1600° C General Use Furnace

Manufactured by

A.S. Scientific Products

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Contents:

Standard I.L.L. 1600°C Furnace

I. General Description

- A top loading vacuum furnace with full sample visibility (360° in scattering plane, ±20° degrees out of plane) manufactured by AS Scientific Products Ltd
- Radiative heating via a niobium foil element (2 concentric cylinders 0.05mm thick) driven by a high current/SCR power supply
- 8 thermal shields (0.05mm Nb) and a water cooled aluminum outer vacuum can (1.2 mm thick at neutron window)
- Active Heating/Sample Zone : 4.2 cm diameter x 10 cm length
- Temperature Range: Ambient to 1600° C
- Temperature Control: Auto-tuning PID Type (Eurotherm Model 818) with high current SCR power source
- Temperature Sensors: type W5 thermocouple (tungsten rhenium alloys: Re5% vs. Re26%)
- Interlocked alarm system for pressure, water flow, and temperature

II. Safety Issues

1. The furnace must be operated under high vacuum (less than 10-4 Torr).
2. **KNOW YOUR SAMPLE!** Users must verify that their sample will maintain low vapor pressure ($< 10^{-5}$ Torr) and not react with its surroundings at high temperature.
3. The furnace requires high vacuum and proper water flow for safe operation. There are two safety interlock systems: One is for over-temperature and is controlled by the small temperature reader on the upper right of the control unit. The vacuum gauge and the water-flow switch share the other. Familiarize yourself with these circuits and verify their operation. The furnace will not operate if either of these interlocks is not functioning.
4. Set Appropriate Current Limits. If using the manual ramping, do not jolt the furnace with high current spikes. Slowly increment the output limit setting and/or ramp your set point for smooth operation.

III. Sample Preparation

1. Off-Line Testing:
   All samples and holders should be tested off-line in order to ensure that the sample, or sample can, will survive the maximum temperatures in your experiment without reacting, bursting, etc. If possible, test your sample before arriving at NIST. For now, the AS1600C furnace itself may be used outside of the neutron beam areas and with continuous monitoring and safety precautions for off-line tests.
2. Sample Holders:
   The sample probe terminates with a threaded 8 mm 1.25 pitch (M8) stud. Since there are currently no standard sample holders for this furnace, researchers must design (NIST assistance is available) holders compatible with our probes. Ventilated (*i.e.* non-pressure building) crucible-type holders made of niobium or platinum are recommended. Any sealed sample holder must be tested (off-line) at the maximum desired temperature before loading into the furnace to ensure that an over-pressure burst will not occur on the beam line.

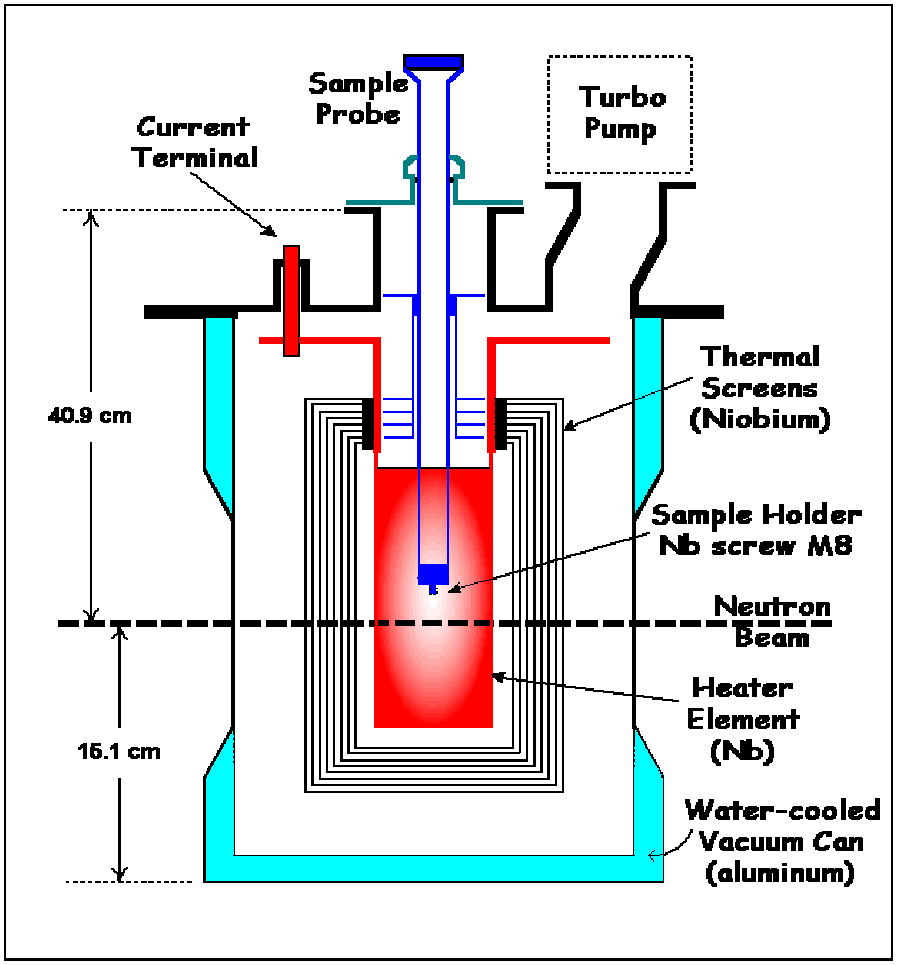


Figure 1. AS1600 C Furnace body cross section schematics

3. Sample Geometry:
   a. Note that the beam center is 40.9 cm below the top of the sample well, and the probe height is adjustable to accommodate a wide range of sample sizes.
   b. Maximum Sample Diameter=4.2 cm
   c. Maximum Sample Height = 12 cm
   d. Your probe/sample length must be adjusted so that it DOES NOT TOUCH THE HEATING ELEMENT OR THE BOTTOM OF THE SAMPLE WELL!

IV. FURNACE SET UP

Required Connections

After the furnace is mounted on the sample table:

1. Connect the cooling water lines: The flow direction is important! The flow interlock (white fixture connected to cooling hose) must be connected to the water RETURN
2. Open cooling water supply/return valves
3. Connect the flow sensor cable (orange w/black connector) to the back of power supply
4. Connect the vacuum hose from rough pump to the solenoid valve located next to the turbo pump
5. Connect the Penning Pressure Gauge cable (Lemo connector w/locking nut) to Penning Pressure sensor located on the furnace
6. Connect the gray 8-Pin cable labeled “Turbo Pump” to the turbo pump
7. Connect the gray 6-pin sample probe connector to the sample probe
8. Connect the braided ground wire to turbo pump. This is the power supply ground
9. Connect the high current furnace leads from the power supply cabinet to the furnace current terminals (notice that these cables MUST be connected to the strain relief bracket located on the furnace; furthermore, these cables MUST be suspended from an extension arm by the cable sling)
10. Apply the tension relief brackets to the high current leads
11. Three 110 AC cords require connection: Turbo controller on power unit, rotary pump on the bottom of the furnace cart, and the solenoid valve on the turbo pump
12. Connect the main 210 Volt cord for the power supply cabinet
13. Finally connect the RS232 remote computer control cable to the proper cable connection

Alarm Reset at Start-Up

1. Turn breaker “CB 1” and “MASTER” switch on.
2. Press the "Water/Vacuum Fault" button. Orange light goes off if water and vacuum are OK.
3. Press the "Overtemp Reset" button. Flashing AL1 goes off if the temperature reading is OK.

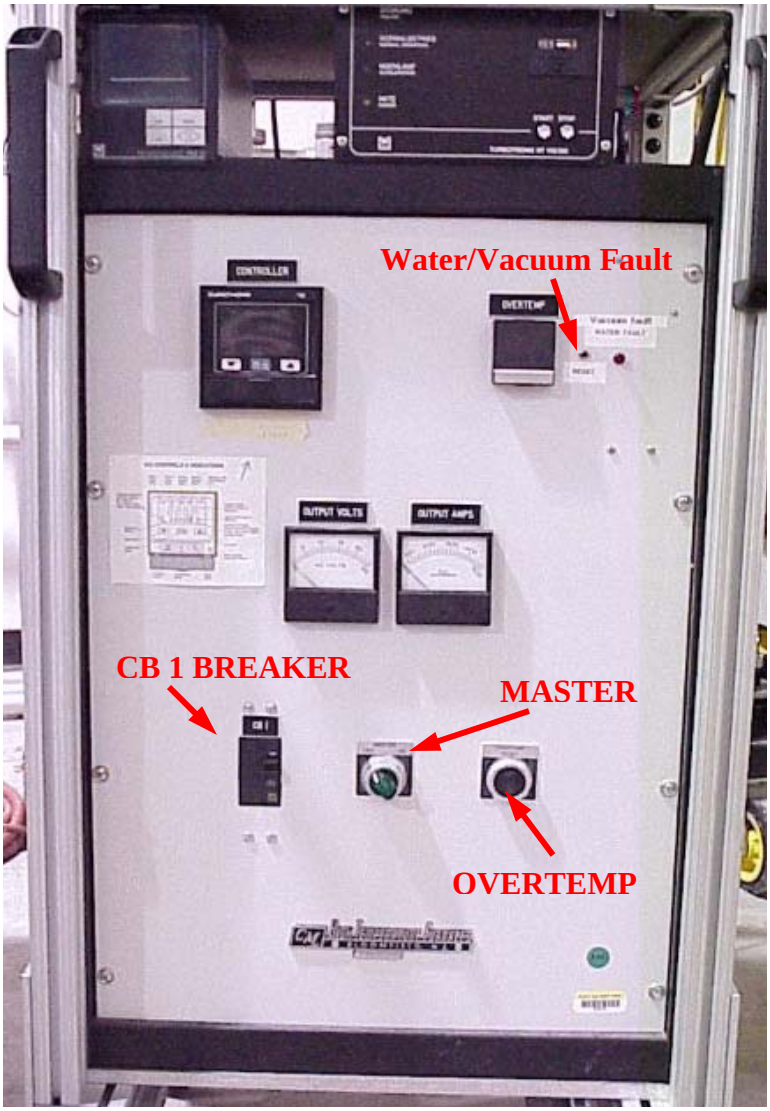


Figure 2. Furnace power cabinet

V. Controller Operation (Eurotherm 818)

The NCNR instrument control program (ICP) is set up for remote set point changes with proper ramp rates and appropriate power output automatically invoked. The computer control set up can be invoked by icp with the “tdev” command. This is the method to be used for all users; nevertheless, some important front-panel operations are outlined below for manual adjustment of controller functions.

- "SHORT SCROLL" refers to quick presses of the scroll button  , which toggle the lower display between SP and OP (set point and output) status.
- "LONG SCROLL" refers to pressing and holding  until the full parameter set {Pr, SP1, SP2, ... , Sbr } begins to cycle. Note that a flashing dot indicates that a parameter may be changed using the up/down arrow keys.

- Operation Mode
  - There are two modes of operation for this controller, manual and automatic, which can be chosen by pushing the center button with a hand on it.
  - Manual mode allows you to set a heater current output that will stay constant. Only experienced users should use this mode and care must be taken not to burn up the heater element.
  - Automatic mode allows you to enter a set point and control at that temperature. This is the normal mode of operation.
- Set Point
  - SHORT SCROLL (if necessary) to “SP” mode, press arrow keys to change value (hold arrow key to accelerate change).
- Output Limit
  - LONG SCROLL to the "Hi" parameter, immediately press the up or down arrow once to display present setting [% full scale]
  - Press up/down arrows as needed to adjust setting
  - Press scroll to accept value (press repeatedly to scroll back to the default display, or wait ~60 seconds)
- Refer to the table at the right for proper settings. When heating from room temperature, increase the hi setting gradually (i.e. DO NOT SUDDENLY DUMP MAXIMUM CURRENT INTO THE FURNACE)

Tmax [C]	"hi" setting
500	15
700	18
900	23
1100	29
1300	38
1500	50

- Displaying & Changing the Ramp Rate
  - LONG SCROLL to "Pr", press arrow to display present value (rate in degrees/hour)
  - Press arrow again to adjust
  - Press scroll to accept and continue scrolling until you return to the default display

  Note: The short scroll parameter "SP" is equivalent to the long scroll parameter "SP1". When ramp is active, SP/SP1 is the ramp target or endpoint. Otherwise, SP/SP1 is the static set point.

- Activating a Ramp
  - Press RUN/HOLD key until the display indicates RAMP (this changes to "E" when ramp is complete). The set point will jump to the present temperature and immediately begin ramping to the target (SP/SP1).

VI. Over Temperature Alarm

There are three keys along the bottom of the alarm unit (down arrow, scroll, and up arrow) and a "secret key" above the middle digit at the top of the display.

- View Alarm Setting
  - The scroll key cycles through three fields (temperature unit, AL 1, and present reading). When “AL1” is selected, press the up or down arrow key to view the present alarm setting in the specified temperature unit (default = 1300° Celsius)
- Change Alarm Setting
  - Press center button until “degrees C” is shown
  - Press the secret key (display now reads "ConF")
  - Press scroll again to cycle through the long parameter list (see table below)
  - Use the up/down arrow keys to change the value of AL1 if necessary. DO NOT CHANGE ANY OTHER PARAMETERS.

Parameter	Default
ConF	6023
id	92
AL 1	1300
Hy 1	10
OFSt	0.0
Line	60

VII. Sample Changing and Furnace Shut Down

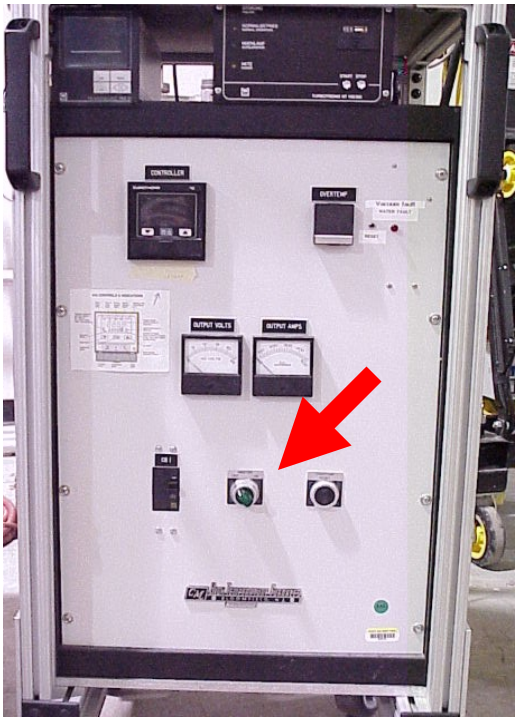
- After cooling the furnace to room temperature the turbo pump can be stopped and left to run down for approximately 10 minutes.
- ***NEVER OPEN THE FURNACE WHILE HOT.***
- Once the turbo has run down valve off the rough pump and turn it off. Do not vent the furnace vacuum space through the rough pump.
- Only then the electrical connections, as described in the setup, can be undone.
- To release the vacuum, slowly open the valve connected to the sintered bronze filter and bring the pressure up to atmosphere as slowly as possible (at least 5 minutes). This is vital to extending the life of the heater element and heat shields.
- At this point the furnace is completely shut down and the sample stick can be removed.

VIII. UGeneral Use Furnace Emergency Shutdown Procedure

PROCEDURE:

TURN POWER OFF TO FURNACE (Turn MASTER switch located on the furnace control rack to off position)

General Use Furnace



Furnace Control Panel detail

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