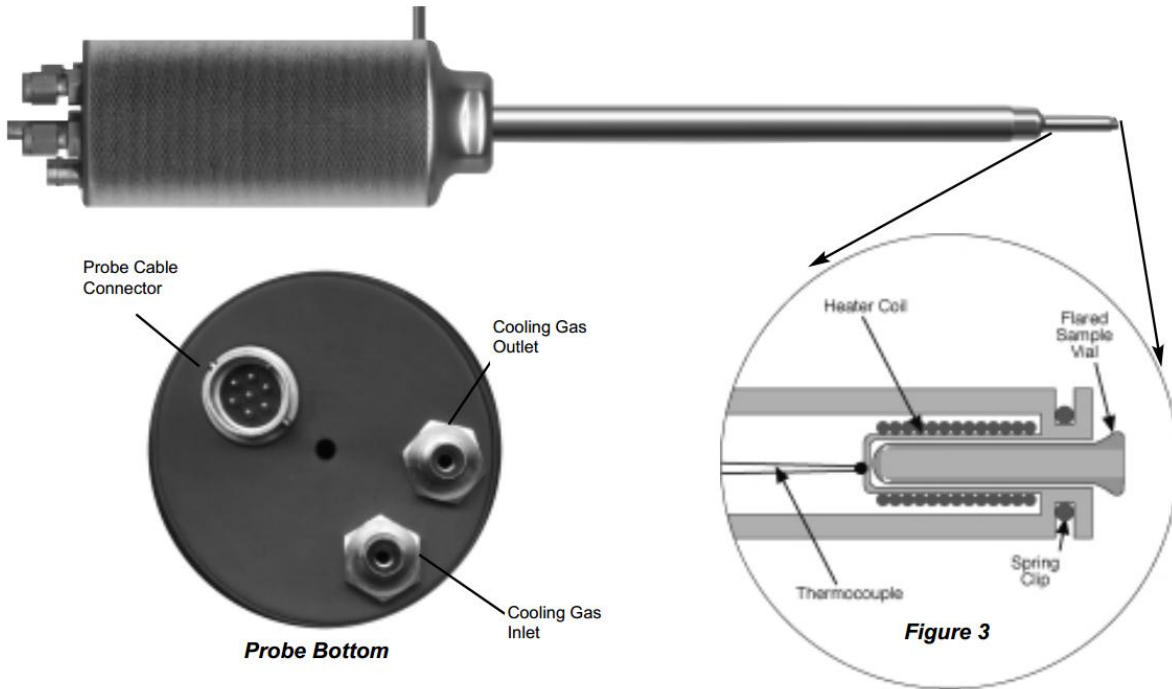


Figure 5: Pictures and diagrams of probe, connections, and vials.

PROBE OPERATION

After hooking up the cable from the temperature controller to the probe and hooking up the cooling gas, the probe shaft should be cleaned by wiping with a cloth using methanol. Load the sample into the sample vial and then insert the sample vial into the tip of the probe. Care should be used in determining sample size so that the mass spectrometer is not overloaded and to minimize potential source contamination. The probe is now ready for insertion into the mass spectrometer and for subsequent analysis.

1. Insert the probe shaft into the probe inlet until the probe reaches the first stop at the front of the inlet. Stop—do not insert the probe any further or damage could occur.
2. Open the pump-out valve on the bottom of the probe inlet to allow rough pumping of the inlet. Wait about three minutes for the probe inlet to be pumped out and then close the pump-out valve.
3. Continue to insert the probe by releasing the ball on the bottom of the inlet. Stop—do not insert the probe any further or damage will result.
4. Slowly open the probe port valve until it is fully open.
5. Continue to insert the probe until it reaches the ion source. Insert the probe slowly until it seats against the outside of the ion source.
6. Turn on the cooling gas.
7. After the analysis is complete, remove the probe about 1/2". Do not remove the probe further while it is hot or damage to the seals could occur.
8. After the probe has cooled to less than 100°C, carefully remove the probe until the stop.
9. Close the probe port valve completely.
10. Remove the probe from the inlet.