

**Standard Operating Procedure for  
Varian SpectrAA 200 or 240 with  
Flame Atomization  
Last Modified October 29, 2010**



**Considerations Prior to Beginning Experiment**

- Be sure standards are in the appropriate concentration range to promote linearity for your analyte/flame combination.
- All solutions should be prepared with ultrapure (18M $\Omega$ ) 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 the SpectrAA instrument.
2. Pull off the burner head to check if the liquid trap is filled. Check by pouring distilled water into the spray chamber through the burner head collar until you observe water running through the drain tube into the waste bucket. Replace the burner head, making sure that it is secure.
3. Make certain that the waste container has been emptied before you start. After emptying the container, replace the drain tube. When replacing the tube, make sure that the end of the tube is NOT submerged.
4. Turn on the exhaust hood before any fuel or oxidant lines have been opened.
5. Open on the air valve that is located on the benchtop by turning the handle parallel with the hose. Check that the regulator hanging on the wall behind the instrument reads around 55-65 psi. At any time during use DO NOT shut the air off.
6. Open the valve on the top of the acetylene tank and verify the pressure remaining in the tank is well above 50psi (right gauge) and that the operating pressure is around 10psi, no more than 15psi (left gauge). If you are using nitrous oxide as your oxidant, open the valve on this tank as well and verify that the operating pressure is around 50-60 psi.
7. Power on the computer and start the SpectrAA software.

**Preparing the Instrument for a Measurement**

1. From the SpectrAA main menu select "Worksheet", then "New" and give your worksheet a file name. All data is stored in a single worksheet. (If you have a saved method you wish to use, load it here.). Under the "Develop" tab, click "Add Method", check flame on the left and uncheck anything else that may be checked, and then select the element of interest from the list. Typically "Cookbook" is selected in the "Load From" portion of the window to provide initial settings for the analysis of 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	<b>Instrument Mode:</b> Select “Absorbance” or “Emission” as appropriate for your analysis <b>Sampling Mode:</b> Select “Manual”
Measurement	<b>Measurement Mode:</b> Typically “Integration” is selected for flame measurements. <b>Calibration Mode:</b> Make the appropriate choice for your analysis
Optical	Make appropriate lamp (if in absorbance mode) and monochromator settings for your analysis
Standards	<b>Concentration:</b> Select the units for your solutions and enter the concentration of your standards.
Calibration	“Linear” is typically a good choice here.
QC Tests	Typically no changes are needed.
Notes	Notes can be added if desired.
Cookbook	Typically no changes are needed.

NOTE: The SIPS and Sampler tabs control an optional autosampler system that we typically do not have installed on our instrument.

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

- Light the flame by pressing and holding the flame ignition button on the instrument (the one with the black flame drawing above it) until you hear and see the flame. You should aspirate DDI H<sub>2</sub>O at this time to flush the nebulizer and spray chamber. (At any time the flame may be put out by pressing the red button to the left of the flame ignition button on the instrument)
  - An air/acetylene flame should be blue/orange with a small, ~0.5cm, crisp blue flame near the burner head (no soot). The standard settings generally produce a reasonable flame (13.50/1.20 L per minute, air/acetylene). Flow rates can be adjusted in the “Optimize” window (described below) to change the flame characteristics BUT THIS CAN RESULT IN AN EXPLOSION IF YOU DO NOT KNOW WHAT YOU ARE DOING!
- Click on the “Analysis” 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.
  - If you are operating in absorbance mode, 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.
  - If you are operating in emission mode, aspirate a high concentration standard and verify that a reasonable signal is observed at the detector.

Once optimized, select “OK” to return to optimization selection window and close the window to return to the worksheet view.

### **General Steps for Making a Measurement**

1. Use the "Select" tool to highlight cells in the sample table that correspond to unknown samples you intend to run. Data for unknowns will only be collected if the appropriate cells are highlighted prior to starting data collection.
2. Click "Start" to begin data collection. The software will ask you to light the flame if you have not already done so, then it will ask you to do one of two things, depending on your measurement mode.
  - For **emission** experiments, you will be asked to present your top standard for peaking. This is your most concentrated standard solution. Aspirate the solution by placing the end of the capillary into your solution. You will hear the solution being drawn up into the flame. Follow the prompts on the screen. After peaking, the software will ask you to aspirate a blank (DI water or other sufficient blank).
  - For **absorbance** measurements, you will be asked to prepare for Instrument Zero. To do so, aspirate a blank (DI water or other sufficient blank) to allow the instrument to calibrate for zero absorbance.
3. At this time, the software will go step by step through the analysis asking you to aspirate each of your standards and finally your sample(s). Do not remove one solution from being aspirated until the software asks you for the next solution, as it will take duplicate measurements. The program will not continue from one solution to the next until you press the OK button. The status of the instrument/program can be viewed at the lower right hand corner of the screen.

### **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. Aspirate DDI water for approximately five minutes.
2. With the flame lit, close the valve on the acetylene tank and let it bleed out the line until the flame goes out.
3. Once the flame is out, close the air valve on the benchtop and the valve on the nitrous oxide tank (if it was used).
4. Shut down the software and computer.
5. Power down the spectrophotometer.

### **General Tips**

- The software runs sluggishly without the instrument powered up. Turn the instrument power ON before starting the software and close the software before powering the instrument down.
- With the flame going NEVER shut off the air as this will cause insufficient air flow to keep the flame above the burner and will result in the flame burning back into the spray chamber and an EXPLOSION.
- The burner position, nebulizer distance, and uptake rate may need to be adjusted to achieve optimal results. Seek assistance for details.
- This is not a complete list of safety checks that need to be completed before making a run. This was intended to be a document describing how to use the instrument after the instrument had been determined safe to run (See Flame AA Maintenance or your instructor for further details). If tanks or tubing has been replaced, the system must be leak-checked prior to use.