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GF 120
GRAPHITE FURNACE ATOMIZER

Revised December 2000

Aurora
Instrument

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THE MODULES OF THE GF 120 CONTAIN ELECTRICAL CIRCUITS, COMPONENTS AND DEVICES THAT OPERATE AT HIGH VOLTAGES, WHICH CAN CAUSE SERIOUS INJURY OR DEATH.

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1. Introduction

1.1 Congratulations:

Congratulations on your new Graphite Furnace Atomizer. Your GF 120 system represents the state of the art in analytical instrumentation and has been designed to provide your laboratory with unmatched analytical performance.

1.2 Warranty:

This instrument comes with a one year warranty and a lifetime of application support from Aurora Instruments Ltd.

The warranty will cover, at Aurora Instruments' service center in Vancouver, Canada, all parts and labor necessary to maintain the equipment at the original manufacturer's specifications. If field service is necessary, a separate purchase order to cover the cost of travel and associated expenses will be required. Alternatively, all inclusive warranty programs are available from Aurora Instruments. Please contact your local sales representative for details.

2. Safety

2.1 In General

The safety practices outlined in this section are designed to create a safe environment for the analyst when using the GF 120 instrument. **All safety instructions must be read prior to operating the instrument.**

2.2 Personal Safety

Operation of the GF 120 instrument involves the use of compressed gases, high temperature and hazardous materials including corrosive fluids and flammable liquids. Explosion and fires hazards exist which have the potential to cause death or serious injury to personnel and severe damage to equipment and property. The consequences of unskilled, improper, or careless operation of the instrument can be serious.

Operators and service personnel must be aware of all hazards, know how to recognize potentially hazardous conditions, and know how to avoid them. Specific hazards and potential hazards are described in the following sections.

2.3 Electrical

> **WARNING!** The modules of the GF 120 contain electrical circuits, components and devices operating at dangerous voltages. Contact with these circuits, components and/or devices can cause death, serious injury or electrical shock.
> Good electrical grounding is essential to avoid potentially serious shock hazards.
> Application of the wrong supply voltage can create a fire hazard or a potentially serious shock hazard, and could seriously damage the instrument.
> DO NOT connect the instrument to the main power supply until you have made sure that the voltage selector is correctly set for the power supply of the specific outlet in your laboratory to which the instrument will be connected.

Before any power is supplied to any of the GF 120 components ensure that all connections are secure.

Always power up the modules in the following order:

Main power switch followed by the Computer

2.4 Vapors and Fumes

WARNING! Heat, vapors and fumes generated by the high temperature of the GF 120 can be hazardous, toxic or otherwise injurious to personnel.

A suitable exhaust system is required for safe operation of the GF 120 instrument. The fume extractor/collection hood should be exhausted to the exterior of the building using a fan and ducting. The exhaust system should have a flow rate of at least 6 m^3/min and local building and worker safety regulations must be obeyed. Regular inspection of the exhaust system should be carried out.

2.5 Compressed Gas Cylinders

Compressed gas cylinders provide at least THREE potential hazards:

1) Rupture of any compressed gas cylinder or the high pressure region of the regulator may produce flying shrapnel or rocket like acceleration of the cylinder such as to cause injury to persons or damage to equipment and property.
2) Venting of any gas, except air, to the atmosphere (i.e. your laboratory) can cause death or serious injury by asphyxiation, anesthetic effects, or poisoning.
3) Explosion or fire can result from the improper storage or use of flammable gas cylinders. Storage and use of compressed gas cylinders must be in strict compliance with local regulations. Some additional guidelines are:

- Store cylinders in a vertical position.
- Secure cylinders to an immovable structure or a properly constructed cylinder stand.
- Move cylinders only on a trolley specifically designed for that purpose.
- Adequately ventilate the area in which cylinders are stored and used.
- Keep cylinders in a cool place.
- Ensure that all cylinders are clearly labeled and return any cylinders that are not clearly labeled.
- Check the gas transfer line prior to connecting any cylinder to the instrument.
- Use only the proper regulators, connectors, fittings, and hoses.
- Always shut off the gas supply at the cylinder when finished for the day.
- Always switch the exhaust fan on before opening or utilizing compressed gases.

Only high purity Ar should be used as the graphite furnace purge gas.

Always use approved regulators and hose connectors. Never try to refill cylinders.

2.6 Argon

Store and use with adequate ventilation at all times. Close valve when not in use and when empty. Regularly check transfer lines and connectors for potential leakage.

The argon pressure should be set to approximately 30 psi (200 kPa) for normal GFAAS operation. The maximum pressure is 40 psi (265 kPa).

2.7 Safety Placards Found on the GF 120



OPEN FLAME DANGER

⚠WARNING

- Open flame under hood.
- Touching burner and associated parts can cause burns.
- Do not remove flame cover.



ELECTRIC SHOCK DANGER

⚠WARNING

- Danger of electrical shock.
- High Voltage electrical wires
- Do not remove flame cover.



EXPLOSION DANGER

⚠WARNING

- Explosive gases in use.
- Do not use oxygen gas as a support gas.
- No open flames, in vicinity of heating apparatus.



HOT SURFACE

⚠WARNING

- Do not touch heater chasis.
- Touching cover and associated parts can cause burns.
- Do not remove flame cover.

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3. Installation

3.1 Unpacking

When unpacking the GF 120 and modules please check the packing list to ensure that all the listed items are present. If any discrepancies exist please notify the supplier immediately. If there is any damage to the packaged items please notify the supplier and the shipper immediately.

3.2 Environment

As with other optical atomic spectroscopic instruments, the GF 120 should be operated in a clean, safe laboratory environment. Sample digestion and preparation should be carried out in a well ventilated area away from the instrumentation. The room housing the instrumentation should be air conditioned to maintain constant ambient conditions of temperature and humidity. The instrument should be located in a position where it will be as free from vibration as possible.

Optimal Environmental conditions

OPERATING TEMPERATURE	**15-25°C**
RELATIVE HUMIDITY	**< 60% (NOT CONDENSING)**
COOLING WATER TEMP	**< 20°C**

Bench Space: The area around the GF 120 and components should be kept unobstructed to ensure safety, adequate flow of cooling air and proper ventilation.

> At all times the operator must ensure that the front panel and the back panel of the instrument are kept clear to allow an adequate flow of cooling air to be maintained.

3.3 Power Requirement

GF 120 Model

GF : AC 220 V, 50/60 Hz, 30 A

A 3-wire power outlet with an **EARTH GROUND** connection must be used with the GF 120. The operator must ensure that the supplied AC power conforms to the load requirements of the system.

WARNING! Use of the wrong power supply voltage, improperly wired supply voltage, or omission of suitable grounding can create a fire or shock hazard. Always use a 3-wire outlet with ground connection and ensure that the connection complies with local safety regulations.

3.4 The Computer

The GF 120 is directly controlled by an IBM compatible personal computer via the communication ports COM1 or COM2. The computer requirements are as follows:

- Pentium 166 or higher.
- 32M RAM or higher.
- 20MB of free hard disk space.
- Windows 95/98
- CD-ROM drive
- a video display card with at least 2MB RAM. 4MB RAM is recommended.
- 14" Super VGA (resolution up to 1024×768) color monitor. 15" or up is recommended.

Important: Please set your display card's resolution to **1024 by 768**. To change your video resolution within Windows 95 simply click on the start button, settings, control panel, display, settings and change the display/screen area accordingly.

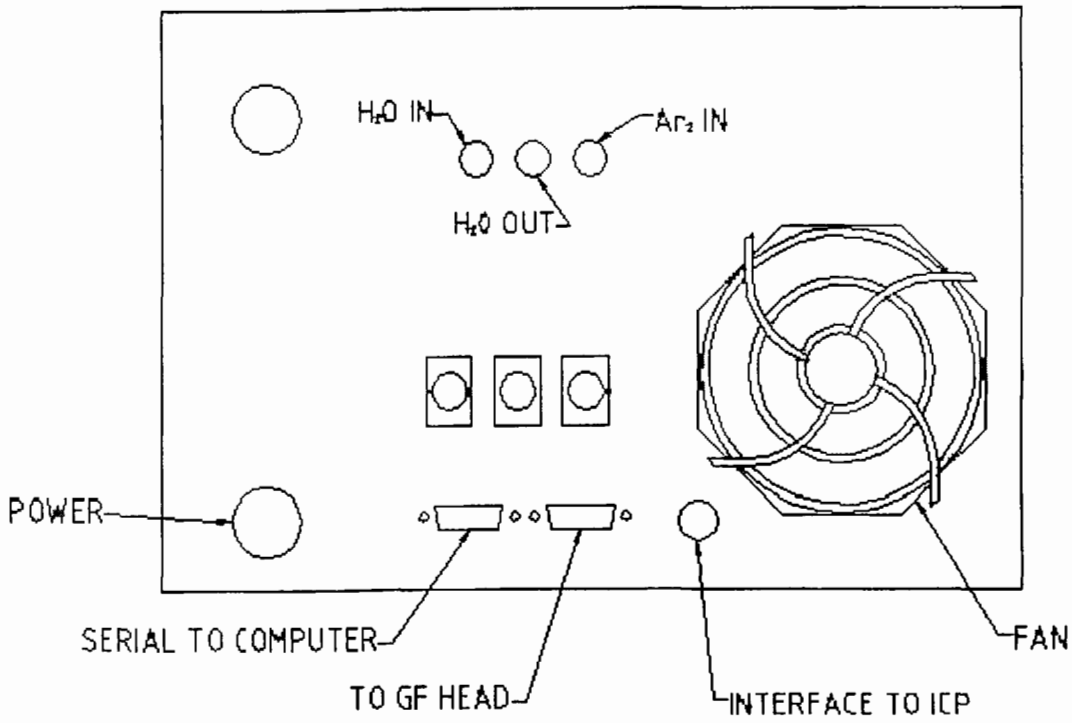
3.5 System Installation

Unpack and assemble the GF Power supply and its peripheral components. Observe the space requirements and clearances for the equipment as outlined previously. Remove all packing materials on the movable components.

3.5.1 Using One-Touch Fittings

All of the water and gas connections to the GF 120 instrument are accomplished using 'One-Touch Fittings'. To attach a piece of tubing to the fitting, simply fully insert the tubing into the fitting and pull gently to lock in place. To remove a piece of tubing, push the flange into the fitting and pull out the tubing.

3.6 Cabling the Instrument



a) Attach the 220V / 50/60 Hz / 30A power imput to back of instrument
b) Attach the in/out tubes for the coolant water for the GF head assembly with the one-touch fittings.
c) The instrument has two 25 pin serial ports on the back, The male cable connects the back of the computer into the 9 pin RS-232 port and the female one connects to the GF assembly.
d) There are 3 optional ports for interfacing to other components (eg. ICP).
e) Connect the argon gas supply tubing to the Ar inlet

.

3.7 Software Installation

Installation

Insert the CD into the CD player. Using the program manager, go to the GF 120 disk, and select setup. Follow the instructions given.

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4. Operation for GF 120 Atomizer

4.1 Start-up

Before you begin, it is very important that all of the gas and water facilities are connected and operational. Failure to do so may result in damage to sensitive hardware components. Under normal operating conditions, the argon gas pressure should be 30 psi (200 kPa). The maximum operating argon gas pressure is 40 psi (265 kPa). For more information about setting up and testing the gas and water connections, please refer to Chapter 3.

Power up the GF 120 by switching on the power found on the back of the instrument. Please ensure that the instrument has fully powered up before starting the software.

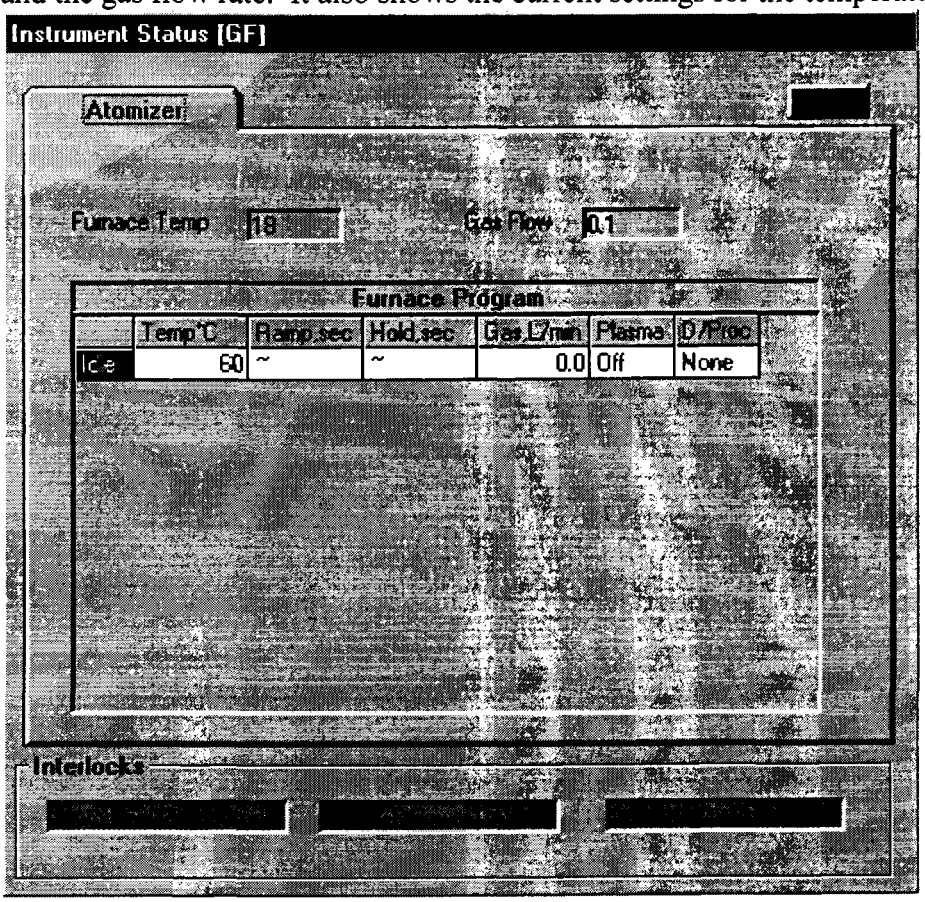
Starting the Software:

Open the GF 120 software by double clicking on the program icon found on your desktop. You should now see the 'Main Window'.

4.2 Software Operation

Instrument Status Window:

This window shows the current status of the instrument including furnace temperature and the gas flow rate. It also shows the current settings for the temperature program.

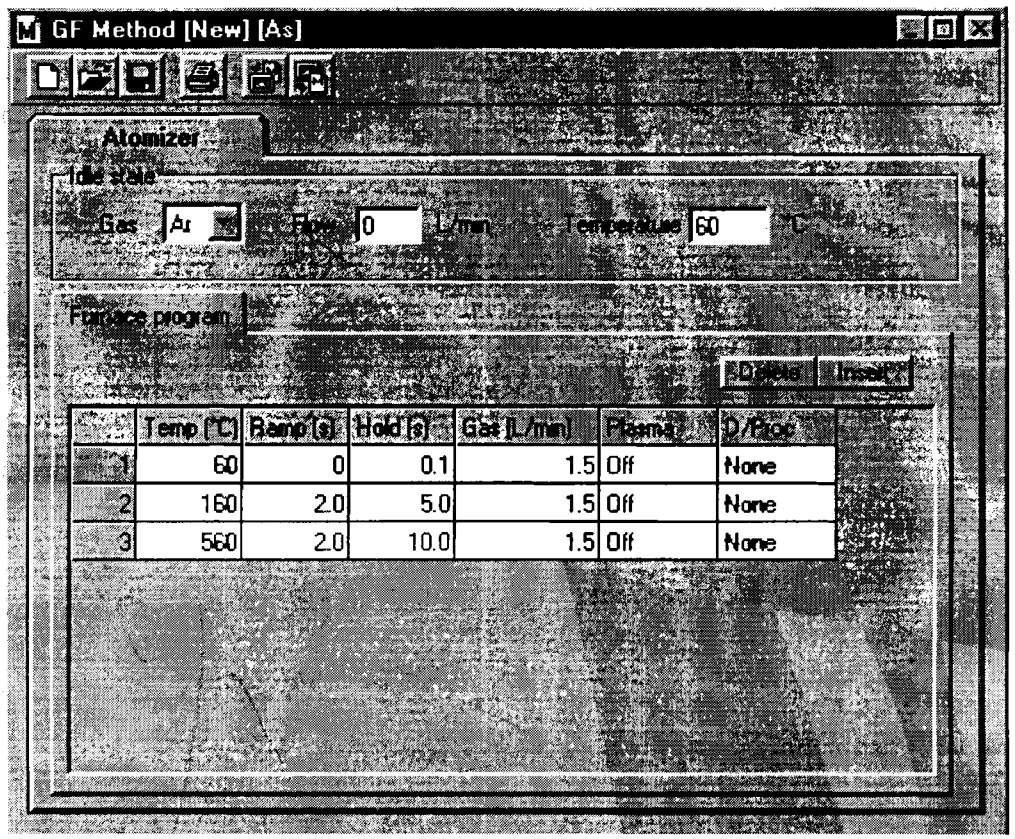


Opening a new or existing method:

Before the user can run the furnace the operator must either open an existing method or start a new method. The instrument will come shipped with some methods pre-installed into the system.

GF Method Window:

This window allows the user to set up the instruments functions. The user can enter the flow rate of the gas and program in the temperature for the heating rate.



4.3.Furnace Program:

The furnace program can be entered into the system through the method window. The user can specify up to 30 different steps. Each step can be programmed separately through the interface. The heating time (Ramp) of the graphite tube to the preprogrammed temperature can be varied from 0 sec. up to 25 sec. in steps of 0.1 of a sec. The hold time can be also be selected from 0 sec. up to 25 sec. in steps of 0.1 sec. The internal gas flow rate can be set from 0 mL/min to 8000 mL/min.

Temp: The user can enter the temperature at which he wants the furnace to be. This temperature can be between ambient and 3000 °C.

Ramp: This value is the time that it takes to go from the current temperature to the target temperature.

Hold: This value is the time that the user wants to keep the furnace at the current temperature.

Gas: This value is the flow rate of the gas at that step.

Plasma On/Off: This option if set to on will send a CMOS (open drain) signal to ICP

The user should refer to section 4.3 for more details on developing GF methods and the various stages associated with the furnace program.

 *Copy Method Settings to Instrument*:

This function will copy all of the settings that the user has inserted into his/her method to the instrument status window.

 *Copy Instrument Settings to Method*:

This function will copy all of the settings in the instrument status window to the method settings.

4.3.1 Furnace Program (Heating curve profile):

During furnace program development, the operator can check the results of any changes by saving the method and initiating a single run. Details pertaining to the single run can be found in Section 4.4.

Drying Step

The drying step should provide efficient, even drying of the test solution droplet thus avoiding sample ejection or splattering around the inside of the tube. Sample loss lowers sensitivity and raises detection limits and uncertainty. Quickly raising the temperature to a few degrees below the boiling point of the solvent, then slowly raising the temperature past the boiling temperature normally provides the best results.

Example: for aqueous solutions:
1 sec ramp to 90 °C ,
hold for 1 sec,
follow it with a 25 sec ramp to 120 °C and
hold for 3 sec.

Ashing Step (also known as the charring step)
Charring or thermal pre-treatment is used in conventional GF AAS to remove all or as much of the dry and/or semi-dry matrix left over from the drying step as possible without removing any of the analyte species. Matrix modifiers such as $Pd(NO_3)_2$ or $Mg(NO_3)_2$ can be added to stabilize the analyte during charring while the matrix is removed. Chemical modifications can also be used. For example, addition of oxygen or hydrogen to the purge gas, can change the chemical nature of the analyte and remove some of the matrix.

Charring curves are constructed by plotting atomization or emission intensity (peak height) as a function of charring temperature. The optimum charring temperature can be selected from the charring curve - it is normally the highest temperature prior to loss of analyte on atomization.

Atomization / Excitation Step
During the atomization step, the solid phase analyte containing species are vaporized and/or atomized in the high temperature environment of the furnace. The transversely-heated integrated contact furnace, coupled with high speed temperature control and feedback, provides a furnace heating rate of over 3800 K/s and a temporal temperature profile that is nearly isothermal. This is important in order to reduce the amount of analyte expelled from the furnace by gas expansion during the temperature ramp.

It is also advantageous to have the analyte vaporized after the furnace has reached the optimum atomization temperature. The transversely heated integrated contact furnace provides a spatial temperature profile that is nearly isothermal. This also prevents analyte from being lost during the atomization step. Atomization curves are constructed by plotting absorbance or emission intensity (peak height) as a function of atomization temperature. The optimum atomization temperature can be selected from the atomization curve; it is normally the lowest temperature at which the largest signal is obtained.

4.4 Single Run

After the above instrument settings and method development have been completed, the operator may find it useful to perform a single run to view the signal profile and/or gather data to further optimize a method.

Click on the single run button on the tool bar and the chart window will appear. To start the single run click on the run button located in the top right hand corner of the window. The user must ensure that the method settings have been copied over to the instrument. If this has not been done they will be prompted to do so.

4.4.1 Chart Window

When the operator clicks on the run icon the chart window will start the real time display of the current temperature and programmed temperature.

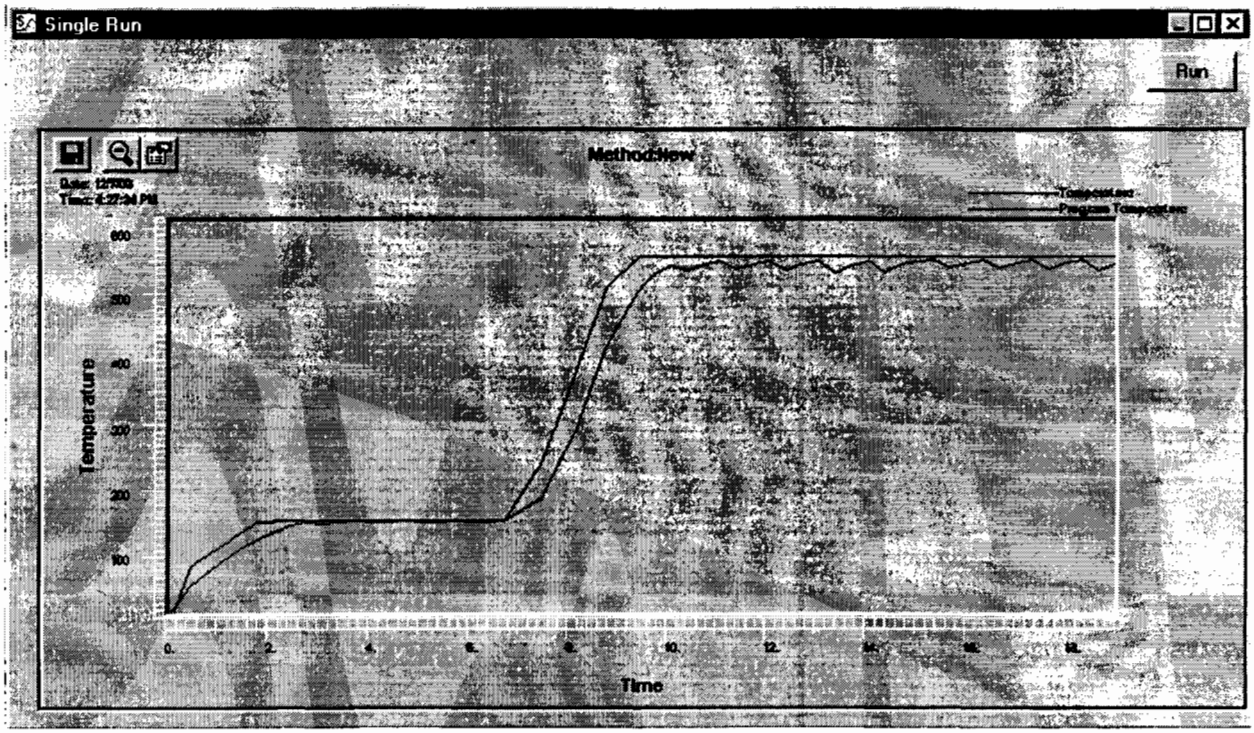


Figure 4 - 4 *Example Chart Window for GF 120*

Time is on the x-axis and the Temperature on the y-axis

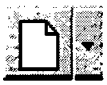
There are two parameters plotted in the Chart Window:

Temperature:	Red line. This is the actual temperature (°C) inside the graphite furnace (as read by the thermocouple for low temp and IR sensor for high temp).
Program Temperature:	Blue line. This is the temperature profile (°C) specified by the Furnace heating program. Ideally, the actual temperature and the program temperature profiles should almost overlap.

Clicking and dragging the left mouse button in the chart will zoom in on the selected area. To zoom out, click on the 'Zoom Out' icon button in the Chart window.

When the run is complete, the profile of both the temperature program and the actual temperature will be shown. The final Chart window can be saved or printed if desired.

4.5 Tool Bar Icons



New Icon: Clicking on the icon creates a new method file.



Open Icon: Clicking on the icon opens an existing method file



Save: Saves the active file under the current name.



Print: Prints a hard copy of the current selected window. A version of Microsoft Excel is required for this function to work properly.



Instrument Setup: Activates the Instrument Status window (brings it to the front).



Start Single Run: Initiates a single run, opening the 'Single Run' window.

A.1 GF Temperature Calibration Procedure - For on-site service and post-installation testing

GF temperature calibration is divided into low temperature calibration and high temperature calibration. The temperature range of room temperature to 469°C is considered **low** temperature. Temperatures above 470°C (up to 3000°C) are considered **high** temperature.

Prior to beginning the GF temperature calibration it is important and expedient to proceed with a quick visual check of the GF temperature.

A.1.1 Visual check of GF Temperature.

"hi_temp.mth" method: use a 4 step procedure as follows:
Step 1 – Temp (°C). 120°C; ramp time 2.00 seconds; hold time 1.00 seconds.
Step 2 – Temp (°C).. 250°C; ramp time 3.00 seconds; hold time 3.00 seconds.
Step 3 – Temp (°C).. **2850°C** *; ramp time 0.00 seconds; hold time **2.00** * seconds.
Step 4 – Temp (°C).. 30°C; ramp time 0.00 seconds; hold time 1.00 seconds.

*** When testing the GF high temperature for the first time following instrument installation, relocation or prior to service, it is advisable to use a 0.5 sec hold time to protect the graphite tube. It is also advisable to start at a lower atomization temperature and then work up to 2850°C. When making the appropriate changes to the furnace program it is important to always save the changes and then re-open the method to institute the new parameters.**

A suggested furnace program to begin the visual check of GF temperature is:

Step 1 - Temp (°C)..120°C, ramp time 2.00 seconds; hold time 1.00 seconds.
Step 2 - Temp (°C).. 250°C; ramp time 3.00 seconds; hold time 3.00 seconds.
Step 3 - Temp (°C).. 500°C; ramp time 0.00 seconds; hold time 0.50 seconds.
Step 4 - Temp (°C).. 30°C; ramp time 0.00 seconds; hold time 1.00 seconds.

Click on the icon for single run and observe the graphite tube during atomization. At 500°C, there should be little change in the color of the graphite tube. Increase the temperature to 600°C, the tube should begin to glow a red color, increase the temperature to 1100°C, the tube should now be glowing bright orange. At temperatures over 1600°C the tube should be glowing brightly. See the following table for more information.

The following visual signs should be expected when testing the high temperature calibration:

TEMP °C	Graphite Tube Color
550 - 600	Low red glow
700 - 900	Deep red glow
1000 1500	Bright orange
1600 - 2500	Glowing brightly
>2500	Bright white, no smoke

If the correct colors were observed at each temperature, the instrument is roughly calibrated at high temperature. It would now be appropriate to proceed with the low temperature calibration.

A.1.2 Low temperature calibration

For the GF-120, a thermopiler sensor was employed for the low temperature readings. The output of the thermopiler sensor is linearily related to the actual temperature inside the furnace tube. A series of coeffiecients were used to calibrate the low temperature and all of these coeffiecients were saved in the EEPROM which are mounted on the PCB. These coefficients can be accessed and modified by the tools button on the tool-bar.

To set the coefficients click on the service mode in tools pull-down menu. This will activate the service mode allowing you to access the GF temperature calibration menu. When selecting the calibration menu a window will pop up with a table showing GF temperature coefficients for the low temperature range. These coefficients are for the calibration of the low temperature range. The K_0 coefficient is the slope of the temperature calibration curve and the K_1 coefficient is the intercept.

The entire low temperature range was divided into two ranges; 30° – 269°C and 270° – 469°C. Each range is affected by the coefficients respectively.

To calibrate the low temperature the user must first establish a method called "Low_Temp" with the following parameters:

STEP	TEMP (°c)	RAMP	HOLD	GAS FLOW
1	60	0.0	2.0	2.0
2	100	2.0	20.0	2.0
3	260	2.0	20.0	2.0
4	280	2.0	20.0	2.0
5	460	2.0	20.0	2.0
6	60	0.0	5.0	2.0

Prepare a thermocouple temperature meter. (The best is to use a Type-K thermocouple. An 80PK2A Immersion Probe manufactured by Fluke is recommended.)
Insert the thermocouple probe into the furnace tube and ensure that the probe is in contact with the inside wall of the furnace tube.

Start the heating cycle. When the heating cycle ends, by observing the thermocouple meter, you will see the furnace cooling down. When the temperature drops down to 60°C, re-start the heating cycle. This time you should record the measured temperatures for each step. The measured temperature should be recorded at the end of each step.

Compare the measured and programmed temperatures.

To calibrate the instrument the user must first check the slope of the calibration curve (K_o) for example:

The program temperature is 280 and 460 and the measured temperature of the furnace is 300 and 460, then the increment for the programmed temperature is 180 but the increment for the measured temperature is 200. This means that the K_o should be increased i.e. from 100 to 110. The user can save this and then repeat the process again. If the increment is ok but the actual temperature is still to high then the user can reduce K_1 to change the real temperature. After the one range is done then you can try the other range.

Aurora recommends that the user calibrate the low range first because the low range will affect the measurement of the high range.

Repeat the above process until the deviations between measured and programmed temperatures are within 5% for all the temperature points (or at least for the majority of the temperature points).

A.1.3 High temperature calibration

The coefficients listed in the high temperature range of the temperature calibration table were for PID amplifiers. To really calibrate the high temperature the R35 on the ATI board should be adjusted at 2850°C.

High temperature calibration is more complicated. The following is a procedure for **rough** calibration for the purpose of on-site service.

Note: Use of a new furnace tube is highly recommended for high temperature calibration.

When the low temperature calibration is finished, the instrument is ready to undergo high temperature calibration.

"non-std" method: use a 4 step procedure as follows:

Step 1 – final temp. 120C; ramp time 2.00 seconds; hold time 1.00 seconds.
Step 2 – final temp. 250C; ramp time 3.00 seconds; hold time 3.00 seconds.

Step 3 – final temp. 2850C; ramp time 0.00 seconds; hold time 2.00 seconds.
Step 4 – final temp. 30C; ramp time 0.00 seconds; hold time 1.00 seconds.

Warning! Caution is required during high temperature calibration to avoid overheating the furnace. Overheating the furnace may damage the furnace tube. To avoid overheating, follow the points below:

When beginning high temperature calibration do not set the atomization temperature to 2850°C immediately. Rather, start the calibration procedure at a lower atomization temperature, such as 1000°C and a shorter hold time, such as 0.5 seconds. Gradually increase the atomization temperature to 2850C, still with a 0.5 second atomization time. Then, increase the atomization time to 2.00 seconds.
Cooling time should be 20 seconds or longer.

It is safer to use a 0.5 sec hold time when first testing the high temperature calibration to protect the graphite tube. It is also advisable to start at a lower atomization temperature and then work up to 2850°C.

The following visual signs should be expected when testing the high temperature calibration:

TEMP °C	Graphite Tube Color
550 - 600	Low red glow
700 - 900	Deep red glow
1100	Bright orange
1600 - 2500	Glowing brightly
>2500	Bright white, no smoke

If everything looks good at 2500 °C, then proceed to the next step.

Using the original "non-std" heating program detailed on page 4, (atomization hold time of 2.0 sec and atomization temperature of 2850 °C), start the heating cycle and carefully watch the heating process. A small amount of visible fumes will be produced. In this case, the actual temperature is higher than 2850°C and the value of R35 must be *increased.* If there are no fumes then the actual temperature is lower than 2850°C and the value of R35 must be *decreased.* Adjust the R35 value so that a small amount of fumes are produced from the furnace at

an atomization temperature of 2850°C.

Do not change the values of any other coefficients in the initial file.

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