

**Standard Operating Procedure for
Varian SpectrAA 240FS with
Graphite Furnace
Last Modified April 22, 2010**



Considerations Prior to Beginning Experiment

- For most samples, standard solutions should be in the ppb range, particularly for quantitative work.
- All solutions should be prepared with ultrapure (18M Ω) water and trace metal grade reagents.
- All glassware for samples and standards should be washed with dilute nitric acid followed by rinsing with ultrapure water.

Instrument Start-up Procedure

1. Power on both the SpectrAA 240FS spectrophotometer and the GTA 120 furnace controller. *These must be powered up first or the software runs very slowly.*
2. Open the valve on the Argon tank and check to see that the pressure on the left regulator gauge is approximately 60 psi.
3. Open the valve on the water spigot (located to the right of the instrument on the benchtop) to start water flow. Check to see that water is flowing into the drain. The valve does not need to be fully open. A flow rate of 1-2 liter per minute is sufficient.
4. The container attached to the bottom of the sample carousel should be filled with dilute trace metal grade nitric acid. If it is not, prepare additional acid solution by diluting a few mL of trace metal HNO₃ in 18 M Ω water and fill the container.
5. Check to ensure that the waste tube is placed in the waste bottle located on the floor below the instrument. If the bottle is full, dispose of the contents appropriately.
6. Start the SpectrAA software.

Preparing the Instrument for a Measurement

1. Remove the sample carousel from the autosampler. Load standards and samples into autosampler vials and place the vials in the carousel. Be sure to note the position of all standards and samples. DO NOT load sample vials while the carousel is in the autosampler! Spills can cause serious damage to the instrument! Once all vials are loaded, return the sample carousel to the autosampler.
2. From the SpectrAA software main menu select "New Worksheet", followed by "Add Method". (If you have a saved method you wish to use, load it here.)
3. In the "Add Methods" window that appears, make the selections that are appropriate for your experiment. Since the graphite furnace is typically installed in the instrument, be sure that "Furnace" is checked. (If you are using another sampling method, consult the instrument manual or your instructor for assistance.) Scroll down the element list and select the element of interest. Typically "Cookbook" is selected in the "Load From" portion of the window to provide initial settings for the analysis the element selected. Click OK to conclude your selections.

- Click on “Edit Methods” and use the tabs in the window to make additional selections to configure your method. Typical parameters that are adjusted are shown in the table below.

Tab	Parameter and Purpose
Type/Mode	Units : Set concentration units used for presenting results
Measurement	Measurement Mode : Typically “Peak Area” is selected Calibration Mode : Make the appropriate choice for your analysis
Optical	Make appropriate lamp position and monochromator settings for your analysis
Standards	Concentration : Set concentrations that the sample will prepare for standards
Sampler	Volume : Set sample and total volumes Reagent Tubes : Identify where bulk and make-up solutions are and indicate concentration of bulk standard.
Furnace	Furnace Injection : Make appropriate choice
QC Tests	Typically no changes are needed.
Notes	Notes can be added if desired.
Cookbook	Typically no changes are needed.

Once you are certain that all selections are made, click OK. Clicking OK will “lock” some parameters and prevent changes, so do so on when you are sure of your settings.

- Click on the “Instrument” tab. In the window that appears, click “Optimize” to begin optimizing light throughput and detector signal. Depending on your method settings, you will see either one or two signal bars. Adjust the alignment of the light source(s) to maximize the signal in each bar (typical maximum signal is approx. 0.9 volts). If you are unable to obtain this maximum, contact your instructor. You may need to click “Rescale” if your signal is out of range. Once optimized, select “Exit” to return to the worksheet window.
- From the top menu of worksheet window, select “Instrument”, then “Furnace Facilities” to bring up a window to prepare the furnace for use. Click “Align Probe” to align the sampling arm with the center of the sample vials and with the graphite furnace itself. Your instructor will demonstrate alignment procedure. Once you are satisfied with the alignment, click OK. You can use the built in webcam to help with alignment.
- Click “Rinse” to rinse the probe. Do this twice. Your instructor will demonstrate how to remove air bubbles from the probe, should they appear.
- Click “Tube Clean” to run the tube through a heating cycle. Click “Exit” to return to the worksheet window.

General Steps for Making a Measurement

- Use the “Select” tool to highlight cells in the sample table that correspond to sample vials in the sample cell. Vials will only be sampled if they are highlighted prior to starting data collection.
- Click “Start” to begin data collection. As standards and samples are collected, data will be displayed on the calibration curve at the right of the window and eventually in the data table. The units used in the data collection are those that were selected during development of the Method.

Data Output and Storage

Data are automatically stored with the worksheet. To retrieve old data, simply load the corresponding worksheet

Instrument Shut-down Procedure

1. Remove the sample carousel; remove all sample vials from the carousel and dispose of them properly. Return the carousel to the autosampler.
2. Shut down the computer.
3. Power down the spectrophotometer and GTA controller.
4. Close the valve on the argon tank.
5. Close the valve on the cooling water.

Troubleshooting Tips

- The precision of your experimental results is highly dependent on the autosampler tube reproducibly dropping the sample into the graphite furnace. It is critical that the probe gently transfer a drop of sample onto the base of the tube. If irreproducible results are obtained, check the probe/tube alignment first!
- Graphite tubes are typically good for over 250 firings. If performance is erratic and samples are being injected correctly into the graphite tube, it may be an indication of a worn tube. Contact your instructor for tube replacement instructions.